SYNTHESIS, CHARACTERISATION OF BIO-ADDITIVES FOR PERFORMANCE STUDY OF BIO-LUBRICANTS

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by

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DECLARATION

I, hereby declare that the thesis entitled "**Synthesis, Characteristics of Bio-additives for performance study of Bio-lubricants**" is an original work carried out by me under the supervision of Dr. Rajiv Chaudhary, Professor, Department of Mechanical Engineering, Delhi Technological University, Delhi and Dr. R. C. Singh, Professor, Department of Mechanical Engineering, Delhi Technological University, Delhi. This thesis has been prepared in conformity with the rules and regulations of the Delhi Technological University, Delhi. The research work presented and reported in the thesis has not been submitted either in part or full to any other university or institute for the award of any other degree or diploma.

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CERTIFICATE

This is to certify that the thesis entitled **"Synthesis, Characteristics of Bio-additives for performance study of Bio-lubricants"** submitted by **Mr. Anshul Kumar** to the Delhi Technological University, Delhi for the award of the degree of **Doctor of Philosophy** in **Mechanical Engineering** is a bona fide record of original research work carried out by him in our supervision. His research work is in accordance with the rules and regulations of Delhi Technological University, Delhi. The results presented in this thesis have not been submitted in part or full to any University or Institute for the award of any degree or diploma.

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न हि सुप्तस्य सिंहस्य मुखे प्रविशन्ति मृगाः"

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ABSTRACT

Stringent global environmental regulations have spurred research into alternative fuels. There had been a total of 216 medium (7-700 tonnes) oil spills and 55 large (> 700 tonnes) oil spills in the environment from 2000 to 2023, primarily caused by human negligence. These spills have significantly disrupted ecosystems, harming plant, animal, and human life. Despite its widespread use in automotive engines due to its availability, cost-effectiveness, and performance, mineral oil poses severe environmental challenges and is derived from non-renewable resources. This underscores the urgency of exploring alternative lubrication solutions.

The environmental pollution caused by mineral oil-based lubricants, coupled with the depletion of petroleum reserves, has driven research into biolubricants. Biolubricants are the lubricants derived from renewable sources such as vegetable oils, animal fats, and synthetic esters. Biolubricants offer several advantages, including superior viscosity, biodegradability, and reduced toxicity when compared to petroleum-based lubricants. In the presented study, an apricot oil based biolubricant was produced to determine its tribological performance compared to the mineral oil based lubricant.

To assess the performance of apricot oil as a lubricant, various blends of apricot oil-based biolubricants and mineral oil-based lubricants were tested using the Four-ball tester and High Temperature Pin-on-disc Tribometer. These tests evaluated parameters like friction reduction, wear prevention, and durability under high pressure (~ 4.45 bar) and high temperature (~150^{o}C) conditions. The results revealed that pure apricot oil-based biolubricants exhibited higher wear and friction coefficients compared to standard mineral oil lubricants like 15W40 or SAE40. However, an optimized blend (70% 15W40 oil and 30%

apricot oil biolubricant) demonstrated less coefficient of friction and wear rate over either component used individually. Further testing on a journal bearing test rig (TR-660) confirmed that the apricot oil-based biolubricant performed less effectively than 15W40 mineral oil in terms of load-carrying capacity as the generated maximum pressure (~ 1.21 MPa) for biolubricant is less than that of 15W40 oil (~ 3.02 MPa). However, the blend of 15W40 mineral oil and apricot oil biolubricant offered enhanced performance, with the optimal ratio determined to be 70% mineral oil and 30% apricot oil-based biolubricant.

The study concludes that while apricot oil-based biolubricants alone may not match the performance of conventional mineral oil lubricants, carefully optimized blends can achieve superior tribological properties. Given the rapid depletion of petroleum reserves and increasing environmental concerns, vegetable and animal oil-based lubricants present a viable, sustainable alternative to mineral oil-based products, contributing to a more sustainable future.

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NOMENCLATURE AND ABBREVIATION

ANOVA	Analysis of Variance
ASTM	American Society of Testing and Materials
DLC	Diamond like Coating
DoF	Degree of Freedom
FAME or FAEE	Fatty Acid Methyl Ester or Fatty Acid Ethyl Ester
FFA	Free Fatty Acid
FTIR	Fourier Transform Infrared
FTP	Flash Temperature Parameter
LVDT	Linear variable differential transducer
MS	Mean square
PAH	Polycyclic Aromatic Hydrocarbons
PEE	Pentaerythritol ester
POME	Palm oil methyl ester
PO	Palm Oil
RSM	Response surface methodology
SBO	Soybean Oil
SFE	Supercritical fluid extraction
SS	Sum of squared deviation
TMP	Trimethylopropane
VI	Viscosity Index
WSD	Wear scar diameter
ZDDP	Zinc di-alkyl dithiophosphates
A'	Cross sectional area

A_r	Real contact Area
F	Frictional Force
F_p	Ploughing Force
F_s	Adhesive Force
H	Material Hardness
K	Wear coefficient
L	Bearing length
N	Normal Load
N_s	Rotational speed
p	Pressure
R	Radius of steel balls
s	Wear scar radius
S	Shearing strength
t	Sliding time
T	Temperature
T_f	Frictional torque
V	Velocity
W	Applied load
W_v	Volume of material removed
μ	Coefficient of Friction
η	Lubricant Viscosity
ε	Eccentricity ratio
θ	Angular bearing position

CHAPTER 1: INTRODUCTION

This chapter contains the motivation behind the presented research work and also contains the introduction of tribology and lubrication. Further, the chapter provides the introduction to biolubricants based on vegetable oil and animal oil, along with the development in the field of biolubricants.

1.1 Motivation

Friction arises when two solid surfaces in relative motion rubs against each other, acting as a force that opposes this motion and generates heat, which in turn raises the temperature of the surfaces. In many mechanical systems, friction is beneficial, such as in brake system, clutch system, and belt-driven pulleys; and also there are mechanical systems where friction is detrimental. Excessive friction in an engine leads to a loss of mechanical energy, requiring more fuel to accomplish the same task. Additionally, increased friction can cause the engine to overheat and may result in the wear or seizure of its moving parts.

Wear occurs when two surfaces interact, leading to the removal or deformation of one or both surfaces due to friction. This process often takes place at the microscopic contact points, known as asperities. In automotive engines, even a small amount of wear could lead to component failure and necessitate replacement of the worn out component.

To reduce friction and wear, lubricants are applied to the contacting surfaces. In an automotive engine, oil is used to lubricate engine components, which is crucial for optimal engine performance and wear prevention. Lubricants not only facilitate smoother movement but also help transfer heat away from engine parts, cooling them down. Additionally, they clean internal engine components by removing small particles of metal and dirt. A well-designed lubrication system can lead to quieter engine operation and extend the engine's lifespan.

Environmental pollution caused by mineral oil is a significant concern and should not be underestimated. Oil spills on land and water primarily result from human negligence, such as the leakage of used engine oil or crude petroleum. In marine environments, spills can occur due to accidents involving oil tankers or offshore oil platforms. The number of medium (7-700 tonnes) & large (>700 tonnes) oil spills per decade (1970-2023) is shown in Figure 1 [1]. Oil spills have detrimental effects on plant, animal, and human life. The oil forms a layer on the water's surface, blocking sunlight and hindering the survival of aquatic plants and animals.

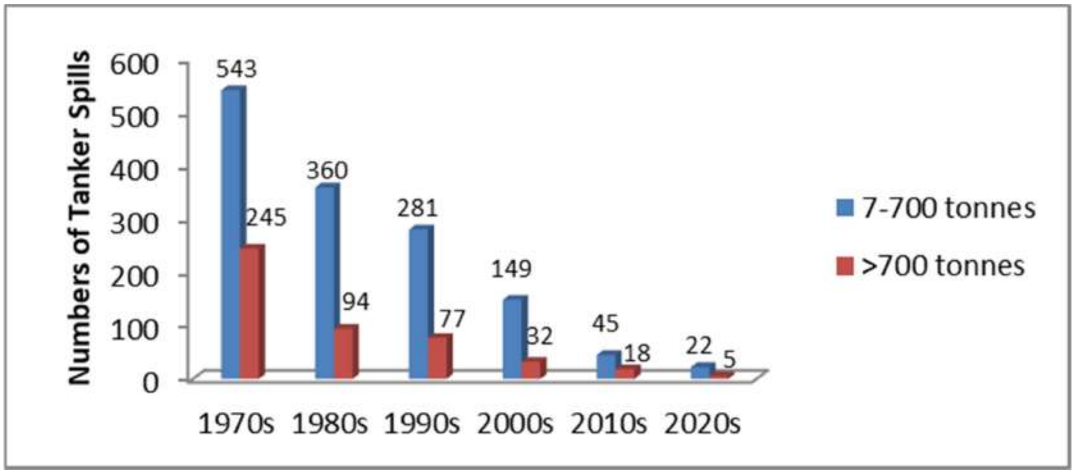


Figure 1: Number of medium (7-700 tonnes) & large (>700 tonnes) oil spills per decade, (1970-2023).

Given the information above, there has been growing interest in exploring biolubricants as a sustainable replacement for mineral oils. Derived from renewable sources like vegetable oils and their esters, biolubricants offer a greener alternative. They are biodegradable and possess lower toxicity compared to mineral oils, making them significantly more environmentally friendly. However, vegetable oil-based lubricants do have some limitations, including poor oxidative stability, which could negatively impact their performance in tribological applications. Therefore, it is crucial to conduct a tribological analysis of vegetable oil-based lubricants to explore their potential as biolubricants in mechanised systems.

1.2 Tribology

The term Tribology was derived from a Greek word *Tribos* meaning "to rub" and *Logos* means "study of". Tribology is a study of friction, wear and lubrication that occurs between the rubbing surfaces. The field of tribology combines the principles of mechanical engineering, material science, chemistry and physics, to understand the behaviour of interacting surfaces under various conditions. Tribology is an important aspect while designing more efficient, reliable and durable systems for industrial applications. Most of the research work in the field of tribology aims to reduce energy losses in a mechanical system, prevent equipment failures and to extend life of components. The study of tribological behaviour of a system includes the determination of friction and wear under a lubricating environment.

1.2.1 Friction

Friction is a force between two contact surfaces and it resists the motion among the surfaces. Friction plays a crucial role in day-to-day human lives and for the proper functioning of machines. From the grip of car tires on the road to the heat produced by rubbing hands together, friction is essential for movement control and energy transfer. Friction also causes wear and energy losses in a system, making it an important factor while designing machines and to improve their efficiency in the physical world.

1.2.2 Wear

Wear is phenomenon of gradual material loss that occurs when two surfaces are in rubbing contact with each other. There are many factors which influence the wear between the surfaces, including properties of materials in contact, the force applied on the surfaces, the speed of the motion between the surfaces, and the surrounding environment of the interaction. With time, the wear of surfaces can affect the functionality, efficiency and the lifespan of machine components in a wide range of applications. The understanding of wear mechanisms is crucial for improving material durability, reducing maintenance costs and improving the system performance.

1.2.3 Lubrication

Lubrication implies the process of minimising friction and wear between the contacting surfaces which are under relative motion with each other. Lubrication is achieved by a employing a lubricating substance, such as oil, grease or other fluids, between the surfaces. The usage of lubricants can lead to more efficient and smooth functioning of machines and mechanical systems. Also, the usage of lubricants extends the life-span of mechanical systems and reduces energy consumption. A lubricant reduces the direct contact between the contacting surfaces by forming a layer of lubricant and the lubricant layer not only prevent the wear and overheating but also improves the overall reliability and safety of machines.

Lubricants are mainly derived from mineral oil or petroleum oil by refining process. The refining process removes the impurities and it results into stable, versatile base oil with low volatility, making the product lubricant suitable for wide range of temperature conditions, hence making the mineral oil based lubricant popular in automotive and industrial applications.

Lubricants based on mineral oil are mainly used for industrial and automotive applications. However, due to the chemical composition of mineral oil based lubricant, their widespread use poses environmental and human health risk.

The prolonged exposure of mineral oil-based lubricants causes health risks of humans. Humans working in an environment exposed to mineral oil based lubricant may inhale or absorb toxic components through skin and it leads to potential health problems like skin diseases, respiratory disorders and even cancer. The polycyclic aromatic hydrocarbons (PAHs) present in lubricants have carcinogenic properties which further increases the health risks [2]. The mineral oil based lubricant enters into the environment either through leaks while transporting the lubricants or improper disposal of lubricants. The mineral oil based lubricants degrade very slowly in the nature; hence it gets accumulated in the soil and water bodies. When mineral oil-based lubricants were disposed-off onto the soil, it can inhibit the plant growth by interrupting the soil aeration and reducing the soil nutrient availability which can harm the flora and fauna of the locality and impact the agricultural productivity. The spillage of mineral oil lubricant onto the water bodies leads to contamination of aquatic ecosystems, harming marine life and disrupt food chains.

The environmental pollution caused by mineral oil is a serious concern. Oil spills on land and in water are often the result of human negligence, including the improper handling of used engine oil or crude petroleum. At sea, oil spills may originate from tankers or offshore drilling platforms. From the year 2019 to 2023, the numbers of oil spills incidents have been reduced, maintaining the downward trend that has been persisted over the last decade. Table 1 shows the annual number of oil spills from year 2019 to 2023.

Table 1: Annual number of oil spills from year 2019 to 2023. [1]

Year	Mid-sized oil spills (< 700 tonnes)	Large sized oil spills (>700 tonnes)
2019	2	1
2020	4	0
2021	5	1
2022	4	3
2023	9	1

Oil spills pose significant risks to plants, animals, and humans. The oil that floats on water can block sunlight, creating harsh conditions for marine plants and animals to thrive. Oil spills onto the water bodies is one of the devastating disaster, affecting the marine life and coastal ecosystems, wildlife and human communities. The following points discuss the effects of oil spills on the environment.

1. Marine ecosystems: Due to the oil spills, the oil spreads over the water surface, forming a layer of oil that block the sunlight. This leads to the reduction of photosynthesis in marine plants and further disrupts the entire aquatic food chain. The oil spills also leads to the destruction of marine habitat. The spilled oil can suffocate the corals and destroy the mangroves essential for the fish and other marine organisms.
2. Coastal ecosystems: The spilled oil that reaches the shore can kill the plants and leads to the soil erosion and loss of habitats for species dependent upon the shore areas. Oil that gets accumulated on shore contaminates the beaches, wetlands and estuaries. It takes decades for these ecosystems to recover.
3. Wildlife: The spilled oil gets coated on the bodies of marine animals which disrupt their ability to maintain body temperature and move freely. The restricted movement of marine animals often leads to hypothermia, drowning or starvation. Also, when animals accidently ingest spilled oil they experience harm to internal organs like liver and kidney failure, respiratory issues and reproductive complications.
4. Human communities: Oil spills can severely impact local economies, especially in areas dependent on fishing, tourism, and recreation, by causing the loss of marine life, polluting beaches, and damaging fisheries, which can lead to prolonged financial challenges. The prolonged exposure to chemicals from oil spills either through direct contact, inhaling fumes, or consuming contaminated seafood, can harm human health, resulting in skin irritations, respiratory issues, and potentially serious long-term diseases, including cancer.

The problems arises from the wrongful disposal or leakage of mineral oil and its products, has become a matter of concern. Also, the mineral oils are derived from non-renewable sources and rapidly depleting in nature due to rising high demand of mineral oil based lubricants. The researchers are more focused towards finding an alternative to mineral oil for industrial and automotive applications. Biolubricants is a renewable alternative for mineral oil based lubricants for industrial and automotive applications.

1.3 Biolubricants

The term "biolubricant" refers to lubricants that rapidly break down in the environment and are safe for both humans and aquatic life. These lubricants are produced from renewable biological sources, such as plant-based oils. Biolubricants offer main advantages of being biodegradability, having non-toxic nature and reduced environmental impact over the mineral oil based lubricants. There are multiple sources of base oil of biolubricants, as discussed below:

1. Vegetable oils: Vegetable oils are the most widely used source of base oil of biolubricants. Oils from crops such as soybean, sunflower, rapeseed, palm, and castor contain fatty acids with properties that make them suitable for lubrication. These oils are plentiful, renewable, and highly biodegradable due to their inherent makeup. They also provide excellent lubricity and thermal stability.
2. Animal fats: Animal fats can be utilized to make biolubricants, albeit they are less prevalent than plant oils. These fats, which are derived from fish oil, tallow, and lard, have natural lubricating qualities and are biodegradable. They are less popular because of supply constraints, ethical issues, and the requirement for particular processing.
3. Synthetic esters: Synthetic esters are chemically designed to replicate or improve the characteristics of natural oils and are derived from organic acids and alcohols. They may be made to function well in harsh environments, including high pressures or a temperature, which makes them appropriate for use in industrial settings. They are altered to obtain particular properties like low volatility, oxidation stability, and greater flash points, even though they are partially derived from natural sources.

Biodegradation refers to the breakdown of organic materials by enzymes generated by living organisms. This process is significant in fields like ecology, waste management, and bioremediation. Organic matter can decompose in the presence of oxygen, known as aerobic degradation, or without it, termed anaerobic degradation. A bio-mineralization is a process which involves the conversion of organic substances into minerals. Biodegradation is the chemical transformation of a material through the actions of organisms or their enzymes.

Vegetable or plant oils have distinct characteristics compared to mineral oils owning to their specific chemical composition. Primarily, plant oils consist of approximately 98% triacylglycerols, where different fatty acids are bonded to a single glycerol molecule. The fatty acids in these oils are generally long, unbranched aliphatic chains where hydrogen atoms attached to carbon atoms and chain end with a carboxylic acid group. Shorter fatty acid chains, like those with six carbons, are water-soluble due to the polar group [3]. As the chain length increases, the fatty acids exhibit oily or fatty properties and become less water-soluble. Saturated fatty acids have straight carbon chains, but when hydrogen atoms are absent from neighbouring carbons, double bonds form, creating unsaturated fatty acids, which have lower melting points than the saturated ones. When double bonds are present at multiple sites, the fatty acid is termed polyunsaturated. Thus, fatty acids can be categorized as saturated, mono-, di-, or polyunsaturated, based on the number of double bonds present [4].

Biolubricants derived from vegetable or plant oils have the following advantages:

a. Benefit of being environmental friendly: Biolubricants are biodegradable in nature which makes far less harmful to the environment if spilled or released. Vegetable oil contains fewer toxins and pollutants compared to the mineral oil based lubricants.
b. Renewable sources of oil: Vegetable oils are derived from renewable sources which reduces the dependence on non-renewable petroleum sources. This helps in conserving the fossil fuel reserves and supports sustainable agricultural economies.
c. Higher Lubricity: Vegetable oil based biolubricants have high lubricity which leads to lower frictional losses and hence, yield more power and better fuel economy.
d. Higher viscosity indices: The biolubricants have higher viscosity index compared to mineral oil lubricant. Hence, the viscosity of vegetable oil based lubricants does not vary with temperature as much as mineral oil lubricant. High viscosity index is advantageous while designing lubricants for applications of wide temperature range.
e. Lesser carbon emissions: During the production of biolubricants, they have smaller carbon footprints compared to mineral oil based lubricants.

Apart from having numerous advantages, vegetable oil based lubricants have following disadvantages:

a. Higher cost of production: The production of biolubricants can be more expensive compared to the conventional mineral oil lubricants due to raw material sourcing and refining processes.
b. Poor thermal oxidative stability: When a substance is subjected to heat and oxygen, it undergoes a chemical breakdown known as thermo-oxidation. This procedure may result in the production of hazardous byproducts, including acids, sludge, and varnish, which have an adverse effect on the performance of biolubricants. These byproducts have the potential to accelerate wear on machinery parts, decrease the oil's lubricating qualities, and raise its viscosity.
c. Low hydrolytic stability: The potential of a substance to withstand chemical breakdown in the existence of water is known as hydrolytic stability. Biolubricant can be a concern due to the nature of their base oils, which can be more prone to hydrolysis compared to traditional petroleum-based lubricants.

1.4 Production of biolubricants

A biolubricant refers to a lubricant derived from natural sources, including vegetable and animal oils. Vegetable oils used in biolubricant production are extracted from plant seeds. Some of the vegetable oils used for biolubricant production are castor seed, karanja seed, palm oil, soybean oil, sunflower oil. Triglycerides are the main content of vegetable oils required for producing biolubricant. Triglycerides are molecules of glycerol having three long-chain polar fatty acids bonded to a hydroxyl group through ester linkages.

Biolubricants are produced by chemical modification of vegetable oils. The following are the chemical modification methods employed for production of biolubricants from vegetable oil [5].

1. **Transesterification Reactions:** Transesterification is a chemical process used to produce esters by combining an acid-containing compound with an alcohol, removing the water generated during the reaction. In this process, triglycerides interact with alcohols such as methanol or ethanol, in the surrounding of a catalyst, often a strong base like sodium hydroxide or potassium hydroxide. This reaction breaks down the triglycerides into fatty acid methyl esters (FAMEs) or fatty acid ethyl esters (FAEEs). FAMEs are the main components of biodiesel and they are also valued for their lubricating properties, hence making them suitable as biolubricants. The efficiency of transesterification process is controlled by parameters such as temperature, reaction time, and molar ratios of the reactants.

   The biolubricant produced through transesterification has several environmental benefits. It is biodegradable, non-toxic and has reduced carbon footprint compared to mineral oil lubricants.
2. **Hydrogenation reaction:** It is a chemical process of transforming vegetable oils into biolubricants. This process involves the chemical addition of hydrogen to the chain of unsaturated fatty acids of vegetable oil. Hydrogenation is performed by exposing the vegetable oil to hydrogen gas under the controlled condition of temperature and pressure, and in the presence of catalyst like nickel or palladium. During hydrogenation, double bonds in the unsaturated fatty acids are saturated with hydrogen. Hydrogenation process increases the oil's stability and its resistance to oxidation, hence making the product suitable for use as a lubricant. The biolubricant produced from Hydrogenation has improved viscosity, more durability and it has the capacity of working effectively under mechanical and thermal stresses.

3. **Epoxidation reactions:** Epoxidation is a chemical process of adding oxygen atom to the carbon-carbon double bond, present in the fatty acid chains of vegetable oil, to form an epoxide group. The double bonds present in unsaturated fatty acid of vegetable oil are susceptible to epoxidation which is carried out by using oxygen source such as hydrogen peroxide, and an acid catalyst like formic acid. The oxygen atom binds to each double bond forming an epoxide ring and reducing the reactivity of the vegetable oil towards oxidation and thermal breakdown. The molecular structural modification enhances the oil stability and resistance to oxidation. Epoxidised oils exhibits enhanced lubricity and viscosity, making it effective in reducing wear in mechanical systems.
4. **Estolides of Fatty acids:** Estolides are branched ester compounds created when the carboxylic acid group of one fatty acid bonds with the unsaturated site of another fatty acid, forming oligomeric esters in the presence of an acid catalyst (H_2SO_4) and an oxidant ($HClO_4$). This bonding generates a carbocation, which can undergo nucleophilic attack from additional fatty acids, leading to the formation of an estolide. Compared to triglycerides, these branched esters show greater resistance to hydrolytic degradation. The hydrocarbon chain length and estolide number have been found to significantly impact the biolubricant's physicochemical properties, as demonstrated with estolides derived from 2-ethylhexanol, oleic acid, and lauric acid. These estolides exhibit enhanced lubricity, improved thermo-oxidative stability, a higher viscosity index, and a lower pour point, all of which are advantageous for biolubricant applications.

1.5 Use of additives to improve lubricant properties

Additives play an important role in biolubricants for enhancing their performance, stability and lubricant properties. The biolubricants lacks certain desirable properties as found in the synthetic lubricant. Additives are used to address these limitations and improve aspects like oxidative stability, viscosity, wear protection and corrosion resistance. Phosphorus, sulfur, and zinc di-alkyl dithiophosphates (ZDDP) are common examples of long-established additives. However, additives containing metals, sulfur, phosphorus, and chlorine are known to negatively impact the environment due to their high toxicity.

In contrast, plant-based compounds, such as cystine Schiff base ester, serve as efficient alternatives in applications like anticorrosion, antiwear, and antifriction. This efficiency arises from their di-sulfide groups, which create a protective surface-complex film on metal surfaces under contact conditions [6]. Ethyl cellulose and ethylene-vinyl acetate have also shown potential as additives for viscosity modification, as even small amounts can significantly increase a biolubricant's viscosity. These compounds also contribute to reducing wear and friction between metal parts [7, 8].

In addition, non-toxic inorganic oxide nanoparticles, including ZnO, CuO, and TiO_2, form smooth, thin films that reduce surface asperities and wear by aligning in parallel sheets to promote sliding. This alignment enhances the overall friction and wears resistance of the biolubricant. Other layered materials, like boron nitride, graphene, molybdenum, and tungsten sulfides, are also effective as additives in these applications, providing additional protection and durability [9, 10].

Ionic liquids have demonstrated considerable potential as additives in various sliding materials due to several beneficial properties, including low flammability, minimal vapor pressure, low volatility, and relatively high thermochemical stability. Additionally, they possess unique lubricating qualities, such as high load-bearing capacity, anti-wear capabilities, and reduction of friction. However, primarily polar biolubricants among these ionic liquids face challenges with immiscibility, though these issues can often be addressed by incorporating them in an emulsion [11, 12].

1.6 Application of biolubricants

Biolubricants are becoming increasingly popular as a sustainable alternative to conventional mineral oil-based lubricants. They are valued for their reduced environmental footprint, biodegradability, and reliance on renewable resources. Biolubricants are used across various industries such as automotive, industrial machinery etc. The applications of the biolubricants are given below:

a. Engine oil: Engine oil contributes in reducing friction and wear between moving components while also protecting the engine system from corrosion. Biolubricants, which contain low levels of sulfur and phosphorus, demonstrate reduced volatility and improved performance compared to traditional mineral oil-based lubricants. Various biolubricants, such as castor oil, palm oil, and coconut oil, have shown promise as effective alternatives for use in two-stroke engine oils [13].
b. Hydraulic fluids: Hydraulic fluids are specifically formulated to transmit power within a hydraulic system while also providing necessary lubrication. A critical factor in their performance is compressibility. Vegetable oils, in particular, demonstrate low isothermal compressibility and suitable viscosity, making them viable candidates for

hydraulic applications. Oils such as rapeseed, palm, moringa, and rubber seed oil have exhibited outstanding properties when employed as hydraulic fluids [14–16].

c. Transmission oils: Transmission oil requires high viscosity, excellent thermal stability, and strong resistance to friction, along with efficient heat dissipation capabilities. However, the application of bio-based lubricants as base materials in transmission oils remains limited. Research indicates that bio-based transmission oils offer higher weld load capacity and produce a smaller wear scar diameter when compared to traditional transmission oils [17, 18].
d. Compressor oils: Compressor oils are designed to remain stable under extreme pressure and temperature conditions. A key challenge for these oils is ensuring thermal stability at high temperatures. Research has shown that epoxidized vegetable oils demonstrate excellent thermal stability even at temperatures exceeding 260°C. This suggests that epoxidized vegetable oils can serve as highly effective alternatives to mineral oil-based compressor oils, achieving comparable temperature performance [19].

1.7 Global market of biolubricants

With a growing awareness of environmental issues, both consumers and industries are increasingly favoring sustainable products like biolubricants made from renewable sources. Strict regulations aimed at minimizing the impact of petroleum-based lubricants on environment, are also driving the shift toward biodegradable options. Technological evolutions have further improved the efficiency and affordability of biolubricants, making them a more viable choice for manufacturers. The expanding automotive and machinery industries are actively seeking sustainable eco-friendly alternatives, which are further fueling market growth.

The biolubricant market faces several challenges that may slow its growth. A primary obstacle is the relatively high production cost of biolubricants compared to conventional petroleum-based options, which can discourage manufacturers from switching to eco-friendly options. Moreover, there is a need to increase awareness and understanding among end-users about biolubricants, as this affects the demand of the same. In certain industrial applications, the performance of some biolubricants may fall short of the rigorous standards required, creating reluctance toward widespread adoption. Additionally, the volatility in the supply and pricing of raw materials, such as vegetable oils, can impact the stability and scalability of biolubricant production.

In 2024, the vegetable oil segment led the market, capturing a substantial 89.6% share of total revenue. It is anticipated to maintain its leading position throughout the forecast period. Biolubricants produced from vegetable oils and animal fats are gaining traction due to their environmentally friendly qualities. The automotive sector led the market in 2024, capturing over 62.4% of total revenue. Engine oils in this segment stand out, as they perform better than traditional engine oils. Furthermore, bio-based engine oils are gaining traction due to their higher biodegradability, reduced toxicity for aquatic life, and low bioaccumulation, making them particularly suitable for automotive applications.

United States of America held over 76.5% revenue share of the overall North America biolubricant market. The biolubricants market in the U.S. is growing as the Air Force encourages the use of plant-based biodegradable products as a strategic approach to national security, which is a significant factor driving the market growth. North America is well-positioned to benefit from its ample supply of soybean and rapeseed feedstock, largely due to the region's high levels of biodiesel production. The biolubricants market in Europe is experiencing significant growth, driven by the robust automotive sector within the European Union, one of the largest industries globally and a vital component of the region's economy. As reported by the European Commission, the automotive industry directly employs around 2.6 million individuals in vehicle production, accounting for roughly 8.5% of the EU's total manufacturing workforce. The biolubricants market in Asia Pacific is expected to grow Countries like China, India, Indonesia, and other Southeast Asian nations are increasingly producing and exporting passenger cars and vehicles to developed regions. The relocation of production facilities to Asian countries due to favorable government regulations and lower labor costs is expected to further increase industrialization and automotive spending in the region. Additionally, the growing trend towards sustainable vehicles with improved efficiency will likely enhance both the production and demand for biolubricants.

In February 2024, Kraton launched SylvaSolve, a new line of bio-based oils tailored for use in various industrial fields, such as coatings, adhesives, and personal care products. Being derived from renewable sources, SylvaSolve provides a sustainable alternative to conventional petroleum-based oils, helping address the increasing demand for environmentally friendly options and supporting clients' sustainability objectives.

Meanwhile, in April 2023, Exxon Mobil revealed plans to invest approximately USD 110 million to build a lubricant production facility in India. The plant, slated to commence operations by the end of 2025, is expected to produce up to 159 million liters of lubricants annually. This strategic move is designed to meet the rising domestic needs across industries such as steel, manufacturing, mining, power, and construction, as well as the commercial and passenger vehicle sectors.

This chapter shows the introductory knowledge regarding the components of tribology i.e. friction, wear and lubrication. Further, the introduction to the biolubricant derived from vegetable oil and animal oil was provided. This chapter shows the progress of biolubricant production and its applications in various fields where it can act as an alternative to mineral oils.

CHAPTER 2: LITERATURE REVIEW

The preceding chapter provided an introduction to Tribology and Biolubricants and its production, along with the application. This chapter provides summary of preceding research pertinent to this thesis. The literature review addresses the key areas of tribology i.e. Friction, Wear and Lubrication. Further, the literature regarding extraction of vegetable oil and its conversion into biolubricant along with the chemistry concepts of conversion was reviewed. The literature of tribological performance of biolubricants was reviewed in three parts:

1. Pure vegetable oil biolubricant
2. Vegetable oil biolubricant blended with other oil
3. Vegetable oil Biolubricant blended with additives

2.1 Friction

Friction is a force that resists the motion of one surface sliding against another. The idea of friction was initially described by Guillaume Amontons (1663–1705), who observed that the frictional force increases proportionally with the normal force but is not influenced by the size of the contact area [20]. The relationship was expressed by equation 1.

$$F = \mu N \tag{1}$$

Where, $\boldsymbol{F}$ is the frictional force, $\boldsymbol{\mu}$ is the coefficient of friction, and $\boldsymbol{N}$ is the normal load.

Later, researchers Bowden and Tabor [21] has developed a more detailed model for metallic friction. They suggested that frictional force comprises two main components: the adhesive force (F_s) and the ploughing force (F_p). The relation between components was shown by equation 2.

$$F = F_s + F_p \tag{2}$$

The adhesive force plays a key role in breaking the bonds that form at points of contact during the relative movement of two surfaces. In contrast, the ploughing force is required for the asperities, on the harder surface to press into the softer one. According to Bowden and Tabor [21], true contact between surfaces only happens at these asperities, referred to as the "real contact area". The actual area of contact is quite small and remains unaffected by the apparent contact area, though it is proportional to the applied load, accommodating the deformation of asperities. Equation 3 shows the formulated friction force.

$$F = A_r S + A' p \tag{3}$$

The real contact area, denoted as $\boldsymbol{A_r}$, represents the actual area where the surfaces interact. The parameter ***S*** refers to the shearing strength, while ***A'*** is the cross sectional area of the ploughing track. The term ***p*** signifies the pressure necessary to induce plastic deformation in the softer metal. As the load is enforced, the asperities of the softer material deform within the contact zone until the true contact area expands enough to adequately bear the load.

Using, $N=p.A_r$, the equation 3 can be written as:

$$F = \frac{NS}{p} + A'p \tag{4}$$

The coefficient of friction is given by the equation 5

$$\mu = \frac{F}{N} = \frac{S}{p} + \frac{A'p}{N} \tag{5}$$

2.2 Wear

Wear is the process of gradual loss of material from contact surfaces due to relative between them. Equation 6 presents the Archard wear equation which relates wear rate to normal load.

$$W_v = \frac{KN}{H} \tag{6}$$

Where, $\boldsymbol{W_v}$ represents the volume of material removed from surface per unit of sliding distance (m^3/m), ***K*** is the wear coefficient, ***N*** denotes the applied normal load, and ***H*** stands for the material's indentation hardness (N/m^2). Archard equation assumes a linear correlation among the wear, normal load, sliding distance and material hardness [22].

The general wear behaviour of materials in sliding contact is shown by the Wear Curve as depicted in Figure 2. A wear curve represents the progression of material volumetric wear over time or usage, showing how a component's durability change as it undergoes friction & stress. Wear curve has three phases, namely: run-in phase/break-in phase/wear-in, steady state phase and accelerated wear/failure phase/wear-out. The initial stage is called the run-in or break-in phase where the wear between the sliding surfaces occurs rapidly as the surface asperities interacts with each other and consequently breaks off leading to the smooth contact between the surfaces. After the initial break-in phase, the wear rate stabilises, resulting slower and consistent wearing of material over the time. This phase is known as the steady-state phase. This phase represents the usable life span of a component as the performance and wear remain relatively predictable. The third phase is the failure phase or accelerated wear phase. In this phase, the material approaches to a point where wear rate accelerates significantly. This phase is caused by the material fatigue, degradation or the breakdown of the protective coatings, which leads to increased friction and wear. The failure phase is the indication of end of usable life of a component. The wear rate accelerates and the structural integrity of a component may be compromised which leads to the failure of a component [23].

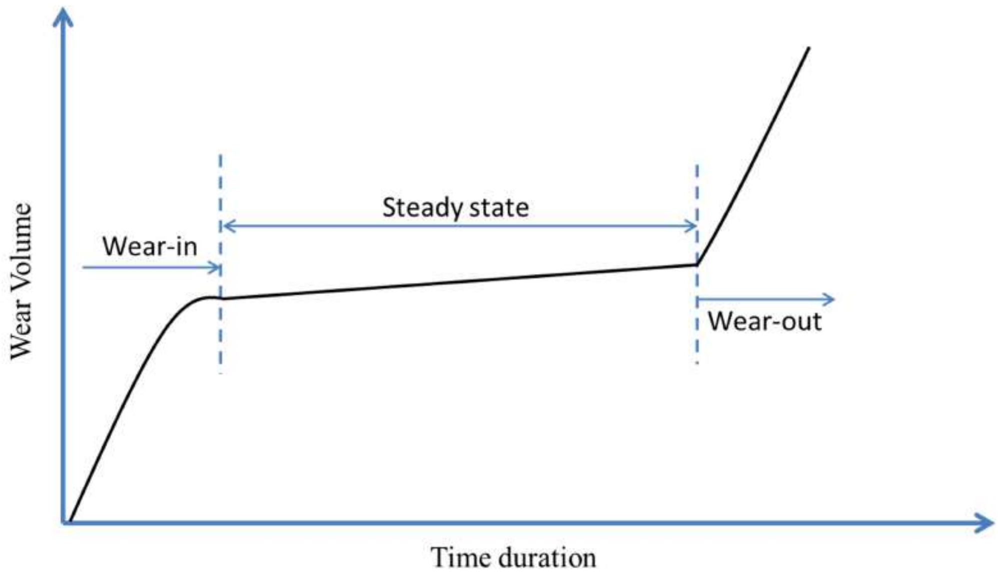


Figure 2: Standard Wear Curve [23]

2.3 Wear mechanisms

Wear occurs when two solid surfaces in relative motion are in contact and the contact surfaces lack adequate protection. The wear mechanisms explain the process of surface wearing. The four primary types of wear mechanisms are adhesive wear, abrasive wear, surface fatigue wear, and chemical wear.

2.3.1 Adhesive wear

Adhesive wear arises when material is removed through sliding contact, as surface asperities interact under a normal load. Adhesive wear process begins when micro-welding occurs at the contact points of the asperities, facilitated by the generation of sufficient heat. The shearing of micro-welds at the asperities is followed by the welding process. Adhesive wear happens when a fracture occurs beneath the surface of one of the materials involved. A key characteristic of this type of wear is the formation of transfer films, where material moves from one surface to another and later detaches, forming wear particles. Figure 3 shows the mechanism of adhesive wear [24].

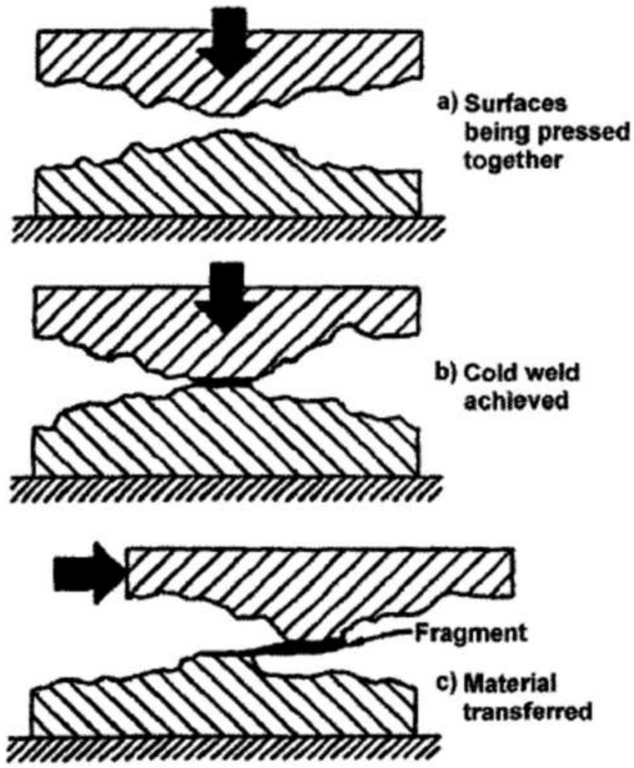
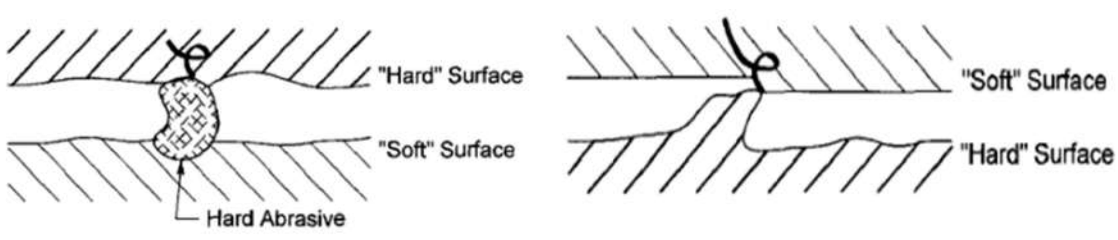


Figure 3: Mechanism of Adhesive Wear [24]

2.3.2 Abrasive wear

Abrasive wear arises when hard particles, called wear debris, or hard surface projections, known as asperities, slide against one another on contact surfaces. When wear results from hard particles, it is classified as three-body abrasion. Conversely, two-body abrasion takes place when harder asperities penetrate a softer material. In both scenarios, material is removed through processes like ploughing or micro-cutting, influenced by factors including the size and shape of the particles and the hardness of the materials involved. Figure 4 shows the mechanism of abrasive wear [24].



(a) (b)

Figure 4: Mechanism of Abrasive wear, (a) Three-body wear, (b) Two-body wear [24]

2.3.3 Fatigue wear

When two or more surfaces come into contact, particularly at points where rough edges or peaks, known as asperities, meet, high-stress concentrations often arise. This interaction leads to the deformation of asperities on one surface by those on the opposing surface. With repeated cycles of contact, cracks start to develop. As these cycles continue, the cracks expand, eventually causing small particles to detach from the material. This particle detachment occurs due to cracks that propagate through fatigue, a process termed "fatigue wear." The strain on the worn surfaces is substantial, typically resulting in plastic deformation that alters the material's microstructure and impacts the wear process. Figure 5 shows the mechanism of fatigue wear.

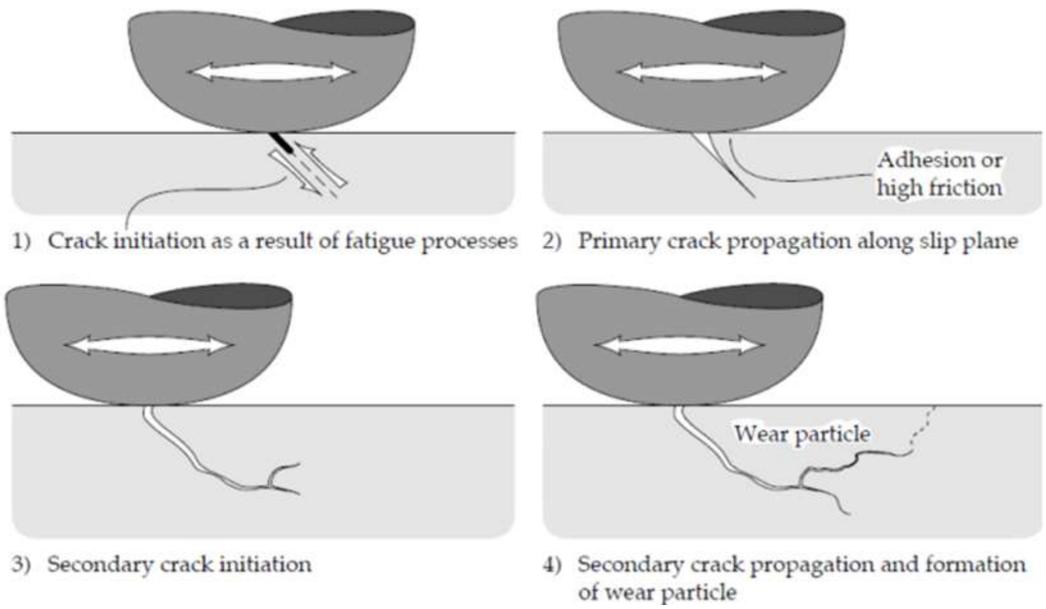


Figure 5: Stepwise mechanism of Fatigue wear [25]

2.3.4 Chemical wear

Chemical wear, also referred to as corrosive wear, occurs when surfaces in motion come into contact within a corrosive environment, leading to degradation. This wear type arises from chemical or electrochemical interactions between metal surfaces and substances like lubricants or corrosive contaminants, including salts, water, and acids. The chemical wear often results in pitting on the impacted surfaces. When chemical wear occurs in the presence of air, it is frequently termed oxidative wear due to oxygen acting as the primary corrosive agent. Figure 6 shows the mechanism of chemical wear [26].

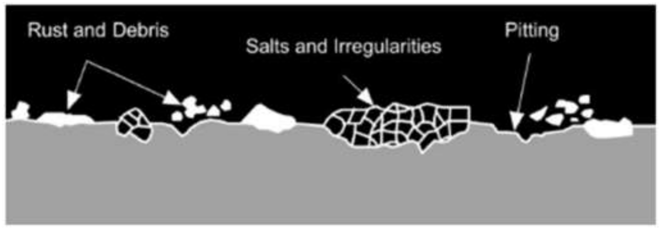


Figure 6: Mechanism of chemical wear [26]

2.4 Lubrication

Lubrication contributes in reducing friction and minimizing wear between two contacting surfaces. Lubrication is achieved by forming a layer of lubricant with low shear strength, allowing the surfaces to move against each other with significantly less resistance than if no lubrication were present. An effective lubricant supports efficient heat transfer, removes contaminants and helps in preventing corrosion along with reducing friction. Lubricants are available in both solid and liquid forms: solid lubricants, such as graphite, usually exist as powders, whereas liquid lubricants are typically composed of base oils enriched with additives to enhance performance [25].

Lubricants' primarily function is to create a protective layer over the two contacting surfaces that diminish friction and limits wear. The effectiveness of lubrication, however, depends on the normal load applied, which leads to different lubrication states. These states are typically divided into distinct lubrication regimes, with the most common being hydrodynamic, mixed, and boundary lubrication. The Stribeck curve, shown in Figure 7, shows the representation of these regimes, showing the relationship between the coefficient of friction and the ratio of lubricant viscosity (η), rotational speed (N_s), and load per unit bearing area (P) [27].

In boundary lubrication, the lubricant film is even thinner, usually around 1 to 3 nanometers, which is less than the height of some surface asperities, leading to significant asperity contact. This regime arises when speeds decrease or loads increase, resulting in more intense contact conditions compared to other lubrication regimes.

Mixed lubrication occurs in conditions such as lower speeds, increased loads, or higher temperatures, which considerably reduce the lubricant's viscosity. In this regime, surface asperities or peaks of roughness, may intermittently contact in certain areas [28].

In hydrodynamic lubrication, the sliding surfaces are completely moved apart by a lubricant film that is thicker than 0.25 µm. This prevents any direct contact between the metal surfaces. Under such conditions, the friction coefficient is primarily influenced by the viscosity of the lubricant, provided that the load and speed remain constant. A specialized form of this lubrication, called elasto-hydrodynamic lubrication, occurs when the applied load is high enough to induce elastic deformation in the surfaces. The resulting lubricant film in this scenario is generally much thinner, ranging from approximately 0.025 to 5 µm, compared to that found in standard hydrodynamic lubrication [27].

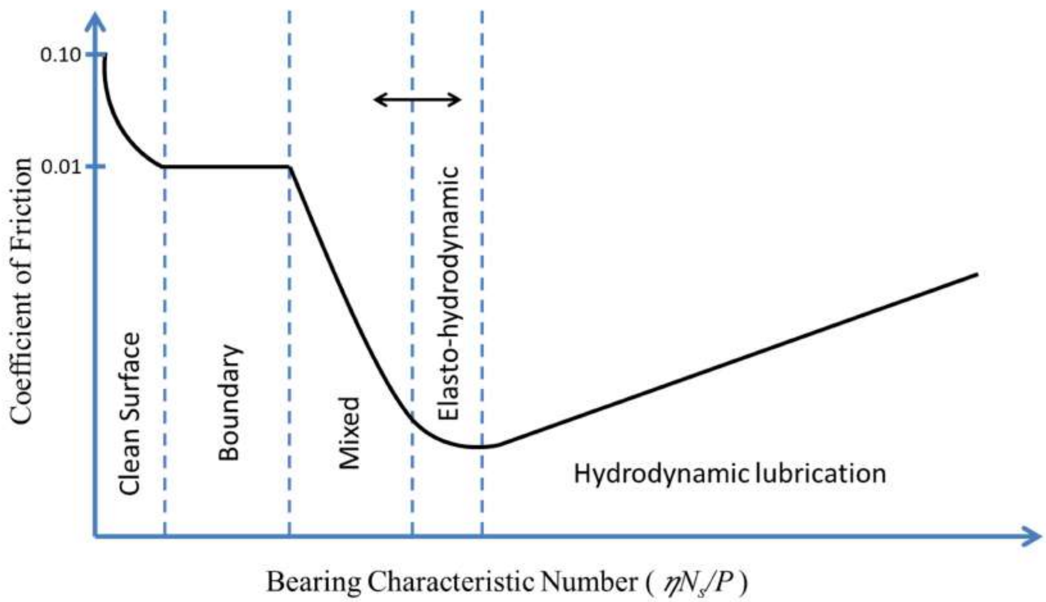
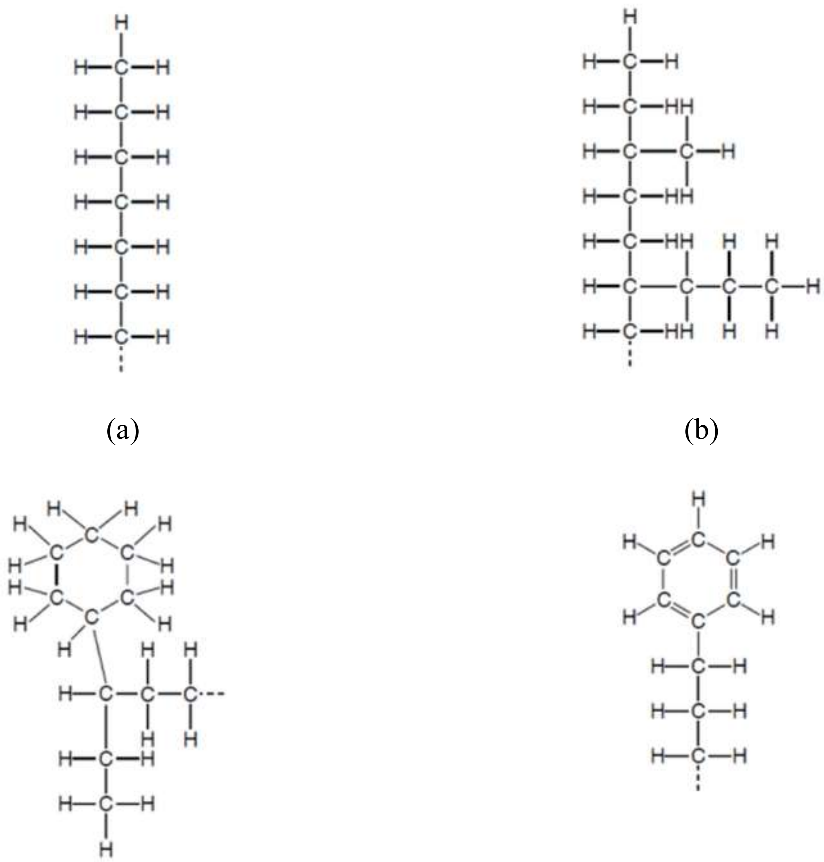


Figure 7: Stribeck Curve representing different lubrication regimes [27].

Lubricants are essential substances designed to diminish friction, wear, and heat between moving solid surfaces. Through a protective layer, lubricants allow smoother interactions between components, which help machines, operate more efficiently and with less damage over time. They also play a critical role in preventing corrosion and rust, enhancing the longevity of parts, and maintaining overall machinery performance. The lubricants are mainly composed of mineral oils and vegetable oils.

Mineral oils are created through crude oil refining and they are widely used as lubricants in industrial applications like engines and turbines. According to the chemical structures, mineral oils are classified into three types: paraffinic oils, having straight and branched hydrocarbons; naphthenic oils, made up of cyclic carbon molecules; and aromatic oils, which contain benzene-like compounds. The types of mineral oil are illustrated in Figure 8. Differences in molecular structures among these oils influence their physical properties, such as the variations in viscosity-temperature behavior between paraffinic and naphthenic oils [25]



(a) (b)

(c) (d)

Figure 8: Different types of mineral oils, (a) Straight-chain paraffin, (b) Branched-chained paraffin, (c) Naphthene, and (d) Aromatic. [25]

Engine oils formulated from mineral bases are typically blended with various additives. These additives, which are synthetic compounds, are included to improve or introduce specific properties to the base oil. In lubricants, additives generally constitute between 1% and 25% of the total composition [29]. The primary types of additives utilized in lubricating oils are summarized in the Table 2 below.

Vegetable oils are extracted from plants seeds, like of Jatropha, sunflower and mustard. Most of the vegetable oil are edible in nature and are essential for food preparation, often serving as a medium for cooking and frying. While some vegetable oils, like karanja oil, jojoba oil and jatropha oil, contain toxic elements that make them unsuitable for human use [30].

Vegetable oil contains glycerol and fatty acids which form compounds known as triglycerides. A triglyceride molecule contains one glycerol molecule, which is an organic alcohol, bonded to three fatty acids. The structure of triglycerides is shown in Figure 9.

Table 2 : Types of lubricant additives [31]

Additives	Purpose	Example
Anti-oxidants	To delay the oil ageing	Zinc dialkyl dithiophosphates (ZDDP)
Viscosity Modifiers	To obtain desired viscosity index	Polyalkylmethacrylates
Anti-foam Agents	To avert lubricant foam	Polydimethylsiloxanes
Anti-wear and Extreme Pressure Additives	For wear reduction	ZDDP
Friction Modifiers	For reduction of coefficient of friction	Molybdenum di-sulfide
Corrosion Inhibitors	For protection of metal surface from corrosion	Petroleum sulfonates
Pour-point Depressants	For enabling lubricant to flow at low temperatures	Polymethacrylates

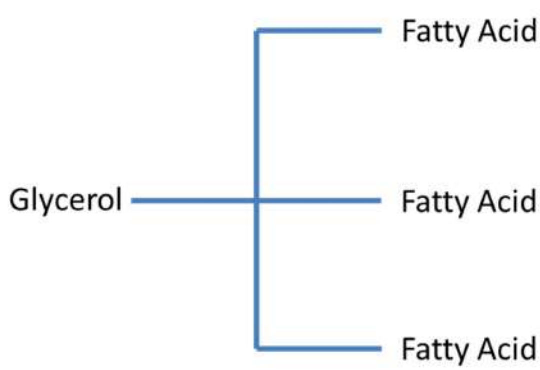
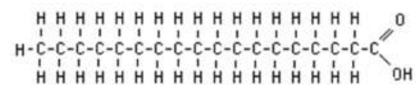
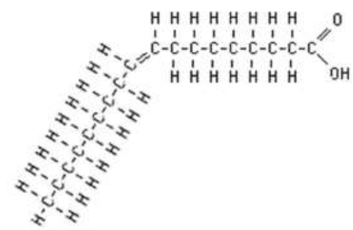
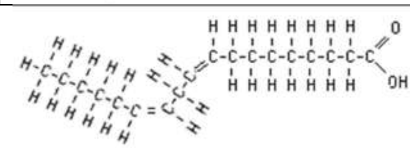


Figure 9: Structure of Triglycerides.

Fatty acids are grouped as two categories: saturated and unsaturated, depending on the presence and quantity of C=C bonds in its carbon chains. Saturated fatty acids have straight carbon chains with zero C=C double bonds, whereas unsaturated fatty acids contain one or more C=C double bonds. Unsaturated fatty acids are further categorised into mono-unsaturated and poly-unsaturated types.

Mono-unsaturated fatty acids, such as oleic acid, have a single C=C double bond, while poly-unsaturated fatty acids, like linoleic and linolenic acids, feature two or more double bonds. This structural difference gives saturated fatty acids a linear form, while unsaturated fatty acids adopt a bent configuration due to the presence of double bonds [32]. Table 3 represents the classification of Fatty Acids.

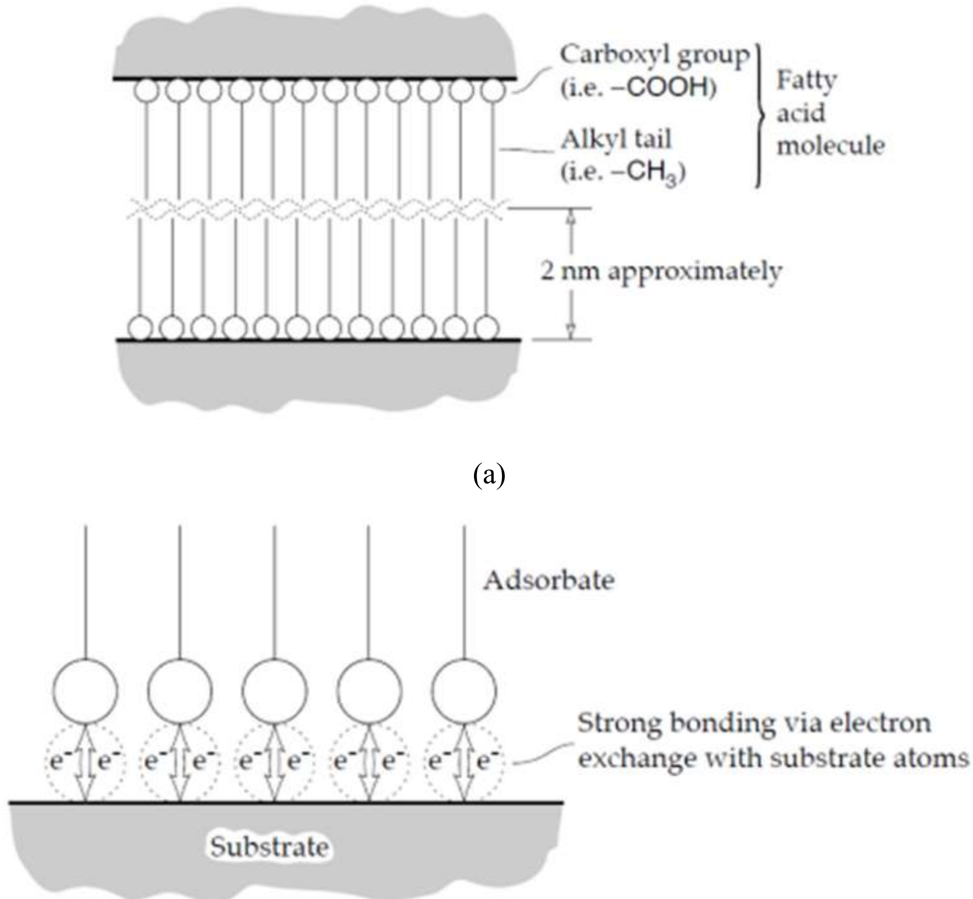
Table 3: Classification of Fatty Acids [32]

Fatty acids group	**Number of C=C in HC chain**	**Example**	**Molecular structure**
Saturated	0	Stearic acid	
Mono-unsaturated	1	Oleic acid	
Poly-unsaturated	2	Linoleic acid	

Fatty acids are polar organic compounds that contain a carboxyl group (COOH) within their molecular chains. The polarity causes the carboxyl end to interact with metal surfaces, while the alkyl group at the other end is repelled. This behavior, referred to as physical adsorption or physisorption, is driven by intermolecular forces such as van der Waals interactions, resulting in a single-molecule layer that helps reduce surface friction. Alternatively, in chemical adsorption, or chemisorption, a chemical bond forms between the carboxyl group and the metal surface via electron exchange. The mechanism of physisorption and chemisorption is shown in Figure 10 [33, 34].

2.5 Extraction of vegetable oil

Oil extraction is essential in food and crop processing, aiming to retrieve valuable substances from raw materials. Mariana et al. [35] discuss the long history of oil extraction techniques from oilseeds, with a focus on maximizing oil yield and minimizing costs. The concentration of vegetable oils varies across different plant tissues, including seeds, pulp, stone fruits, tubers, and sprouts. Oils are derived from a variety of plants, including oil-rich seeds like sunflower, soybean, and rapeseed; oily fruits such as olives, coconuts, and palms; oil-rich tubers like peanuts; and oil-rich germ like corn. Dyer et al. [36] identify prime oil-producing crops, including palm, soybean, canola, sunflower, linseed, coconut.



(b)

Figure 10: Mechanism of (a) Physisorption, (b) Chemisorption [34]

Alenyorege et al. [37] identify three crucial factors for assessing the performance of a oil extraction process:

1. Extraction Yield: This represents the quantity of oil extracted from a defined amount of oleaginous material.
2. Extraction Efficiency: This is the proportion of the extracted oil relative to the total oil content in the material of oleaginous nature.
3. Extraction Loss: This reflects the weight of the remaining waste after extraction, which includes oil residues or leftover cake.

Through years of research, oil extraction methods have seen significant technological advancements. The common aim of all the oil extractions methods was to optimise the process and achieve high-quality oil. The primary oil extraction methods are discussed below.

2.5.1 Traditional Hot water floatation method

The Hot water flotation method is also known as the aqueous extraction or wet extraction. It is a process of extracting vegetable oil from oilseeds in a small scale. This method used significant amount of water causing oil to separate and float on to the surface. According to Head et al. [38], the procedure starts with heating and grinding the oilseed kernels, which are then mixed with boiling water and simmered for a minimum of 30 minutes. This causes the oil to move from the solid seed form to the liquid water form, allowing it to float on the surface of water. During the boiling process, additional water is added to compensate for evaporation and assist the oil in floating upwards. The oil is then carefully skimmed off using a shallow container, and any remaining moisture is eliminated through evaporation.

The Hot water flotation method is a cost-effective process since it doesn't rely on chemicals, hence making it an environmentally friendly option. Hot water flotation method is labour-intensive, time-consuming process, and it results in a lower yield of extracted oil [35].

2.5.2 Mechanical extraction method

The mechanical extraction method of vegetable oil involves application of pressure to the processed oilseeds to separate oil. Mechanical extraction method is also called pressing. This method uses compressive forces generated by specialized machines called presses to extract oil from oleaginous or oil-rich materials [39]. During the process, the oil-rich material is placed in-betweens the perforated barriers, and pressure is applied by reducing the available volume, which forces the oil out. Mechanical extraction is often used in smaller-capacity facilities, for specialty products, or as an initial step before solvent extraction in larger operations [40]. The benefits of mechanical extraction method are its low cost and the purity of the extracted oil, which is free from contaminants. Moreover, mechanical extraction is preferred for its low environmental and health risks, as it avoids hazardous solvents like n-hexane. The two main types of mechanical extraction methods are the screw press and hydraulic press.

A screw press, also called an extruder or expeller, extracts oil by using a rotating helical screw, or worm, within a confined press chamber. This equipment includes a feed inlet, a horizontal barrel that contains the screw, perforations for collecting the oil, and an outlet for the expeller's processed material, known as cake. The screw design gradually increases in diameter, creating maximum pressure in the exudation zone to force the oil out. Seeds are fed into the press continuously, where the screw crushes, grinds, and extracts the oil, generating considerable heat from the friction [41].

In the study by Adetola et.al. [42], the palm oil was extracted using the screw pressing method. The processed palm fruit was fed into the screw press via a hopper where it was transported through a cylinder by a worm shaft. The pressure generated between the worm and cylinder forces the palm oil through the mesh. The extracted palm oil was then flows into a tray via an oil channel for collection while the remaining cake was expelled through an outlet. Figure 11 shows the setup of a screw pressing machine for oil extraction.

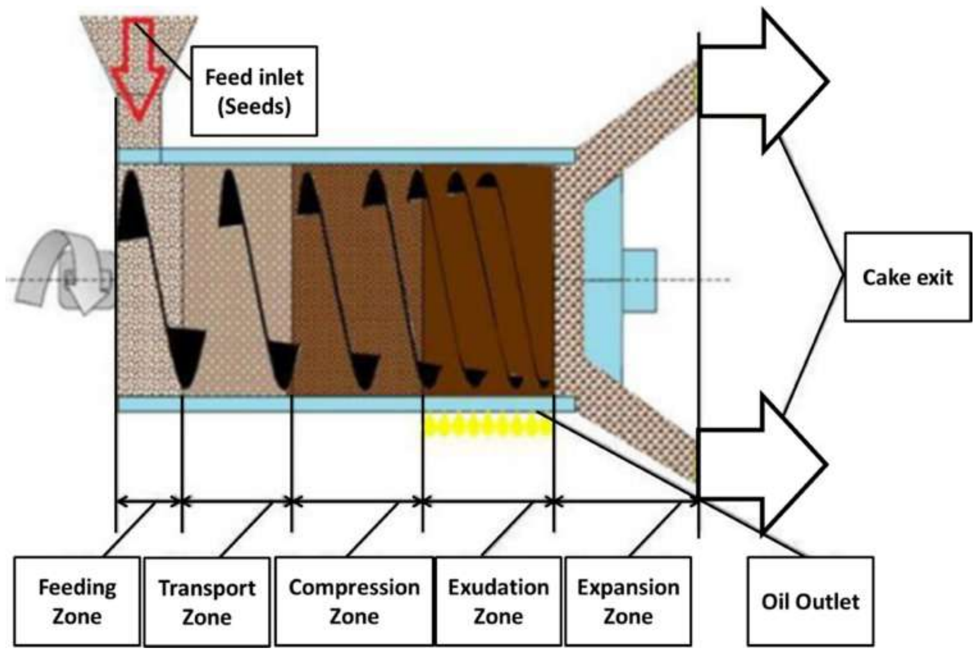


Figure 11: Setup of Screw pressing machine for oil extraction [42]

Hydraulic presses are machines designed to apply force in one direction using fluid pressure. According to Mariana et al. [39], hydraulic oil extraction involves compressing a mash of oil-bearing material within a cylindrical chamber using a ram. This compression causes the material to compact along its length, while the oil flows outward through perforations along the chamber's sides. The process flow of oil extraction using a hydraulic press is shown in Figure 12. Unlike screw presses, the hydraulic presses for oil extraction operate in batches, and oil yield is slightly less due to their discontinuous operation.

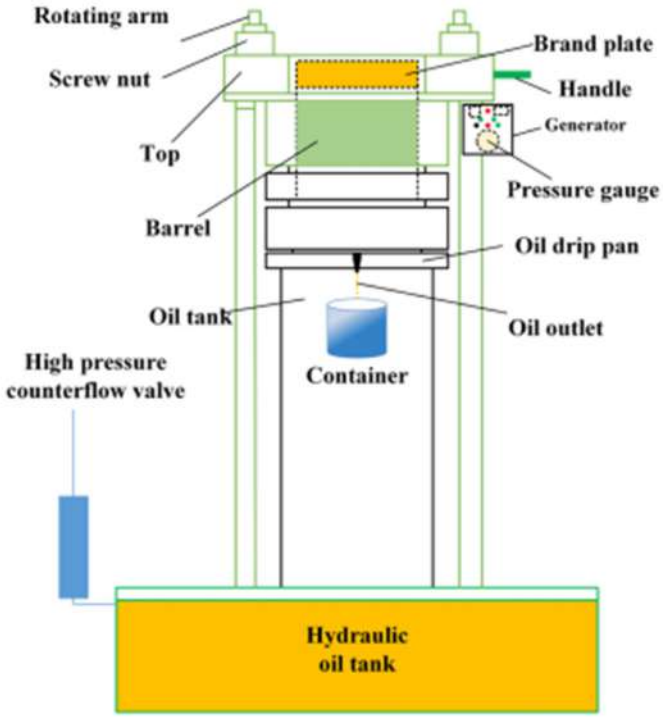


Figure 12: Hydraulic process for oil extraction [43]

Despite some limitations, hydraulic presses remain valuable for processing materials that need careful handling, such as cocoa butter [44]. The hydraulic presses have been utilized for oil extraction purposes. Sabarish et al. [45] investigated oil extraction from rubber seeds using a hydraulic press and examined the kinetics of the acid esterification process. The studies conducted by Santaso et al. [46] and Oscar et.al. [47] had compared the mechanical extraction method with solvent extraction method, which observed that mechanical extraction method offered benefits such as oil purity, simplicity of the process, and cost-effectiveness. Kehinde's [48] research on coconut oil extraction with a uniaxial hydraulic press identified key factors affecting oil yield, such as moisture, pressure applied, duration of pressure applied, time of heating, and sample temperature.

2.5.3 Solvent extraction method

In a study, Adepoju et al. [49] defines Solvent extraction is a chemical method for oil extraction which make use of solvents to separate oil from a liquid-solid mixture. In this process, a solvent is added to the mixture, causing the oil to transfer from the solid material into the solvent. The solid material absorbs some of the solvent until equilibrium is achieved.

Solvent extraction can be performed in batch processes, such as Soxhlet and fixed column extraction, or in continuous processes, including counter-current extraction.

Avram et al. [50] noted that the efficiency of solvent-based oil extraction is influenced by several factors including moisture content, solid material thickness and surface area, temperature, and residence time. Solvent extraction process has achieved up to 99% extraction efficiency in commercial settings. Adepoju et al. [49] had listed additional benefits of solvent extraction method including cost-effectiveness, straight-forward operation, and fast processing times.

Solvent extraction method has some drawbacks. In a study Mariana et al. [39], highlighted that many chemical solvents pose risks to human health, and the resulting extracted oil has lower quality compared to mechanically pressed oil, hence making it unsuitable method of oil extraction. Additionally, the chemical solvents can be flammable, hence creating the risks of fire or explosion. The solvent extraction method demands significant capital and energy resources.

Research on solvent extraction from certain crops is extensive. Mani et al. [51] examined the efficiency of various solvents, including hexane, petroleum ether, and acetone, for extracting moringa seed oil while optimizing conditions such as particle size, extraction temperature, and extraction time. Adepoju et al. [49] optimized sour-sop oilseed extraction using a Box-Behnken design, studying the oil's physicochemical properties and fatty acid composition. Avram et al. [50] created a bench-scale experimental setup to analyze solvent extraction in crops like rapeseed, soybean, and sunflower through a percolating process.

2.5.4 Supercritical fluid extraction method

Supercritical fluid extraction (SFE) method utilizes a supercritical fluid as a solvent to separate desired oil from oil-rich materials. At the supercritical state i.e. above critical temperature and pressure, a fluid seamlessly shift between liquid and vapour phases without a distinct phase change.

SFE offers distinct benefits over traditional solvent-based extraction methods, such as improved mass transfer capabilities and the ability to easily modify solubility by adjusting factors like pressure, temperature, or by adding polar modifiers. This technique often yields high-quality oils that typically require little to no further refining, unlike oils obtained via conventional solvent extraction methods. Xu et al. [52] noted that carbon dioxide is the primary solvent used in SFE for oil extraction, with critical conditions of 30.9°C and 7.28 MPa.

The SFE method has gained notable research attention in vegetable oil extraction. For instance, Honarvar et al. [53] conducted mathematical modeling on SFE applied to oil extraction from canola and sesame seeds, providing an in-depth explanation of the process. The setup includes a gas tank with a condenser to maintain the CO_2 in liquid form. A pump then pressurizes the liquid CO_2 to the required levels. The sample material is placed in a high-pressure vessel, along with ethanol as a modifier and glass beads to prevent channeling of the flow. The pressurized CO_2 passes through a surge tank and enters the packed bed vessel. During extraction, the solvent flows through the vessel via a back pressure valve, which is electrically heated to prevent freezing. The extracted oil is then collected at the end. Figure 13 shows the schematic diagram of the SFE oil extraction process.

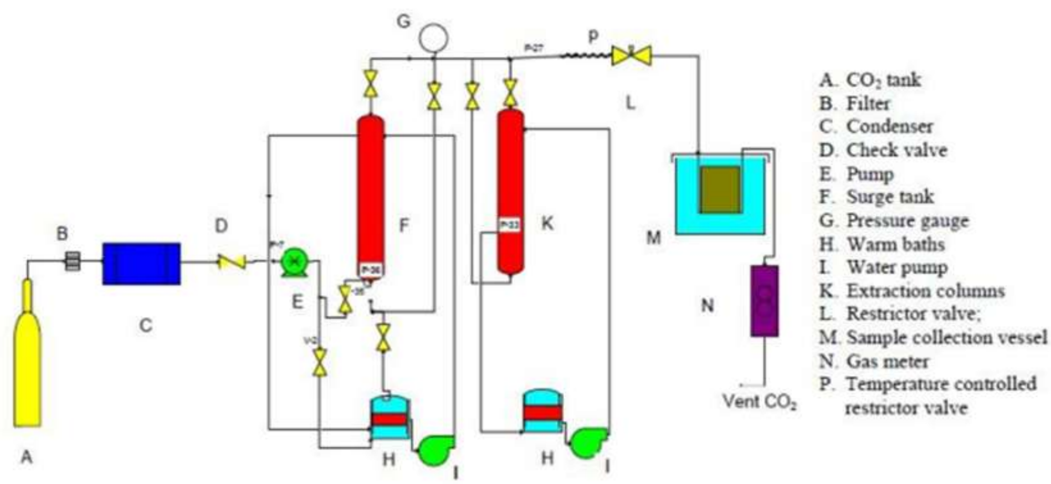


Figure 13: Schematic diagram of the SFE oil extraction process. [53]

2.5.5 Steam distillation method

Steam distillation is a method commonly used to extract plant oils. This approach works well because essential oils are volatile, meaning they readily evaporate with steam, and are hydrophobic, allowing them to separate from water during condensation. A standard steam distillation setup generally includes a heat source, boiler, biomass chamber, still head, condenser, and receiver.

In this process, distilled water is heated in the boiler to produce steam, which then rises into the biomass chamber, capturing both oils and water-soluble components from the plant material. The vaporized mixture moves through still head, cools in the condenser, and is finally collected in the receiver. Here, the oil naturally separates from the water. The products from steam distillation are a concentrated essential oil and a water-based byproduct called hydrosol [54].

2.6 Production of biolubricants from vegetable oils

Vegetable oils require chemical modification before being used as lubricants because the natural properties of vegetable oil are not suited for it being a lubricant. The vegetable oils have poor oxidation stability, hydrolytic instability and inadequate thermal stability. The vegetable oils have to modify through various chemical methods to improve its properties and to convert a vegetable oil into a suitable biolubricant. The primary chemical techniques used to alter vegetable oils include transesterification, esterification, epoxidation, hydrogenation, and estolide formation [55]. Figure 14 illustrates the various chemical processes employed to modify vegetable oils for the production of biolubricants.

Many of the researchers utilise Transesterification reactions to modify vegetable oils into biolubricants. The transesterification method has several advantages over other methods of biolubricant production. It has been observed that transesterification reactions produce biolubricants with superior chemical and physical properties which includes thermal stability, viscosity and oxidation resistance [56]. Transesterification process results in high conversion rate of raw vegetable oil into desired biolubricant which leads to high yields and reduced impurities compared to direct esterification [57]. Transesterification reactions can be applied to numerous options of feedstock including vegetable oils, animal fats and used cooking oils, making it a flexible and sustainable option [58]. Biolubricants produced through transesterification complies with international standards for engine oils which ensure better compatibility and performance in industrial applications.

In transesterification reactions, the glycerol component of triacylglycerides is substituted with a long chained alcohol. On the other hand, esterification reactions occur when the free fatty acids (FFAs) in natural oils react with long-chain alcohols to form corresponding esters [58]. In a study, Aziz et.al. [59] uses palm oil methyl ester (POME) as raw materials to produce a biolubricant. The research utilises a tranesterification reaction with sodium methoxide as a catalyst in different concentration such as 0.5 wt%, 0.75 wt%, 1.0 wt%, 1.25 wt% and 1.5 wt%. The experiments were performed by reacting POME with pentaerythritol (PEE) under stirring speed of 700RPM. The catalyst was then added to the mixture and allowed to react for a specific time duration. The formation of biolubricants was confirmed by detecting the reaction products pentaerythritol tetraoleate (PETO) and trimethylolpropane ester (TMPE), which result from the interaction of POME and PEE. The optimum condition for the transesterification process were found at temperature of 431.15 K, a catalyst concentration of 1.19%, molar ratio of 4.5:1, and reaction time of 01 hour. The oil yield under optimum conditions was 37.56%.

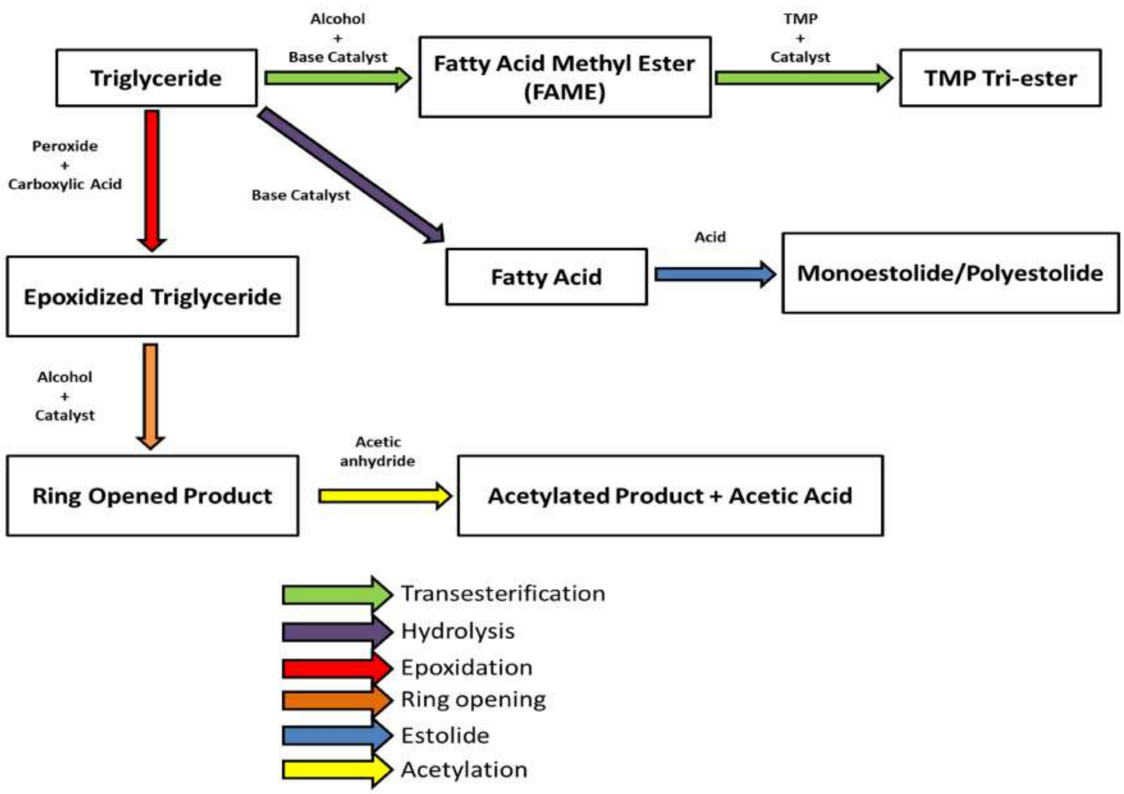


Figure 14: Chemical methods of modifying vegetable oils to produce biolubricants

In a study, Zulkifi et.al. [60] tested the lubricity of biolubricants derived from trimethylopropane (TMP) and pentaerythritol ester (PE) synthesied from transesterification of POME. The reaction included sodium methoxide ($NaOCH_3$) as a catalyst. The result of the test shows the low friction values for the synthesised biolubricant indicating the suitability of biolubricant for application in vehicle engine.

Koh et al. [61] investigated the synthesis of biolubricants using palm methyl ester (PME) and trimethylolpropane (TMP) with sodium methoxide ($NaOCH_3$) as the catalyst. The transesterification reaction was carried out in an oscillatory flow reactor at temperatures between 110°C and 150°C. The most favorable reaction conditions were determined to be 140°C for duration of 25 minutes. Under these optimal conditions, the biolubricant yield achieved was 94.6%, with diesters accounting for 14.7% and triesters for 79.9%.

Reeves et al. [62] conducted a study exploring how the composition of fatty acids affects the viscosity index of several seed oils, such as avocado, canola, corn, olive, peanut, safflower, sesame, and soy. Their results showed the fatty acid profile plays a critical role in determination of viscosity of these oils, with peanut oil displaying the highest viscosity at 70.24 cP. In a related study, Zubaidah et al. [63] explored the use of palm oil derivatives, specifically trimethylolpropane, as raw materials for producing bio-lubricants. This production process employed a vacuum reactor and a transesterification reaction, achieving a maximum conversion rate of 66% under optimal conditions of temperature 120°C with reaction time of over 2 hours. Furthermore, Syaima et al. [64] developed bio-lubricants using palm oil mill effluent through enzymatic hydrolysis and esterification processes. The study determined the ideal reaction conditions to be a temperature of 40°C, pH 7, a stirring speed of 650 rpm, an enzyme concentration of 20 U/ml, and a POME composition of 50% (v/v).

2.7 Tribological performance of vegetable oil based Biolubricants

Numerous research studies have explored the tribological properties of biolubricants derived from vegetable oils. These studies categorize vegetable oil based biolubricants into three distinct types, depending on the type of oil used:

a. Pure vegetable oil biolubricant
b. Vegetable oil biolubricant blended with other oil
c. Vegetable oil Biolubricant blended with additives

2.7.1 Tribological performance of pure vegetable oil biolubricant.

The suitability of vegetable oil based biolubricant is assessed in its pure form. This approach is worthy of careful attention because the base oils have substantial effect on determining the tribo-chemical characteristics of fluid lubricants. Numerous researches have investigated the tribological performance of several pure vegetable oils, including coconut, safflower, corn, palm, and soyabean.

In a study, Gerbig et.al. [65] examined the tribological properties of sixteen types of commercially available vegetable oils, mineral lubricants, and synthetic esters, utilizing a reciprocating tribometer with ball-on-disc configuration. The experiment simulated two wear mechanisms: adhesive wear—using AISI 52100 steel for both the ball and disc—and abrasive wear, in which alumina (Al_2O_3) served as the disc material. Results indicated that under adhesive wear conditions, linseed, olive, walnut, and safflower oils provided a consistent and low coefficient of friction i.e. approximately 0.11. However, other oils presented more erratic friction patterns. Olive and safflower oils produced the lowest friction but demonstrated minimal wear resistance. Overall, vegetable oils performed poorly compared to mineral oils and synthetic esters in both friction control and wear resistance. Under the state of abrasive wear, all tested oils uniformly low friction (COF ranging from 0.11 to 0.13), though sesame and castor oils were more resistant to abrasive wear. The study concluded that the performance of vegetable oils in tribological applications varies considerably based on the specific tribosystem, including material and contact conditions.

Reeves et.al. [62] carried out a comparative study to investigate the control of fatty acids on the tribological properties of various vegetable oils. This investigation utilized a pin-on-disc test under controlled conditions to assess the aftermath of fatty acid composition on friction and wear across eight types of vegetable oil. The unsaturation number (UN) stand for the average number of double bonds in the fatty acids of oil. The results showed that avocado oil, with the lowest unsaturation number, exhibited superior tribological performance, achieving the smallest coefficient of friction and wear volume among the oils examined. On the other hand, soybean oil, with a higher unsaturation number, demonstrated higher coefficient of friction and wear. The study concluded that reducing friction and wear is closely tied to the formation of a monolayer that minimizes direct metal-to-metal contact, primarily facilitated by saturated and mono-unsaturated fatty acids. Conversely, a higher presence of double bonds, such as those in linoleic acid from soybean oil, weakened the density of this protective monolayer.

The hypothesis proposed by Reeves et al. regarding fatty acids' role in forming monolayers was further validated by experiments conducted by Fox et al. [66]. In the study, Fox et.al. had examined both saturated and unsaturated fatty acids in sunflower oil using a ball-on-plate test rig. The outcomes revealed that both wear and friction tended to rise as the degree of unsaturation in the fatty acids escalated. Further, Fox et al. [66] suggested that the linear structure of stearic acid, which lacks double bonds, enables it to pack tightly on the surface, creating a dense and resilient protective layer. Conversely, the double bonds in oleic and linoleic acids cause kinks in the chains, reducing their packing efficiency and weakening the protective layer.

Lundgren et.al. [67] highlighted that the explanations provided by Reeves et.al. and Fox et.al. are inconsistent with the adsorption test results of unsaturated fatty acids on steel. The study showed that the quantity of unsaturated fatty acids adsorbed on steel surfaces rose with increasing levels of unsaturation when using the spreading solvents hexadecane and heptamethylnonane. Also, the researcher used a surface force apparatus to investigate how unsaturation in fatty acids affects friction on mica surfaces. Their findings showed that the friction coefficient increased with greater unsaturation, aligning with the distinct load regimes observed for linoleic acid.

In a study, Jagadeesh et al. [68] used a four-ball tribotester to scrutinise the performance of five vegetable oils and their oxidized counterparts under boundary lubrication conditions. The results showed that as both aging time and temperature was increased, the peroxide values, fatty acid content, and wear scar diameter was increased. The oxidation process caused a rise in free fatty acids (FFAs), which facilitated the setting up of low-shear-strength metallic soaps, resulting in a lower friction coefficient for the oxidized oils.

Palm oil (PO) and soybean oil (SBO) are the two most commonly used vegetable oils worldwide. Palm oil is higher in saturated fatty acids, which feature carbon chains with zero double bonds, making them less prone to oxidation. This gives Palm oil remarkable stability against oxidation, especially at temperatures around 180°C [69]. In contrast, SBO contains more unsaturated fatty acids with carbon double bonds in their molecular structure, increasing their vulnerability to oxidation [70].

Numerous studies had been conducted to compare the tribological properties of Palm oil and soybean oil on four-ball tribotester. In an experimental study, Jagadeesh et.al. [68] found that at a load of 400 N and an oil temperature of 75°C, Palm oil demonstrated a lower coefficient of friction and exhibited less wear than Soybean oil. However, under reduced loads of 147 N and 392 N, as specified by ASTM D4172, Syahrullail et.al. [71] reported that Palm oil maintain a lower coefficient of friction than soybean oil. However, soybean oil demonstrated better wear resistance under these conditions. Conversely, another study identified that at a higher load of 1236 N, Soybean oil not only had a lower coefficient if friction but also resulted in less wear compared to Palm oil.

Masjuki et al. [72] performed an experiment comparing a plant-based oil (PO) with a mineral oil (MO)-based lubricant using grey cast iron plates for the lower and upper piston rings in a reciprocating test. The experiment was conducted at a low load of 10 N, which equated to a contact pressure of 3.0 MPa, and at room temperature. The findings revealed that the Palm oil resulted in a higher coefficient of friction compared to the MO lubricant and it demonstrated superior wear resistance. Additionally, the research identified abrasive wear as the primary mechanism influencing both piston rings, though the severity of wear differed between them. The lower piston ring exhibited more noticeable grooves, pits, and material transfer, whereas the upper piston ring displayed small cracks.

Zulkifli et.al. [60] inquire into the tribological attribute of a trimethylolpropane ester (TMP) produced from palm oil. The biolubricant was synthesized by transesterifying palm oil methyl ester (POME) with TMP to amplify its thermo-oxidative stability [15]. A four-ball tribometer was employed to assess friction and wear performance under high-pressure conditions. The research compared various proportions of TMP blended with paraffinic oil, revealing that TMP exhibited superior performance in reducing friction and wear compared to paraffinic oil across various load conditions.

Mahmud et.al. [73] studied the outcome of working temperature on tribo properties in the presence of biolubricant based on sunflower oil. The tribological tests were conducted on a four-ball tribo-meter using an uncoated stainless steel ball of 440C grade and Diamond-like-coated (DLC) stainless steel ball. The test temperature was set at 75°C, 85°C and 100°C under the applied load of 390N and speed of 1200RPM. DLC was effective to improve the tribological characteristics. DLC coating is famously known as a solid lubricant. The study showed that the properties of DLC coating fluctuate with the testing temperature. At 100°C, DLC coating reduces the coefficient of friction significantly.

2.7.2 Tribological performance of vegetable oil biolubricant blends with mineral oil

A mixture of oils can be made by combining two or more different types to attain desired performance characteristics. For instance, mixing palm oil with olive oil produces a blend that shares comparable fatty acid profiles and physical-chemical traits, including melting point, viscosity, and iodine value, which vary based on the proportion of each oil in the mixture [74]. The research has shown that commercial mineral engine oil offers better wear resistance, reduced friction, and enhanced oxidation stability compared to vegetable oils [65], [72,75,76]. Hence, mineral oils are generally pricier than pure vegetable oils. By mixing mineral oil with vegetable oil, it is possible to lower the lubricant's cost while also reducing dependency on petroleum sources. The increased wear resistance of mineral oil compared to vegetable oils has sparked to interest in examining its tribological performance when blended with vegetable oils.

The study conducted by Jabal et al. [77] investigates the tribological properties of blends of RBD palm olein and mineral oil (SAE 40) using a four-ball tribotester. The findings reveal that a lubricant composed of 60% RBD palm olein and 40% SAE 40 shows improved performance, characterized by a reduced coefficient of friction and reduced friction torque in respect to a lubricant made entirely from mineral oil. Furthermore, the wear scar diameter (WSD) associated with the RBD palm olein blend is significantly smaller, with a 23% reduction in WSD observed with just a 60% concentration of palm oil relative to the mineral oil lubricant. This suggests that incorporating palm oil may function as an anti-wear additive. Overall, the analysis indicates that RBD palm olein can effectively act as a partial replacement for bio-lubricants, maintaining wear resistance and lubricating efficiency.

Shahabuddin et.al. [78] conducted a comparative tribological analysis of a bio-lubricant derived from Jatropha oil against SAE 40 oil. The oil samples were created by blending Jatropha oil with SAE 40 oil in varying volume ratios from 10% to 40%. The wear rate was observed to vary with the concentration of the bio-lubricants used, with JBL10 showing a wear rate similar to that of the conventional lubricant. Initially, both the wear rate and temperature experienced significant increases. Throughout the duration of the tests, JBL10 biolubricant performed optimally, effectively maintaining its properties related to wear and temperature rise. Elemental analysis post-experiment indicated an increase in iron and aluminum content, likely resulting from the wear of the pin and disc. Conversely, the levels of phosphorus, calcium, and magnesium decreased, possibly because of oxidation and other chemical reactions. Most of the bio-lubricants fulfilled the ISO viscosity grade standards, with the exception of JBL40 and JBL50, which failed to meet the ISO VG 100 specification at 40°C. JBL10 recorded the lowest wear scar diameter (WSD) of 0.35 mm and the lowest coefficient of friction (COF) at 0.045. An increase in the blending ratio corresponded with a rise in WSD for the ball specimens, indicating that higher concentrations may compromise the strength of the lubricant film and elevate the COF. In the Four-ball configuration tests, all bio-lubricants exhibited strong tribological performance, with JBL10 particularly excelling in COF, WSD, and flash temperature parameters.

In a study, Samuel et al. [79] explored the making of biolubricants from palm kernel oil (PKO) through various chemical modification techniques. The authors successfully synthesized biolubricants via transesterification with Trimethylolpropane (TMP) and through an epoxidation-esterification approach. The research work notable as it addresses a gap in existing literature by comparing these two methods for biolubricant production from PKO, which had not been previously undertaken. Among the biolubricants produced, the sample derived from the transesterification of PKO using TMP exhibited superior compliance with established standards when compared to the sample obtained through the epoxidation-esterification method. However, both biolubricants showed promise for application. Fourier Transform Infrared (FTIR) analysis indicated the appearance of C–H and OH functional groups, hinting at the potential for enhanced biodegradability of the produced biolubricants. The study also identified that the transesterification process was significantly impacted by variables including reaction time, temperature, and the mole ratio of reactants, while the epoxidation process was mainly influenced by the same factors, along with the molar ratio of hydrogen peroxide. Ultimately, the chemical modifications applied to PKO yielded biolubricant samples that may be particularly beneficial for lubricating machinery in the food industry, given PKO's classification as an edible oil.

Durak et al. [80] conducted a study to assess the tribological properties of cottonseed oil (CSO) as an additive in SAE 20W-50 motor oil, utilizing a journal bearing test rig. The study tested the performance of CSO at varying concentrations of 2.5%, 5%, and 10% by volume. The experiments were carried out under loads of 260 N, 360 N, and 460 N, lasting 2.5 minutes, while operating at speed ranging between 50 rpm to 1200 rpm, all at a consistent oil temperature of 25°C. The findings revealed that incorporating CSO into the mineral oil significantly lowered the friction coefficient, especially at lower journal speeds and lighter loads. The most pronounced decrease in friction occurred with the highest CSO concentration of 10%. Overall, the study provided strong evidence that CSO serves as an effective friction modifier at room temperature.

In their research, Katpatal et al. [81] examined the performance of a biolubricant combined with copper oxide nanoparticles, alongside ISO VG46 oil and its mixture with Jatropha oil, specifically in hydrodynamic journal bearings. The researchers incorporated CuO nanoparticles at concentrations of 1 wt%, 2.25 wt%, and 2.5 wt% into the 9010, 8020, and 7030 oils, respectively, to ensure that the viscosity levels were comparable to those of ISO VG oils. The study found that the pressure distribution profiles remained stable across various loads and speeds, regardless of the type of oil used. Both load and speed led to an increase in internal pressure within the bearings, with the highest pressure occurring in the oil with the highest viscosity. Notably, the introduction of nano-bio-lubricants did not result in a significant increase in maximum pressure compared to ISO VG46 oil. The performance of the 9010 biolubricant closely resembled that of ISO VG46 oil, suggesting it could be a practical alternative for journal bearings. Frictional torque values increased with speed for all oils, but exhibited only a slight increase with load in both ISO VG46 and the biolubricants. Conversely, the frictional torque values for the 8020 and 7030 nano-bio-lubricants were higher than those of ISO VG46 by 7% and 9%, respectively. The coefficient of friction for all test oils was found to rise with speed while decreasing with load, with the highest coefficients noted in the 8020 and 7030 nano-bio-lubricants, attributed to their elevated CuO nanoparticle concentrations.

In a study by M. Habibullah et.al. [82], the influence of biolubricant on the tribological characteristics of steel was determined using a four-ball tester. The tests were performed at temperature of 75^{o}C with different loads i.e. 15 kg and 40 kg, with the rotational speed of 1500 RPM and test time was 60 minutes. The lubricant used for the test was a mixture of SAE 40 oil and Jatropha oil in different proportions like 1%, 2%, 3%, 4% and 5%. The results of the study indicates that the blending of 1%-2% of Jatropha oil in SAE 40 oil had shown highest wear scar diameter (WSD) at 15 kg load. While under 40 kg load testing, maximum WSD was observed at 1%-3% of jatropha oil in SAE 40 oil and 5% of Jatropha oil in SAE 40 oil exhibited lowest WSD and flash temperature parameter. According to the study results, the 5% of Jatropha oil in SAE 40 oil had shown better tribological performance than base lubricant and it can be used as an alternative lubricant for automotive applications.

2.7.3 Tribological performance of vegetable oil biolubricant blended with additives

Additives are essential for enhancing the performance of lubricants formulated from base oils. Even with top-grade base oils, specific properties are still lacking without these enhancements. The overall effectiveness of a lubricant relies on the synergy between the base oil, additives, and the final formulation. Common additives include phosphorus, sulfur, and ZDDP.

Stachowiak et. al. [34] observed that the anti-wear additives like fatty acids, alcohols, amines and esters, form a molecular lubricant layer that adheres to the surfaces either through physisorption or chemisorption. This lubricant layer consists of highly ordered and tightly packed molecules, with each additive polar head attaching to the worn surface [83]. Strong dipole interactions also occur between these molecular chains, making the strength of the bond between the polar chain ends and the metal surface playing a pivotal role in the lubricant's effectiveness [84]. Extreme pressure (EP) additives were frequently used to provide protection underneath high-pressure conditions [85]. These additives initiate tribochemical reactions that form protective layers composed of iron compounds like sulfides, chlorides, and phosphates [86]. These layers develop into boundary lubricating films, or tribofilms, which are critical for minimizing friction and wear in tribological systems. The characteristics of these tribofilms such as their structure, stability, and mechanical properties, depend on the contacting materials and the specific types of lubricant additives applied [87].

In a study by Masjuki et.al [72] found that incorporating 5 vol% of POME to a base oil lubricant significantly decreased the wear rate of EN31 steel ball bearings. POME was produced from crude palm oil via transesterification. Palm oil methyl ester has a very low sulfur content (0.002 wt%), making it an eco-friendly option. In further research, [88] demonstrated that including palm oil methyl ester in mineral oil reduced the running-in time and minimized the steady-state friction coefficient under loads of 600 to 800N. It was noticed that the friction coefficient difference between mineral oil with and without the ester became more pronounced at loads above 800 N.

Gong et al. [89] examined the effectiveness of two synthetic thiophosphates—tri-n-octyl thiophosphate and tri-n-octyl tetrathiophosphate—as well as tricresyl phosphate when used as additives in rapeseed oil to minimize wear during steel-on-steel sliding. Their results showed that synthetic thiophosphates were particularly effective in reducing wear, likely due to tribochemical interactions that formed a protective boundary layer on steel surfaces. Additionally, a range of long-chain dimercaptothiadiazole derivatives was evaluated as anti-wear (AW) and extreme pressure (EP) additives in vegetable oil using a four-ball test, which demonstrated enhanced EP properties in the base colza oil. Thermal films from these derivatives included ferrous sulfate with small amounts of adsorbed organic sulphides [90]. Tombolini et.al. [91] further found that, under boundary lubrication in paraffin oil at high loads, thiadiazole derivatives provided better friction reduction and AW performance compared to lubricants with ZDDP, along with superior anti-oxidative and anti-corrosive characteristics.

Sharma et al. [92] explored the potential of synthesizing vegetable oil with thiols to create hydroxyl thioether derivatives while maintaining the beneficial attributes of the original vegetable oil, such as high flash point, lubricity, and environmental benefits. This process effectively removed polyunsaturation in the fatty acid chains and introduced polar functional groups that improved metal surface adsorption, resulting in a marked reduction in wear and friction. In a study, Xu et al. [93] showed that catalytic esterification of bio-oil derived from spirulina algae could enhance lubrication. Their study revealed that blending ethanol with esterified bio-oils using potassium fluoride/alumina and potassium fluoride/HZSM-5 zeolite as catalysts reduced the friction coefficient by 22% and 10%, respectively, compared to ethanol mixed with untreated bio-oil. This friction reduction was attributed to the creation of a more effective tribofilm on worn surfaces by the esterified bio-oils.

Shi et al. [94] explored how varying water content in glycerol solutions influenced viscosity, friction, wear, and film thickness underneath elasto-hydrodynamic and boundary lubrication conditions. They found that while increasing water content decreased viscosity and film thickness, there was an optimal water level that minimized friction. However, there was no clear relationship between friction coefficient and wear loss. In elasto-hydrodynamic lubrication, rapeseed oil exhibited a friction coefficient approximately three times higher than pure glycerol, whereas under boundary lubrication, the friction coefficients of rapeseed oil and glycerol were nearly identical. Glycerol solutions with 5–20 wt% water achieved a stable, lower friction coefficient, though excessive water led to a drop in viscosity, reducing load-carrying capacity. This study suggested that glycerol-water mixtures could be viable, environmentally friendly alternatives to traditional rapeseed oil as base oils.

In a study, Jayadas et.al. [75] construed that the addition of 2% weight of ZDDP to coconut oil substantially reduced wear in a four-ball wear test. This modified coconut oil exhibited a welding load that surpassed that of conventional 20W50 oil. It was also observed that coconut oil is consistent than many vegetable oils but it has limited application due to its high solidification temperature. Coconut oil is a triacylglycerol-based vegetable oil and it shares beneficial characteristics with other oils used as lubricants, including a high viscosity index, outstanding lubricity, high flash points, and minimal evaporation loss.

In a separate study, Quinchia et al. [7] examined the frictional behavior and lubricating film-forming capacity of various vegetable oil-based lubricants, including high-oleic sunflower, soybean, and castor oils. In the study, the authors have enhanced the oils with 4% ethylene-vinyl acetate copolymer and 1% ethyl cellulose additives. The results revealed that castor oil exhibited superior lubrication performance compared to sunflower and soybean oils. This performance was accredited to the hydroxyl functional group in castor oil, which enhanced its viscosity and polarity, leading to better film formation, reduced friction, and increased wear resistance. The ethylene-vinyl acetate copolymer contributed moderately to improving the film-forming characteristics by reducing friction and wear during mixed lubrication. Additionally, ethyl cellulose demonstrated significant effectiveness as an additive with castor oil, improving lubrication in both mixed and boundary lubrication regimes.

In a study, Alves et al. [95] demonstrated that modified vegetable oils, including epoxidized sunflower and soybean oils, achieved lower friction coefficients compared to traditional mineral oil and synthetic oil. However, the addition of CuO and ZnO nanoparticles to the epoxidized vegetable oils resulted in increased friction and wear. This increase was attributed to interaction between the nanoparticles and the contact surfaces, where the polar groups of vegetable oils adhered to the surfaces and causing the nanoparticles to roll, leading to three-body abrasion and higher wear. Conversely, when oxide nanoparticles were incorporated into mineral and synthetic oils, they helped minimizing friction and wear by forming a protective tribofilm on worn surfaces.

In a study, Xu et al. [96] found that bio-oil emulsion produced from rice husk pyrolysis achieved the lowest friction coefficient, followed by bio-oil alone and then diesel oil. Although diesel oil resulted in the least wear, bio-oil led to the highest wear. In the study, it was concluded that the emulsified bio-oil had the best overall tribological performance due to its acidic components with polar groups. Xu et al. [97] also observed that blending bio-oil from rice husk pyrolysis with diesel fuel improved lubrication compared to conventional diesel. However, this blend slightly reduced anti-corrosion and anti-wear properties in the diesel fuel. In a research by Suarez et al. [98] indicated that incorporating soybean oil methyl esters and pyrolytic fuels derived from soybean oil enhanced diesel fuel lubricity, highlighting the potential of these biofuels as effective additives for enhancing the wear resistance of fossil fuels.

Yashvir et al. [99] developed a biolubricant using desert date oil as a base, exploring the friction and wear characteristics of the modified oil. Modification involved the addition of copper nanoparticles in varying proportions (0.3%, 0.9%, 1.3%, and 1.6% by weight). The tribological characterization of these lubricants was carried out on a four-ball test, following the ASTM D4172 procedure. The testing adhered to ASTM standards, globally accepted for such analyses. However, the tribological properties of the modified desert date oil deteriorated with an increase in nanoparticle concentration, particularly at 1.6%. Beyond a certain threshold, additional copper nanoparticles failed to enhance the lubricant's characteristics during analysis.

2.8 Literature Gaps

After the extensive study of literature available on the subject topic, the following literature gaps were found:

- The preparation of a Biolubricant derived from Apricot kernel oil remains unexplored in existing literature.
- Despite the growing interest in Biolubricants synthesized from Apricot oil, a comprehensive examination of their tribological performance is notably absent.
- Existing studies on the tribological performance of biolubricants have primarily focused on specific blends with mineral lubricants, leaving a significant gap in the exploration of a broader range of biolubricant proportions in conjunction with mineral lubricants.
- The evaluation of Apricot oil-based biolubricant performance lacks experimentation on both a four-ball tribometer and a high-temperature pin-on-disc tribometer, leaving crucial aspects of its applicability unexplored.
- A thorough investigation into the statistical analysis of biolubricant performance, particularly derived from Apricot oil, is notably lacking, hindering a comprehensive understanding of its effectiveness.

2.9 Research Objectives

On the basis of exhaustive literature study, the following research objectives were decided:

1. To synthesis some promising bio-additives for its characterisation.
2. To study the Tribological behaviour of Bio-additives in lubricant employing four-ball tester/tribometer.
3. To study the effect of lubricant blends with bio-additive in the journal bearing.

2.10 Organization of the thesis

The thesis has been organized into five chapters. The first chapter is **"Introduction"** which presents the introductory knowledge of components of tribology and biolubricants. The second chapter is **"Literature review"**. It contains a detailed review of earlier work in the field of tribology and biolubricants along with their tribological performance. The exhaustive literature study has been carried to find the literature gaps. On the basis of these research gaps, objectives and methodology of present research work has been achieved. The third chapter is **"Methodology and Experimentation"**. It presents the methodology and experimentation utilized for achieving the objectives of the thesis. This chapter has been divided into three parts. The first part elaborates the preparation and characterization of apricot oil based biolubricant. The second part elaborated the methods used for determining the tribological performance of apricot oil based biolubricant. High temperature tribometer and Four-ball tester were used to determine the tribological performance. The third part contains the methods to determine the performance of biolubricant in the field of journal bearings. The fourth chapter is **"Results and Discussion"**. This chapter presents the results of experimental works and statistical analysis. The results of high temperature tribometer, four-ball tester and journal bearing experimentation with discussion were presented. The fifth chapter is **"Conclusions and Future scopes"**. This chapter highlights the main conclusions of the work and also discusses the scope of future works.

CHAPTER 3: METHODOLOGY AND EXPERIMENTATION

In the previous chapter, an extensive literature survey on components of tribology and biolubricant; along with its production and tribological performance has been presented. This chapter outlines the experimental procedures and methodologies utilized to achieve the objectives of this study. It is organized into four sections. In the first section, preparation of apricot oil based biolubricant and its characterisation was determined by conducting the FTIR analysis. The physio-chemical properties were determined by using different instruments operating on ASTM standards. The second section discusses the tribological behaviour of prepared Apricot oil based Biolubricant and its blends with 15W40 oil under different temperature condition using High Temperature Tribometer (ASTM-G99) and using a four ball tester (ASTM-D4172). Also the statistical analysis of the test data was also shown in this section. The third section describes the effects of prepared biolubricant blended with 15W40 oil as bio-additive in the journal bearing. The fourth section contains the summary of the chapter.

3.1 Preparation of apricot oil based biolubricant and its characterisation

Apricot (Scientific name: *Prunus Armeniaca*) is a vital fruit crop in the cold desert region of Ladakh, particularly in the Leh district. It occupies 54% of the total area dedicated to fruit crops, covering approximately 707 hectares, with an estimated annual yield of 2,956 metric tons of fresh fruit. Due to their perishable nature, approximately 85% of the harvested apricots are dried for prolonged availability throughout the year. This fruit not only serves as a primary livelihood source for the local population but also deeply intertwined with the region's culture and traditions [100].

Apricot fruit, also known as Khubani in Ladakh are categorized based on the taste of their kernels: bitter varieties, called "Khante," and sweet varieties, known as "Nyarmo." Sweet kernels are consumed directly and are valued for their rich content of dietary protein, fiber, and oil. Bitter apricot kernels from the region have significant nutritional properties, containing up to 54.21% oil, 17.75-22.56% protein, 21.16-35.26% carbohydrates, 0.84-4.71% crude fiber, and 6.03-22.24% dietary fiber. The oil extracted from these kernels is high in unsaturated fatty acids, particularly oleic acid (66.2%) and linoleic acid (28.2%) [101].

Sweet apricot kernel oil is primarily used for culinary purposes, whether in its pure form or blended with walnut oil. The bitter kernel oil, however, holds significant religious, cosmetic, and medicinal value. Traditionally, it has been used as hair oil and is known for relieving back and joint pain. Its non-greasy absorption makes it an ideal choice for body and massage oils. In local remedies, warming apricot oil and adding a pinch of salt before applying it to the chest is believed to provide relief from acidity [102].

The demand for apricot oil has surged in recent years due to its medicinal properties. In addition to local use, soldiers stationed in the region and tourists often purchase apricot oil as a cherished gift from Ladakh. In order to meet the growing demand of apricot oil, many locals have transitioned from the labor-intensive traditional extraction methods to modern mechanical processes. Despite this shift, oil extracted using traditional methods remains highly valued by both locals and visitors. This oil is distinguished by a unique aroma absent in the mechanically extracted version, making it highly sought after. As a result, traditionally extracted apricot oil commands prices 50-100% higher than its modern counterpart in local markets.

3.1.1 Extraction of apricot kernel oil

The traditional process of extracting apricot kernel oil involves several steps, from collecting fresh fruit to packaging the oil. The overall process of extraction of apricot oil is as follows:

(a) **Fruit Collection and Seed Separation:** Apricots ripen on the tree are either picked up or fallen from the apricot tree. The fruit pulp is manually pressed out and set aside to dry, while the seeds are carefully separated.

(b) **Softening the seed shell:** The hard apricot seeds are softened by soaking them in water for 25-30 minutes, making it easier to break the hard shell. The discarded shells are often repurposed as firewood for the oil extraction process.

(c) **Kernel separation:** A handful of the softened seeds is placed on a flat stone, and their shells are broken using a hand-sized stone tool. The kernels are then extracted from the crushed shells.

(d) **Kernel crushing and grinding:** The kernels are initially crushed into a coarse powder using a stone mortar and pestle, with the pestle typically crafted from apricot or walnut wood. The coarse powder is then ground into a finer paste on a flat or slightly curved stone surface, using a palm-sized stone to achieve paste of desired consistency.

(f) **Oil Extraction:** The ground paste is then processed on a slightly curved stone which has a cup-shaped groove at one end and is heated over a fire. The temperature of the stone is regulated so that it can be touched with bare hands without causing burns. The paste is thoroughly kneaded by hand on the heated stone, and during this process, a small amount of water is sprinkled onto the paste to facilitate oil extraction. The extracted oil flows into the groove and is collected with a spoon before being transferred to a container. The Figure 15 shows the process flow of extraction of Apricot oil.

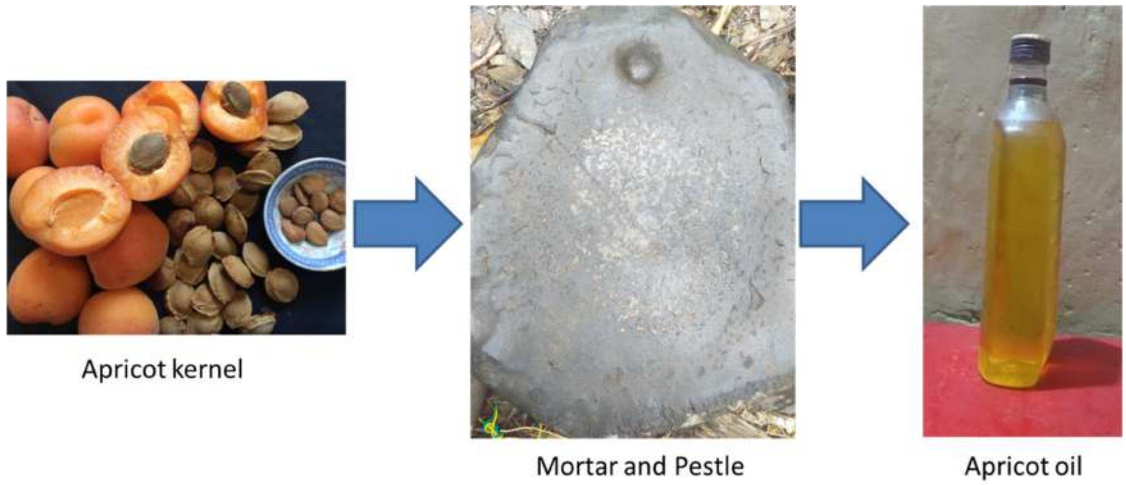


Figure 15: Process flow of extraction of Apricot kernel oil

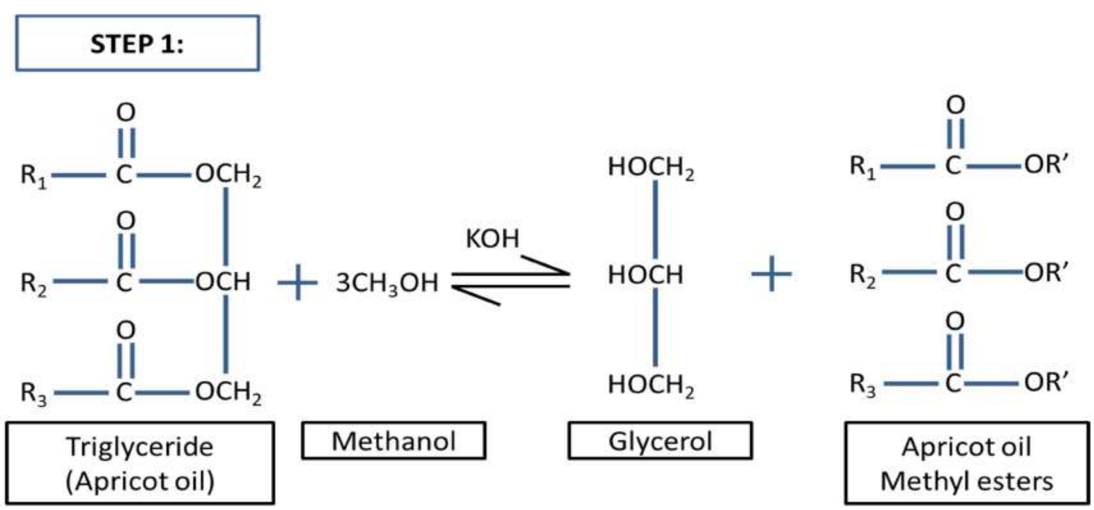
3.1.2 Chemical conversion of apricot kernel oil to biolubricant

Vegetable oils currently available are generally unsuitable for use as lubricants due to their inadequate performance in low-temperature conditions and their low oxidative and thermal stability. Various strategies are employed to address these limitations, including modifying the fatty acid composition through genetic engineering, incorporating additives such as antioxidants, viscosity enhancers, and pour-point depressants, as well as emulsification and chemical alteration of the oils. These strategies aim to improve the overall functionality and stability of vegetable oils. As illustrated in Figure 1, one method involves rearranging the chemical modification stands out as the most promising, offering significant potential to improve both chemical stability and performance across a broad temperature range. Chemical modification typically involves altering the molecular structure of the oils. For example, rearranging the acyl (C=O) and alkoxy (O-R) groups or modifying double bonds within the triglycerides can create new triesters through processes like esterification or transesterification. Additionally, acyl groups can be transformed into estolides by hydrolyzing triglycerides, leading to the formation of branched esters. Another approach involves epoxidizing the double bonds and opening the rings to produce intermediates that facilitate the synthesis of a variety of diesters.

The transesterification is a technique used to create esters (RCOOR') by reacting an acid with an alcohol, with the removal of water generated during the reaction. This method is especially useful for modifying acyl groups, yielding esters with superior physical properties that enhance the performance of vegetable oils as lubricants.

There are several factors which influence the transesterification process, including the type of catalyst used, reaction duration, temperature, and flow rate, water content in the alcohol, the quantity of excess alcohol, and the levels of free fatty acids in the starting materials. Excess water in alcohol can lead to soap formation by consuming the catalyst, thereby reducing its efficiency. In order to achieve maximum conversion, it is preferable to use anhydrous alcohol. The excess alcohol drives the reaction equilibrium towards ester production, increasing the yield of methyl esters.

In the presented research work, Apricot kernel oil was undergone through base esterification. In base esterification, apricot oil is mixed with methanol in a 6:1 ratio, and Potassium hydroxide is utilized as catalyst at a concentration of 1%(w/w). A mixture of Apricot oil methyl ester (AOME) and glycerol was the product of Ist Step of chemical transformation. The glycerol can be separated by keeping the mixture in a conical flask for at least 24 hours and then further removing the traces of glycerol via water wash. After removal of glycerol, AOME is subjected to heat for removal of any water traces. In the IInd Step of chemical transformation, the AOME was combined with trimethylolpropane (TMP) in a 1:3 ratio in the presence of 1% sodium methoxide. The final product is a biolubricant and methanol. The two step chemical reactions for transformation of apricot oil to its biolubricant is shown in the Figure 16 below. The phases of transesterification of apricot oil are shown on Figure 17.



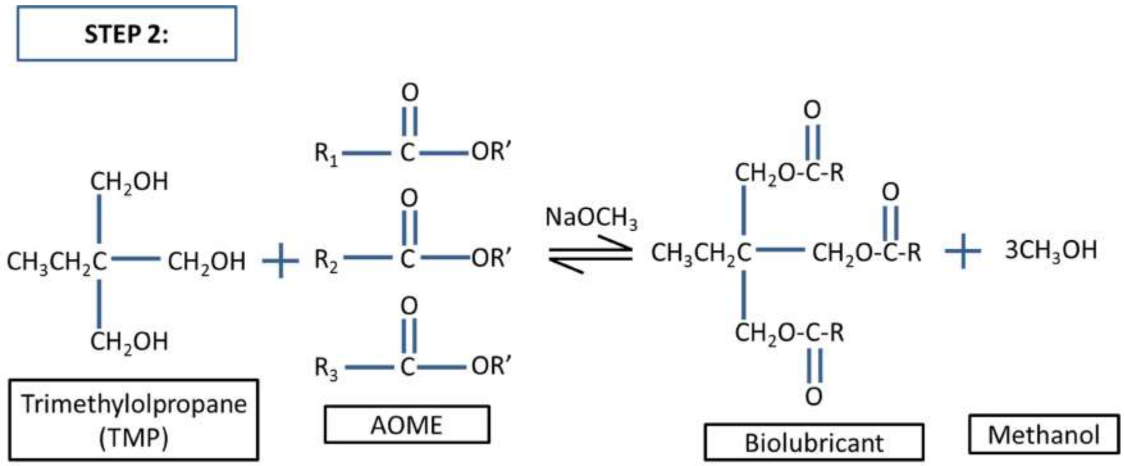


Figure 16: Two-step process Chemical transformation of Apricot kernel oil.

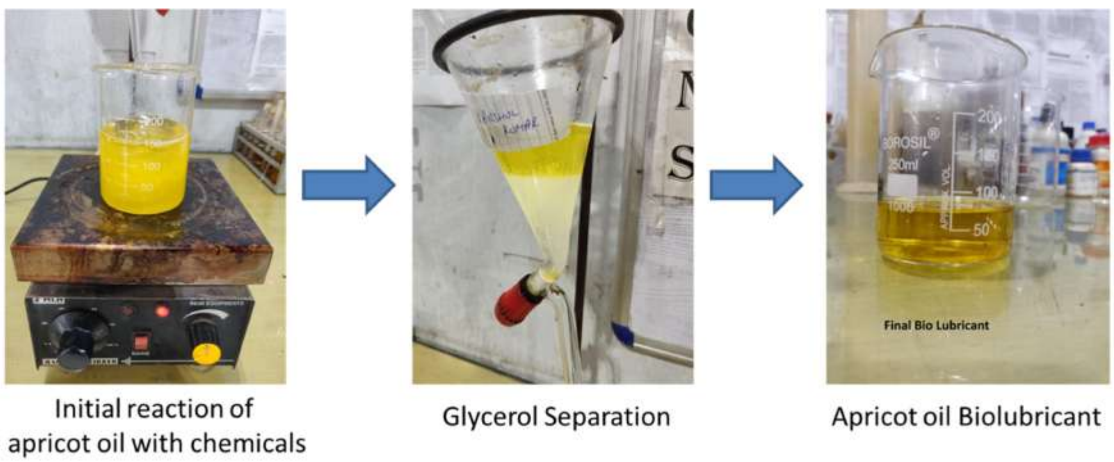


Figure 17: Phases of Transesterification of Apricot oil

3.1.3 Characterisation of apricot oil based biolubricant

Biolubricant has properties that accentuate its performances and suitability for various usages. The prepared Apricot oil based Biolubricants were characterised to determine the physio-chemical properties.

Viscosity: Viscosity is the measure of resistance between the fluid layers during the fluid flow. Viscosity of a fluid also determines fluid thickness. The high quantitative value of fluid viscosity means thicker it will be and more energy will be required to move an object through the fluid. For biolubricants, viscosity is a key parameter as it affects the lubrication efficiency, energy consumption, and wear protection. High viscosity provides better load-bearing capacity but can increase energy consumption due to higher friction. Conversely, low viscosity fluids flow more easily but may not provide sufficient lubrication under high stress.

Viscosity Index: The Viscosity Index (VI) measures how much a lubricant's viscosity is affected by temperature changes. Lubricants with a high VI maintain a stable viscosity over a wide temperature range, making them ideal for consistent performance under varying environmental conditions. Viscosity of a biolubricant decreases as temperature increases; machinery that operates across a broad temperature spectrum requires a lubricant with a high VI. A higher VI minimizes the impact of temperature fluctuations on a lubricant’s viscosity. Biolubricants with a high VI are especially valuable in applications where temperature fluctuations are common [103].

Pour Point: At low temperatures, the viscosity of a biolubricant increases significantly, hindering its ability to flow. The pour point is the lowest temperature at which a biolubricant can flow under the influence of gravity. It represents the final temperature before the lubricant ceases to move, though it is not necessarily the point at which it solidifies. This property is critical for equipment operating in cold environments or handling low-temperature fluids. Oils with high viscosity may stop flowing at low temperatures due to the formation of waxy substances, causing the pour point to exceed the cloud point [104].

Flash point and Fire point: The flash point is the minimum temperature at which a lubricant emits enough vapor to form a flammable mixture in the air. A high flash point is desirable for safety reasons, as it indicates that the lubricant can be used at higher temperatures without posing a significant fire hazard. The temperature at which a lubricant will sustain combustion for at least five seconds is known as fire point. These properties are essential for assessing the volatility and fire resistance of biolubricants, which in turn affect their storage and transport considerations. For instance, biolubricants with flash points below 38°C require extra precautions for safe handling. The flash and fire points are key indicators of a lubricant's flammability [105]. Mahmud et al. [106] reported that the number of carbon atoms in triglycerides significantly influences the flash point of biolubricants.

Acid Value and Free Fatty Acid: It is the measure of acidic component present in a biolubricant which indicates the presence of free fatty acids or oxidation products. High levels of acidic compounds in biolubricants can led to the corrosion of machinery components and the blocking of oil filters due to sludge formation. Hence, a low acid value is preferable for a biolubricant as it is less likely to cause corrosion or degradation of machine parts [107]. The acid value of a biolubricant is determined by titration process with a standard base like Potassium hydroxide (KOH) or Sodium Hydroxide (NaOH) using phenolphthalein as an indicator. The acid value is then determined by calculating the volume of base required to neutralise the acids.

The acid number of apricot oil was determined through titration using a 0.1N KOH solution. Firstly, 1 gram of apricot oil was combined with 10 ml of isopropanol, and two drops of phenolphthalein indicator were added. The 0.1N KOH solution was then gradually added from a burette until a stable pink color was achieved. The volume of KOH solution used in the process was recorded to calculate the acid number of the oil using the following equation 7:

$$Acid\ Value = \frac{56.1 * \frac{1}{10} * Vol.of\ KOH\ consumed}{Weigth\ of\ the\ oil\ sample} \tag{7}$$

The Free Fatty Acids (FFA) are the molecules found in vegetable oil that are released when triglycerides breaks down. FFAs in vegetable oil help to determine important characteristics like viscosity and lubricity. The free fatty acid (FFA) composition in vegetable oils varies based on the source of the oil, which can significantly influence the performance and stability of biolubricants. Oils with a higher concentration of unsaturated FFAs often exhibit improved fluidity and enhanced low-temperature performance, making them versatile for a wide range of applications. However, a higher degree of unsaturation can also increase susceptibility to oxidation, requiring careful formulation and the possible addition of stabilizers to extend the biolubricant's shelf life.

The Free Fatty Acid (FFA) content of vegetable oils is often represented in terms of the percentage of oleic acid or a similar reference fatty acid. The acid number (also known as acid value) measures the amount of potassium hydroxide (KOH) needed to neutralize the free fatty acids in the oil. The relationship between the two is shown in equation 8:

$$FFA\ \approx \frac{Acid\ Number}{2} \tag{8}$$

3.1.4 FTIR spectrum analysis of apricot oil based biolubricant

The Fourier transform infrared technique has emerged as a valuable tool for simultaneously assessing various organic components, such as chemical bonds, as well as organic content like carbohydrates and proteins. In Fourier-transform infrared (FTIR) spectroscopy, samples are subjected to infrared radiation, which interacts with molecular vibrations. This interaction leads to energy being absorbed or transmitted in a manner unique to the sample's molecular structure. This distinct absorption pattern enables FTIR to accurately identify and analyze the specific molecular vibrations in a sample without overlap or interference [108]. The infrared spectrum is separated into three regions according to wavenumbers: the far-infrared region (below 400 cm^{-1}), the mid-infrared region (400 cm^{-1} to 4000 cm^{-1}), and the near-infrared region (above 4000 cm^{-1}). Among these regions, mid spectrum finds the broadest application in sample analysis and is further sub-divided into following regions:

i. The single bond region (2500-4000 cm^{-1})
ii. The triple bond region (2000-2500 cm^{-1})
iii. The double bond region (1500-2000 cm^{-1})
iv. The fingerprint region (600-1500 cm^{-1})

The FTIR spectroscopy works on the principle of radiation interference between two beams, producing an interferogram. This interferogram is a signal that changes based on the difference in path length between the beams. The distance and frequency domains can be converted into one another using Fourier transformation, a mathematical technique. An FTIR spectrometer consists of several components, as illustrated in Figure 18. Radiation from the source passes through an interferometer before coming into contact with the sample and arriving at the detector. The resulting signal is then amplified, filtered to eliminate high-frequency noise, converted to digital format, and processed via Fourier transformation using a computer.

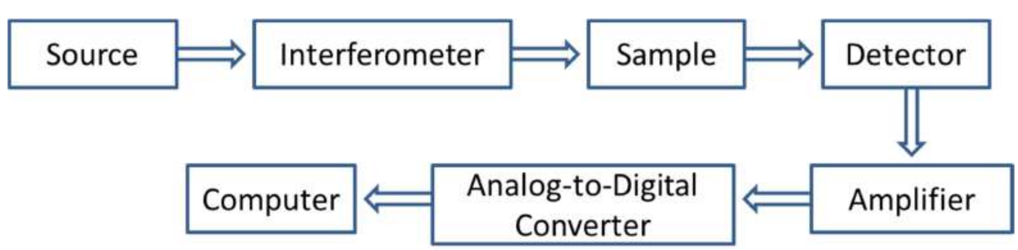


Figure 18: Components of an FTIR spectrometer

In an Infrared (IR) experiment, two types of interactions—absorption and transmission—are crucial. When a molecule in the spectrometer's sample compartment is exposed to continuous IR radiation, certain photons are absorbed by the molecule and do not reach the detector. The IR spectrum displays these missing photons as distinct absorption bands, which are both characteristic and reproducible. Photons that are not absorbed pass through the sample unchanged and are detected.

The infrared spectrum for apricot oil based biolubricant was obtained using Nicolet iS50 FTIR Tri-detector. The spectroscopy was conducted at ATR module with the scanning range of 400-4000 cm^{-1}. Figure 19 shows the setup of FTIR tri-detector.

Figure 19: Setup of Nicolet iS50 FTIR Tri-Detector.

3.2 Tribological study of apricot oil based biolubricant

This section contains the performance study of the apricot oil based biolubricant in the area of tribology. First the oil sample for experimentation was prepared followed by the its testing in High temperature tribometer and Four-ball tester.

3.2.1 Sample preparation

The oil samples were prepared for its tribological analysis by blending the commercially available mineral oil i.e. 15W40 oil and prepared apricot oil based biolubricant. The blending process utilized a volume-to-volume approach, facilitated by a magnetic stirrer capable of operating up to 350^{o}C and at speeds of up to 2000 RPM. Figure 20 shows the setup of magnetic stirrer. Proportionate volumes of each oil were measured, combined in a beaker, and mixed at 40^{o}C using the magnetic stirrer to create a uniform blend.

The oil samples were prepared with different compositions of apricot oil based biolubricant and 15W40 oil. These oil compositions were prepared as per the % V/V given in the Table 4 below.

Table 4: Composition of different oil samples

Sample→	**S1**	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**	**S8**	**S9**	**S10**	**S11**
% 15W40	100	90	80	70	60	50	40	30	20	10	0
% Biolube	0	10	20	30	40	50	60	70	80	90	100

Figure 20: Setup of magnetic stirrer

These oils samples were subjected to different experimental conditions to determine their tribological performance. Different tribometers were used to determine the performance of oil samples. The presented research work makes use of two tribometers: High Temperature Tribometer and Four ball tester.

Tribometers are the instruments used to measure friction, wear, and lubrication between two surfaces in relative motion. Tribometers are essential in field of tribology. Tribometers help to evaluate material durability, predict component lifespan, and design surfaces that minimize wear and energy loss by simulating various contact conditions.

3.2.2 High temperature tribometer

In the presented research work, the high temperature tribometer was utilised in a Pin-on-Disc configuration. A pin-on-disc configuration involves a stationary cylindrical pin pressed against a rotating disc in the presence of a lubricant oil sample. The tests followed the ASTM-G99 standard for tribological evaluations.

In the presented research, a High temperature tribometer of DUCOM make and model TR-20L-PHM800-DHM800 had been used for evaluating the tribological performance of a tribopair in sliding contact under various test oil samples administered through drop-wise lubrication. The pin-on-disc setup comprises a stationary cylindrical pin subjected to an applied load, while a rotating disc operates at a designated speed. The testing parameters include normal load, sliding speed, and lubrication conditions.

The tribometer consists of multiple assemblies, including the spindle assembly, loading lever assembly, sliding plate assembly, thermocouple, and an environmental chamber. These components are integrated into a robust steel structure designed to withstand shocks and support applied loads. The disc is fixed to the spindle using screws and driven by an AC motor connected via a timing belt. The loading lever assembly features a single bar with a specimen holder, and the normal load is applied using dead weights suspended by wire ropes. A central pivot ensures uniform load distribution on the pin. A load cell is employed to measure the frictional force generated during the interaction between the pin and the disc.

The lubrication system is designed to minimize oil spillage, comprising an upper leak-proof steel chamber and a lower cylindrical chamber with a large outlet for draining oil. A wire mesh at the tank's inlet captures debris, facilitating smooth oil flow. The wear between the pin and disc is monitored using a Linear Variable Differential Transducer (LVDT), ensuring a 1:1 leverage ratio by placing it equidistant from the pivot and the specimen. Additionally, a thermocouple is installed within the disc chamber to regulate its temperature during experiments. The frictional force is recorded by a load cell mounted on a bracket at an equal distance from the pivot and specimen. This load cell, attached to a sliding plate, moves accordingly to adjust the wear track diameter during testing. Together, these components enable precise measurement and control during tribological analysis. The Figure 21 shows the experimental setup of High Temperature tribometer.

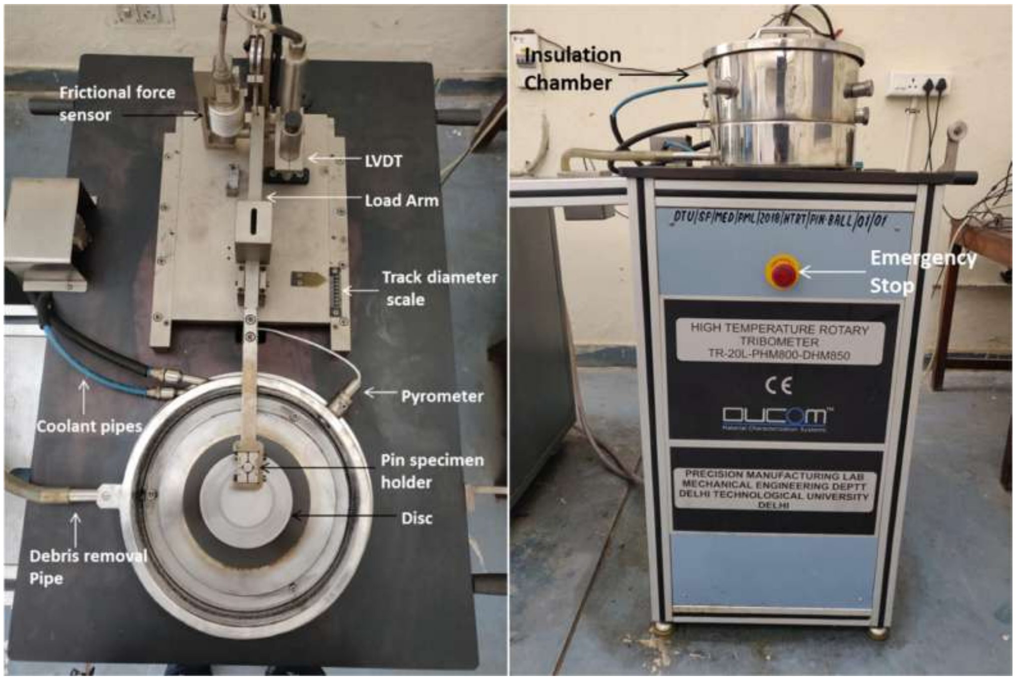


Figure 21: Experimental setup of High Temperature tribometer

A cast iron pin and a mild steel disc were used as a tribo-pair for the tribological analysis. The tribo-pair used for the tribological analysis and their properties are shown in the Table 5.

Table 5: Properties of Tribo-pair.

Material Properties	**Components**	
	Disc	**Pin**
Material	Mild Steel	Cast Iron
Density (kg/m^3)	7885	7142.5
Modulus of Elasticity (MPa)	$2x10^5$	$1.5x10^5$
Specific Heat (J/kg-K)	463	459
Thermal conductivity (W/m-K)	54.2	47.4
Poisson's Ratio	0.3	0.27

The following Table 6 shows the parameters for tribological analysis on High temperature tribometer for oil samples of different compositions, as shown in the Table 4 above.

Table 6: Parameters for tribological analysis on High temperature tribometer

Parameter	Values
Composition of Pin material (%)	C=3.41%, Cr=1.26% Mn=0.63%, Si=2.80%, Fe=91.9%
Composition of Disc material (%)	C=0.17%, Cr=1.42%, Si=0.27%, Mn=0.54%, Fe=97.6%
Oil Specimen	15W40 oil and various blends of 15W40 with apricot oil based biolubricant
Sliding Speed (m/s)	1.3, 1.57, 1.83, 2.09
Normal Load (N)	35
Temperature (^{o}C)	40,100 & 150
Sliding Distance (m)	2000
Pin Diameter (mm)	10
Disc Diameter (mm)	100

3.2.3 Statistical analysis

Statistical analysis is a set of techniques used to interpret, analyse, and infer conclusions from data. These techniques allow the examination of trends, relationships, and patterns, providing valuable insights for decision-making across various fields. Analysis of Variance (ANOVA) is one of the statistical analysis techniques.

Analysis of Variance (ANOVA) is a widely employed statistical method designed to assess the impact of independent variables on dependent responses. Rather than directly analyzing the data, ANOVA quantifies the extent to which each factor influences the variability within the dataset. ANOVA output typically provides crucial information, including the Degrees of Freedom (DoF), Sum of Squared Deviations (SS), Mean Square (MS), and the percentage contribution (% contri) of each factor in the model. Response surface methodology (RSM) is a statistical approach used to optimize responses in situations where two or more quantitative factors come into play. The variables that are influenced by these factors are referred to as responses, while the factors themselves are typically known as predictor variables. The goal of RSM is to systematically investigate the impact of these predictor variables on the responses and, in turn, fine-tune and enhance the desired outcomes [109]

In the presented study, RSM based second order mathematical model is given by the equation 9:

$$Y = \alpha_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i,j}^{k} \beta_{i,j} X_i X_j + \sum_{i=1}^{k} \beta_{ii} X_i^2 \quad (9)$$

Where α_o is the free term of the regression equation and the coefficients β_1, β_2,... β_k and β_{11}, β_{22}...β_{kk} are the linear and quadratic terms, respectively, while β_{12}, β_{13},... β_{k-1} are the interacting terms [110]

3.2.4 Four-ball tester

The four-ball tester consist of three steel balls clamped in a position while the forth ball is fixed on a rotating spindle and pressed against the three stationary balls. This four ball configuration is ideal for testing extreme pressure properties of lubricants as it allows high pressure contact. Four-ball tester is a preferred method in the lubricant industry as it closely simulates the real world performance of lubricants under load.

In this study, a DUCOM TR-30L four-ball tester was utilized to assess wear and the coefficient of friction in accordance with ASTM-D4172 standards. The tribological testing apparatus features four steel balls, each with a diameter of 12.7 mm. Three of these balls are secured within a ball pot using a clamping ring and a locking nut. The fourth ball is mounted atop the other three and attached to a rotating spindle. This spindle is powered by a motor via a 1:1 belt-and-pulley system, with an idler pulley included to maintain tension. The spindle is housed within the device body, supported by two high-precision ball bearings for stability. The lubricant under investigation is poured into the ball pot to ensure all three stationary balls and their contact points with the rotating ball are submerged. The ball pot assembly comprises three main components: the ball pot, ball pot race, and locking nut. The ball pot holds the sample lubricant and secures the steel balls via the ball pot race. The ball race helps position the balls during the tightening of the locking nut. To monitor the oil temperature, a Resistance Temperature Detector (RTD) sensor is installed near the heater. The ball pot is placed beneath the spindle using a designated handle. A loading button inside the pot interacts with the frictional force load cell to transmit data, which is displayed on the control unit. A lever system and deadweights positioned on a loading pan apply the normal load to the balls. A load cell built into the ball pot measures the frictional force. The device operates under adjustable parameters such as load, speed, temperature, and testing duration. After completing a test run, wear scars are observed on all three stationary balls. The diameters of these wear scars are then measured using a microscope for precise analysis. An optical scar measuring microscope with image sensor permits acquiring of wear scar image on a computer. It is possible to measure the size of the scar on a computer screen with the variety of cursors. A data acquisition system acquires and displays on-line normal load, frictional force, temperature and coefficient of friction in the form of graphs. The Table 7 below shows the technical specifications of TR-30L four ball tester. Figure 22 shows the experimental setup of TR-30L four ball tester

Table 7: Technical specifications of TR-30L four ball tester.

Parameter	**Values**
Speed (RPM)	1000-3000
Max. Axial load (N)	10000
Temperature (^oC)	Ambient to 100
Test ball diameter (mm)	12.7
Scar range (micron)	100-4000
Drive Motor (KW)	1.5

The values of friction torque was acquired by the data acquisition system and it was further used to determine the coefficient of friction (μ) using the equation 10.

$$\mu = \frac{T_f\sqrt{6}}{3\ W r} \tag{10}$$

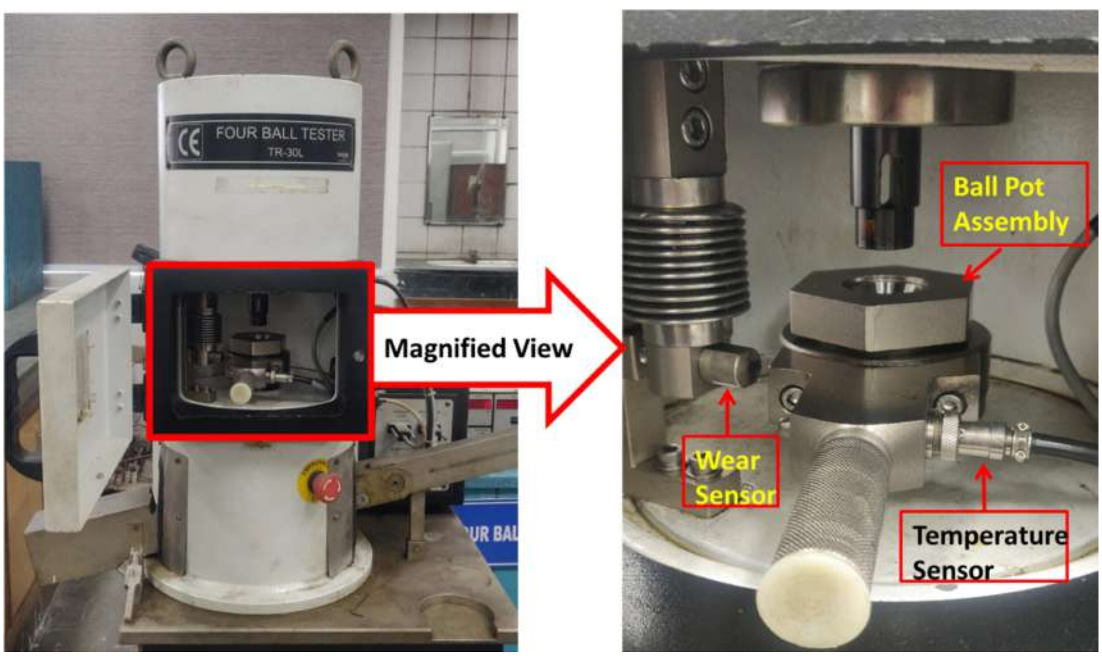
Where, T_f is the frictional torque (N-mm), W is the Applied load (N), r is the distance between the axis of rotation and the centre of contact surface on the stationary balls. Using the geometrical properties, the value of r comes out to be 3.67mm.

Further, the wear scar diameter formed on the surface of the three stationary balls was used to determine the volume wear rate (V_{wr} in mm^3/s) using the equation 11.

$$V_{wr} = \frac{\pi}{3t}\left(R - \sqrt{R^2 - s^2}\right)^2 \left(2R + \sqrt{R^2 - s^2}\right) \tag{11}$$

Where, R is the radius of steel balls (mm), s is the wear scar radius (mm), t is the sliding time (s).

(a)



(b)

Figure 22: (a) Complete setup of Four-ball tester, (b) Magnified view of test section

In the presented research, the oil samples comprised of various blends of 15w40 oil with apricot oil based biolubricants were subject to wear analysis using the four ball tester. The parameters of the wear analysis on four ball tester are shown in Table 8 below:

Table 8: Parameters of wear analysis on four ball tester

Wear analysis	
Parameters	**Values**
Standard	ASTM-D4172
Ball Specimen	Material: Steel, Diameter: 12.7mm, Ra: 0.04 micron, Hardness: RC65
Oil Specimen	15W40 oil and various blends of 15W40 with apricot oil based biolubricant
Load (N)	196 N and 392 N
Spindle speed (RPM)	1200 RPM
Temperature (^oC)	75^oC
Test duration (mins)	60minutes

3.3 Journal bearing test rig

A journal bearing is a cylindrical mechanism designed to support a rotating shaft, primarily to handle radial loads or facilitate efficient transfer of torque. Lubricating oil is filled in space between the bearing and the shaft. In the hydrodynamic lubrication process, as the shaft begins to rotate, it ascends the surface of the bearing. With an increase in rotational speed, the lubricating oil is forced into a wedge-shaped area. This wedge effect generates pressure within the system, contributing to the smooth operation of the journal bearing.

The dynamic performance of a journal bearing based upon the damping coefficients such as stiffness and damping coefficients. These damping coefficients further depend upon the viscosity of lubricating oil, journal rotating speed and load applied on the journal. The forces generated while operating a journal bearing depends upon the pressure generated by the lubricating oil, around the journal bearing. The lubricant oil pressure depends upon the lubricant viscosity, journal rotation speed, eccentricity ratio and bearing dimensions, as mentioned in the equation 12: [111]

$$P(\theta, z) = \frac{3N_s\eta}{c^2}\left(\frac{L^2}{4} - z^2\right)\frac{\varepsilon \sin\theta}{(1+\varepsilon \sin\theta)} \tag{12}$$

Where, N_s is the journal rotation speed (rpm), η is the viscosity of lubricant oil, ε is the eccentricity ratio, θ is the angular bearing position, L is the bearing length, z is the rotational axis along the bearing length.

In this research, a journal bearing test rig (DUCOM model TR660) was employed to measure pressure and frictional torque. It is a robust, user friendly and adaptable apparatus which allows pressure measurement at various angular positions along the lower half of the journal bearing. The setup includes a horizontally mounted journal on a shaft supported by self-aligning bearings. A motor drives the shaft via a timer belt mechanism. A flawless bronze bearing, capable of free movement over the journal, is subjected to radial loads using a lever mechanism that applies force by pulling it upwards against the journal. The bearing is equipped with ten sensors arranged around its circumference, with terminals connected to a junction box. The pressure measurement error margin is within ±1% MPa, while the friction torque sensor has a permissible error of ±1% Nm. The system employs a two-step pulley and belt arrangement to drive the journal and the desired speed can be controlled via software. The test rig is configured to accommodate speeds up to 8000 RPM and radial loads of up to 3000 N. A hydraulic system ensures a consistent supply of lubricating oil. The experiments utilized 15W40 oil and its blends with apricot oil-based biolubricants to measure frictional torque and system performance. The Table 9 & 10 shows the details of journal bearing test rig and input parameters of the experimentations respectively.

Table 9: Details of Journal bearing test rig

S.No.	Parameter	Units	Range
1.	Journal Speed	rpm	40 to 8000
2.	Shaft Diameter	mm	22 ± 0.005
3.	Bearing diameter	Mm	40 ± 0.050
4.	Length/Diameter ration		1
5	Maximum load	N	3000
6.	Loading Ration		1:5

The Figure 23 shows the setup of journal bearing test rig. The calibration of the pressure, frictional torque, and temperature sensors was conducted prior to the experiments. The torque measurement system comprises an arm in contact with the journal-bearing assembly to record startup torque using a data acquisition system.

Table 10: Parameters for experimentation

Material of Journal Bearing	Bronze
Material of Journal	Steel
Oil Specimen	15W40 oil and various blends of 15W40 with apricot oil based biolubricant
Journal Speed (RPM)	1000, 1200, 1500, 1700, 2000
Normal Load (N)	300, 600, 750
Journal Diameter (mm)	40
Length/Diameter (L/D) Ratio	1

During the operation of test rig, the eccentricity between the journal and the bearing creates a wedge-shaped clearance region. This wedging action produces pressure on the journal's surface, elevating it and enabling smooth rotation. For the measurement of pressure distribution across the fluid film, ten sensors were strategically positioned around the bearing's circumference. Figure 24 shows the positions of pressure sensors.

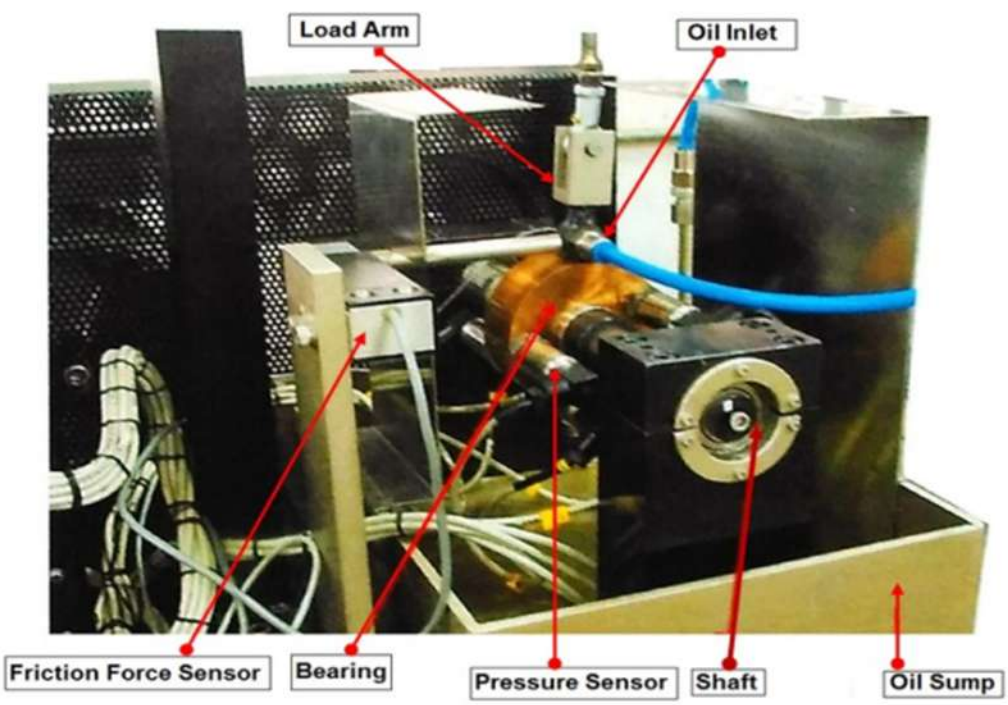


Figure 23: Setup of Journal bearing test rig.

(a) (b)

Figure 24: (a) Pressure sensor and (b) Position of pressure sensor around the journal bearing.

3.4 Summary

This chapter had focused on the methodology and experimentation work carried on to determine the characterization of the developed apricot oil based biolubricant and to determine the tribological performance of the apricot oil based biolubricant in comparison to 15W40 mineral oil. The physio-chemical characterization of the apricot oil biolubricant was carried out as per the ASTM procedures. FTIR analysis is a powerful tool for chemical characterization of the apricot oil biolubricant.

High temperature tribometer of pin-on-disc configuration and Four-ball tester were utilized to determine the tribological behavior of apricot oil biolubricant under different speed and temperature conditions. ASTM G-99 and ASTM-D4172 procedures were used to conduct test experiments. Further, a statistical analysis was performed to interpret, analyse, and infer conclusions from experimental data.

Furthermore, the performance of the apricot oil biolubricant was determined using the journal bearing test rig for the application in journal bearing under different journal speeds and normal load condition. The oil samples tested for tribological performance and journal bearing performance were the various blends of apricot oil biolubricant and 15W40 oil.

Summary of the chapter underscores the effect of various blends of apricot oil biolubricant and 15W40 oil on the tribological performance under different test conditions using the ASTM procedures; and performance in journal bearing under different conditions.

CHAPTER 4: RESULTS AND DISCUSSIONS

The preceding chapter contains the methodology and experimentation utilized in the thesis. This chapter contains three sections, each addressing the outcomes related to the objectives of this thesis work. Section 4.1 presents the characterisation results of the prepared Apricot oil based Biolubricant. Section 4.2 presents the tribological test results for the different oil samples subjected to High Temperature Tribometer and Four-Ball tester. Section 4.3 presents the performance results of different blends of bio-additive with mineral oil in the field of Journal bearing.

4.1 Characterisation of biolubricant

The biolubricant was prepared from the base Apricot kernel oil and using the chemical conversion method called Transesterification. The physio-chemical properties of prepared apricot oil based biolubricant were determined by employing ASTM method. Table 11 presents the physio-chemical properties of the prepared apricot oil based biolubricant. The properties of various vegetable oil based biolubricants were compared with the prepared apricot oil based biolubricant and the commercially available 15W40 mineral oil, and shown in Table 12.

It was observed that prepared apricot oil based biolubricant have low kinematic viscosity compared to the 15W40 mineral oil. The lower kinematic viscosity of biolubricant is due its molecular structure and base vegetable oils. Vegetable oil molecule has triglycerides which are converted into esters using transesterification. The biolubricant contains esters that contribute to a lower viscosity. The 15W40 mineral oil has complex hydro-carbon chains and it is engineered for thicker viscosity to protect the machine parts especially at high temperatures and pressures whereas the apricot oil based biolubricant is less viscous, making it flow more easily at low temperature and pressures [112].

The Viscosity Index (VI) of an apricot oil based biolubricant is higher than the VI of 15W40 oil. This means that biolubricant maintain its flow properties more consistently as temperature fluctuate, hence biolubricant can provide effective lubrication in both hot and cold temperature conditions [112].

Table 11: Physio-chemical properties of Apricot oil based Biolubricants

Properties of Oil	Value	Test Procedure	Equipment
Density (kg/m^3)	876.7	Oscillating U-tube	Viscometer SVM 3000
Kinematic Viscosity@40 ^oC (cSt)	8.72	ASTM D7042	
Kinematic Viscosity@100 ^oC (cSt)	2.89		
Viscosity Index (VI)	215	ASTM D2270	Viscometer SVM 3000
Flash Point (^oC)	155	ASTM D92	Cleveland Flash & Fire point Tester CLA 5
Fire Point (^oC)	163		
Pour Point (^oC)	-3	ASTM D5853	Callisto 100
Acid Value	3.0855	Titration with N/10 NaOH Solution	-
FFA	1.5427	-	-

Table 12: Comparison of properties for various vegetable oil based Biolubricant and 15W40 mineral oil. [5]

Various Oils	KV @ 100^oC (cSt)	KV @ 40^oC (cSt)	Viscosity Index	Flash Point (^oC)	Pour Point (^oC)
Apricot oil	2.89	8.72	215	155	-3
Jatropha TMP	7.9	35.4	205	186	-6
Palm oil TMP	10.2	52.4	186	253	-2
Castor oil TMP	4.47	20.94	220	250	-
Olive oil	8.24	39.62	190	318	-3
Coconut oil	5.5	24.8	169	325	-
Rapeseed oil	10.07	45.60	180	252	-12
Soya bean oil	7.42	28.86	246	325	-9
15W40 Oil	13.9	105	130	203	-36

FTIR spectrum analysis of Apricot oil based Biolubricant

The Fourier transform infrared technique has emerged as a valuable tool for simultaneously assessing various organic components, such as chemical bonds, as well as organic content like carbohydrates and proteins.

The infrared spectrum for apricot oil based biolubricant, as shown in Figure 6, was obtained using Nicolet iS50 FTIR Tri-detector. The spectroscopy was conducted at ATR module with the scanning range of 400-4000 cm-1. Figure 25 shows the infrared spectrum for apricot oil based biolubricant. The following observations can be made after studying the infrared spectrum:

1. Number of absorption bands in entire IR spectrum = 6, which is more than 5. Hence the biolubricant sample is a complex molecule.
2. A sharp absorption peak is observed at 2919 cm-1, which is below 3000 cm-1, and followed by a peak at 1463 cm-1 and 723 cm-1. Hence, the biolubricant sample is a long chain linear aliphatic compound.
3. There is no peak observed in between spectra frequencies 2000 to 2500 cm-1. Hence, there is no triple bond present in the biolubricant sample.
4. A sharp peak at 1741 cm-1, which is in between 1750 and 1700 cm-1, indicates the presence of a simple carbonyl compound such as like ester.
5. The peaks appearing in the regions of 1463.7, 1168.85 and 723.17 cm-1 was for the C-H bending vibrations of the CH_2 and CH_3 groups. Absorptions observed in the region of 2919.69 cm-1 was for the stretching vibrations of the paraffinic C-H bonds.

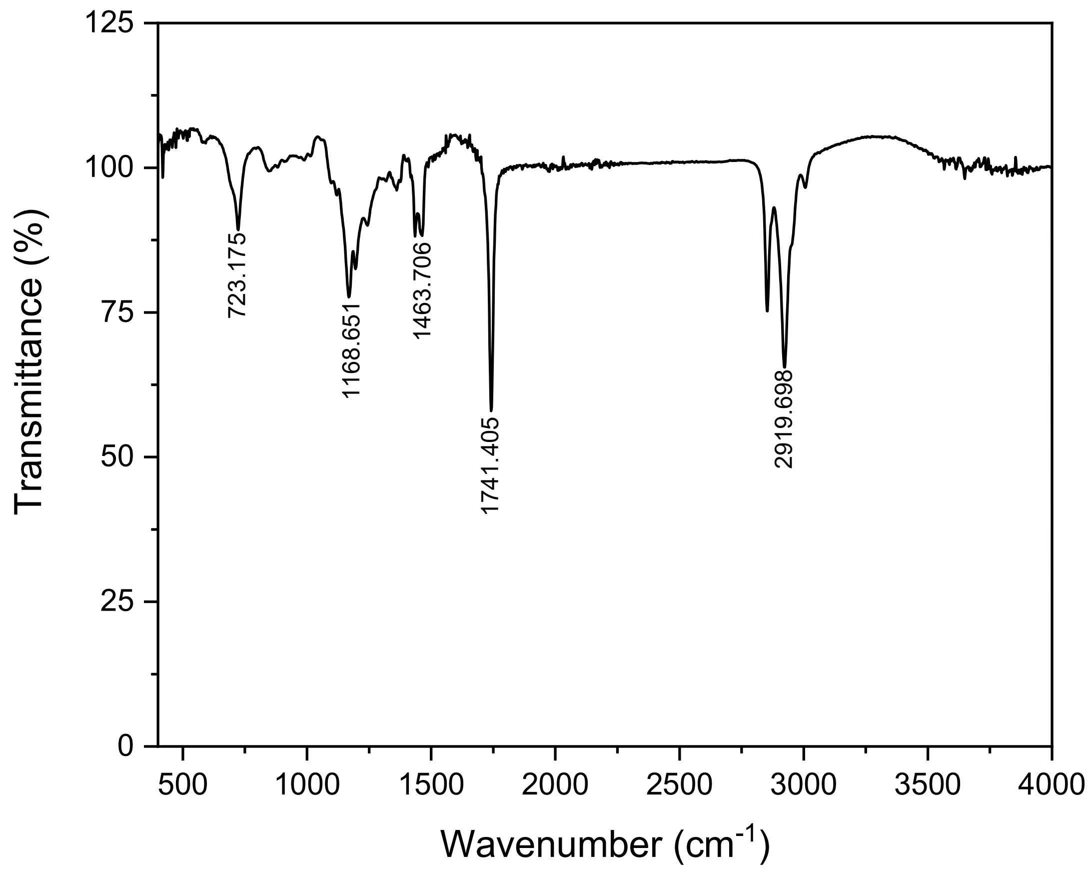


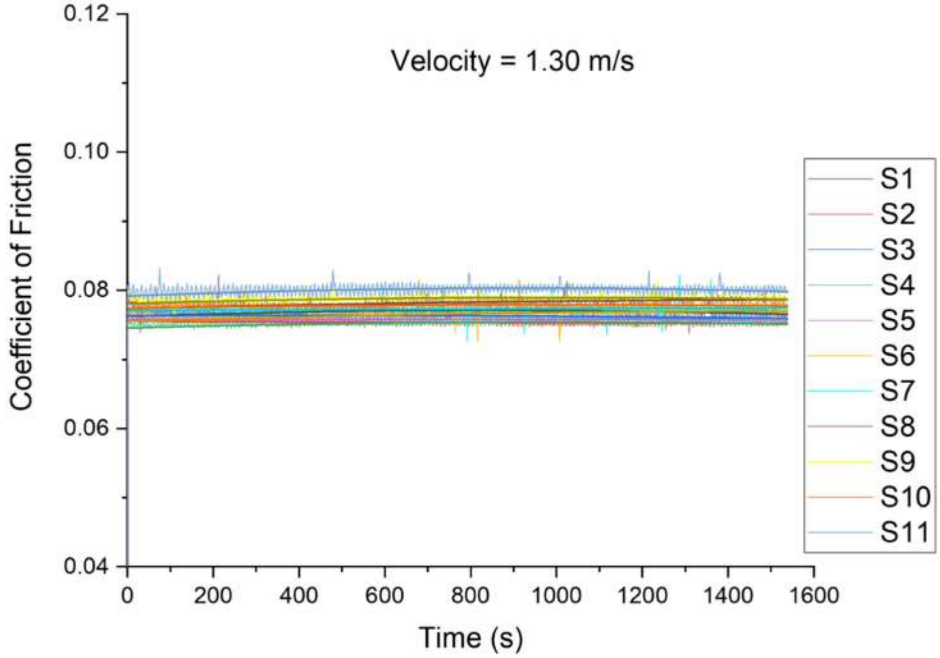
Figure 25: Infrared Spectrum of Apricot oil based Biolubricant.

4.2 Tribological behaviour of Bio-additive in lubricant

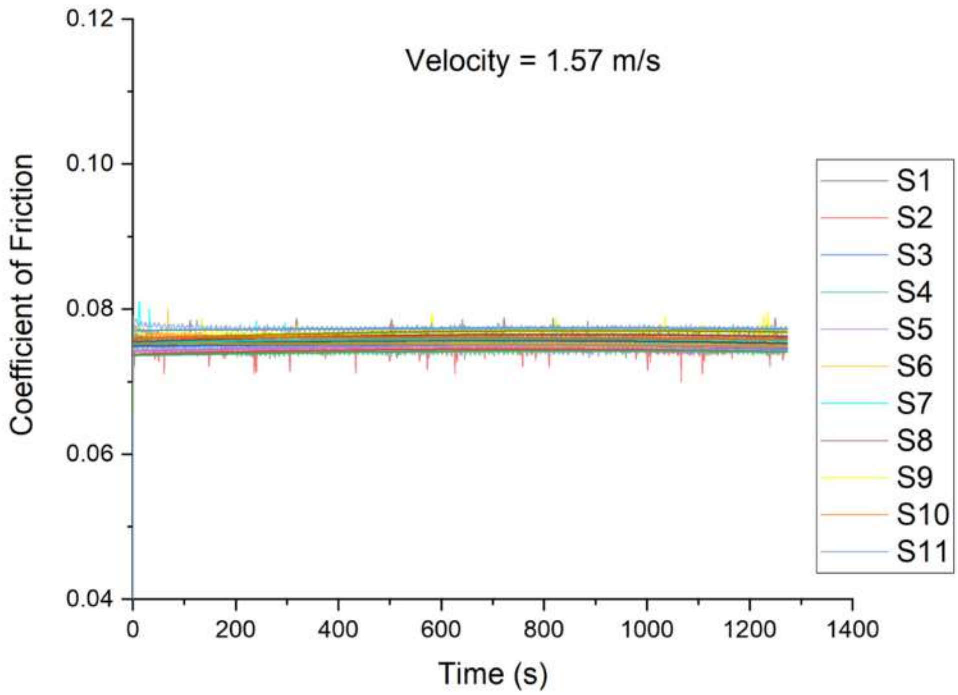
The tribological behaviour of lubricant is determined by analysing the wear and friction of a tribo-pair. In the presented study, the prepared apricot oil based biolubricant is used as a bio-additive in commercially available 15W40 mineral oil. Various oil samples were prepared with different volume-to-volume percentage (v/v) of Bio-additive and 15W40 oil, and further subject to tribometers for tribological behavioural analysis.

4.2.1 High Temperature Tribometer

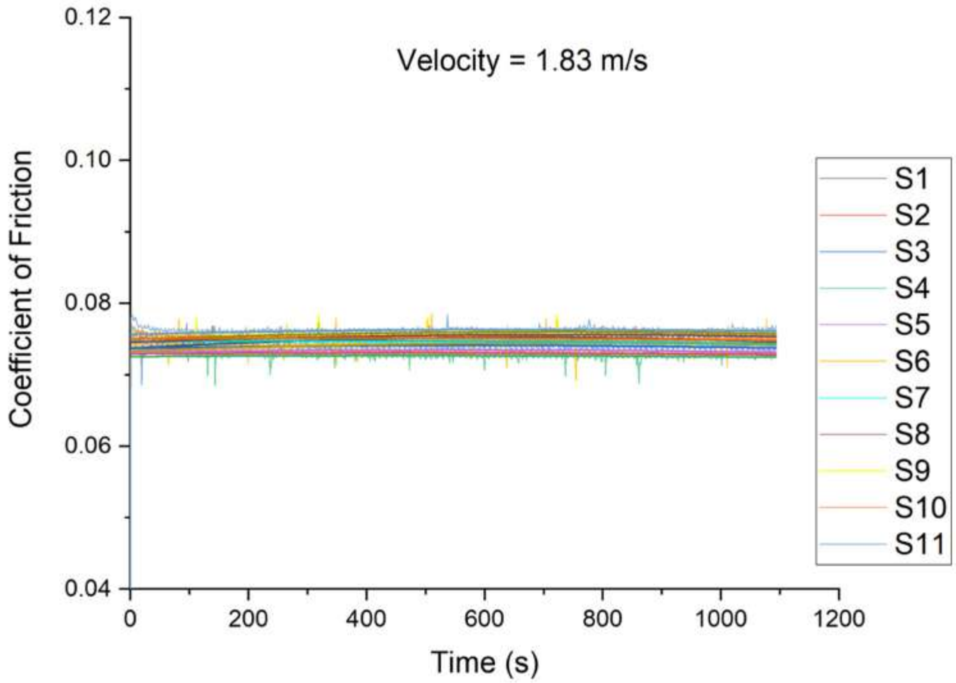
The coefficient of friction was analyzed over time, with its variation plotted against the sliding duration. The test data on the coefficient of friction was collected using a friction sensor connected to the pin-on-disc setup, integrated with data acquisition software. The Figure 26, 27 & 28 illustrates the relationship between the coefficient of friction and sliding time for various oil samples tested under different sliding speeds and temperature conditions.


Velocity = 1.30 m/s
Coefficient of Friction
0.12
0.10
0.08
0.06
0.04
0
200
400
600
800
1000
1200
1400
1600
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

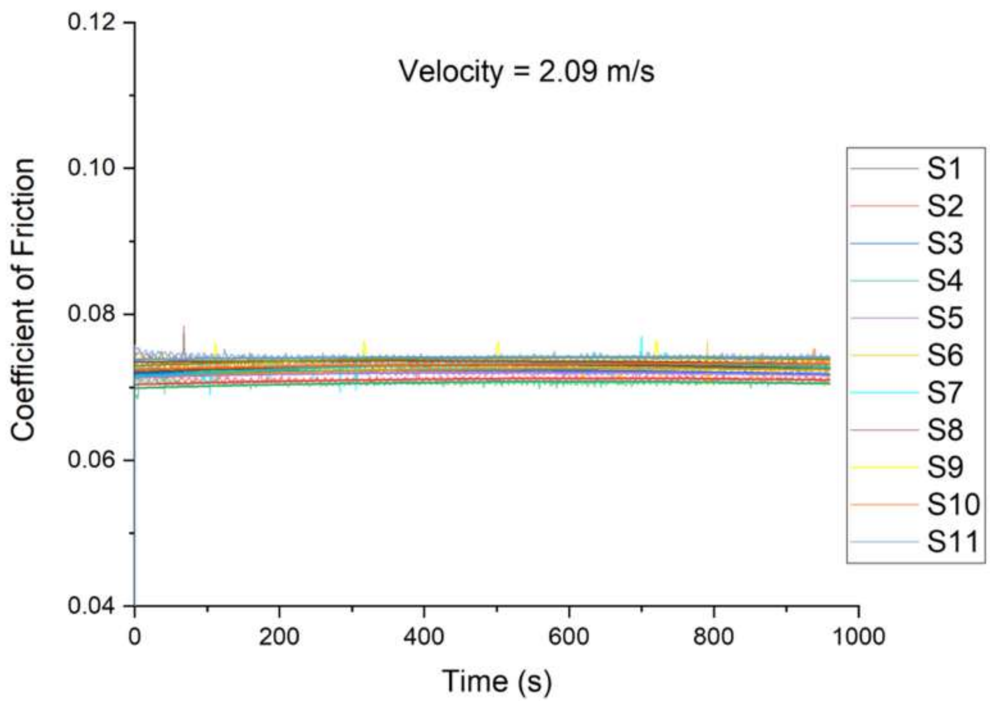
(a)


Velocity = 1.57 m/s
Coefficient of Friction
0.12
0.10
0.08
0.06
0.04
0
200
400
600
800
1000
1200
1400
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

(b)

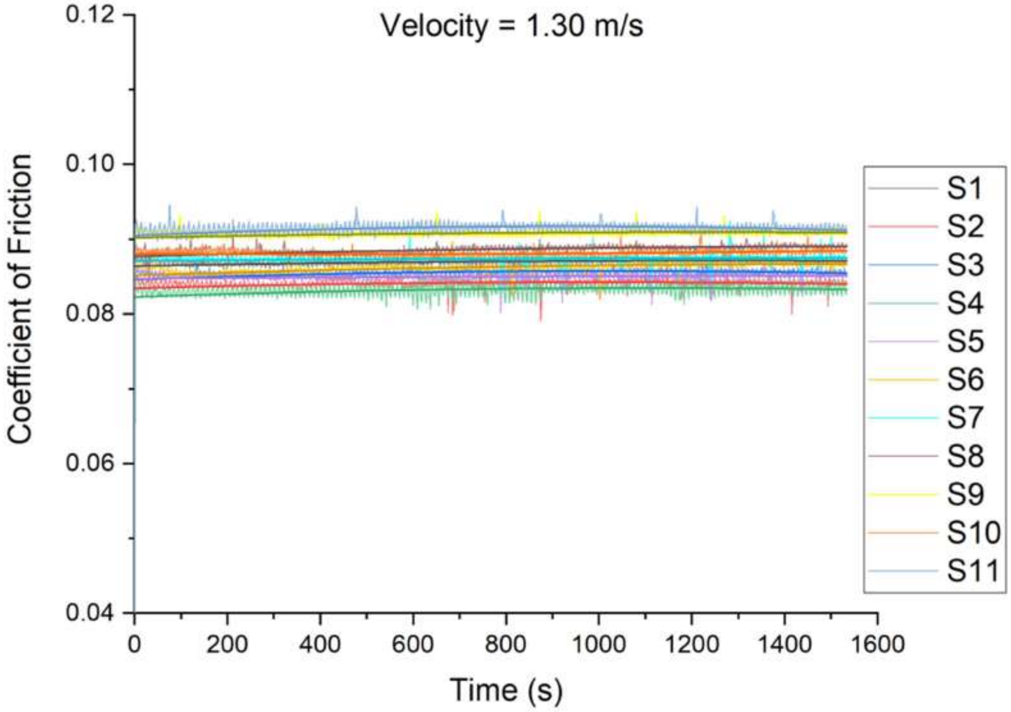


(c)

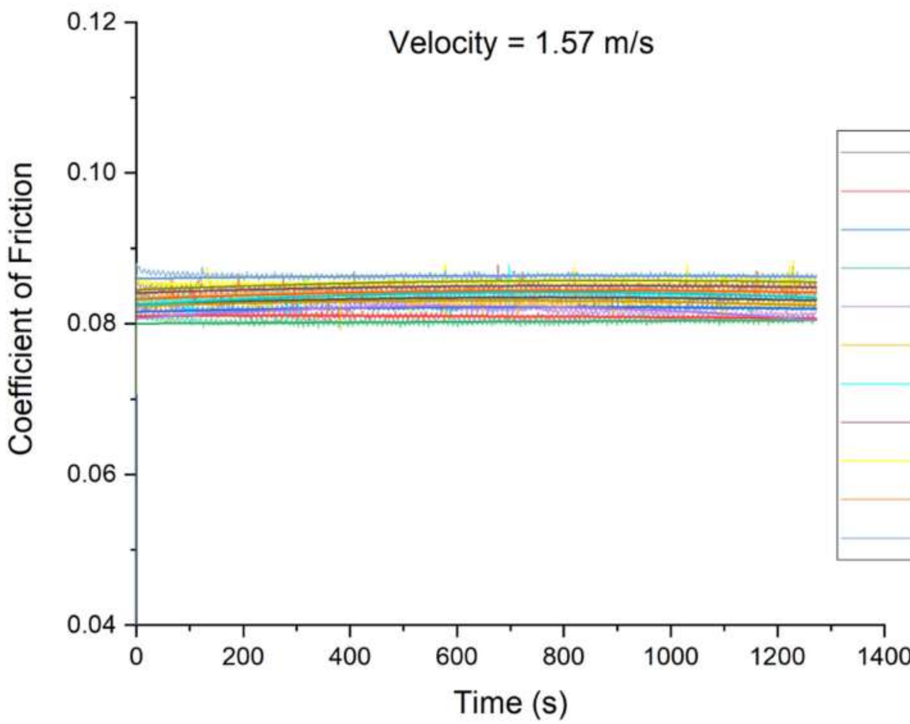


(d)

Figure 26: Variation of Coefficient of Friction against sliding time at 40°C, measured at different sliding speeds: (a) 1.30 m/s, (b) 1.57 m/s, (c) 1.83 m/s, and (d) 2.09 m/s.


Velocity = 1.30 m/s
Coefficient of Friction
Time (s)
0.12
0.10
0.08
0.06
0.04
0
200
400
600
800
1000
1200
1400
1600
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

(a)


Velocity = 1.57 m/s
Coefficient of Friction
Time (s)
0.12
0.10
0.08
0.06
0.04
0
200
400
600
800
1000
1200
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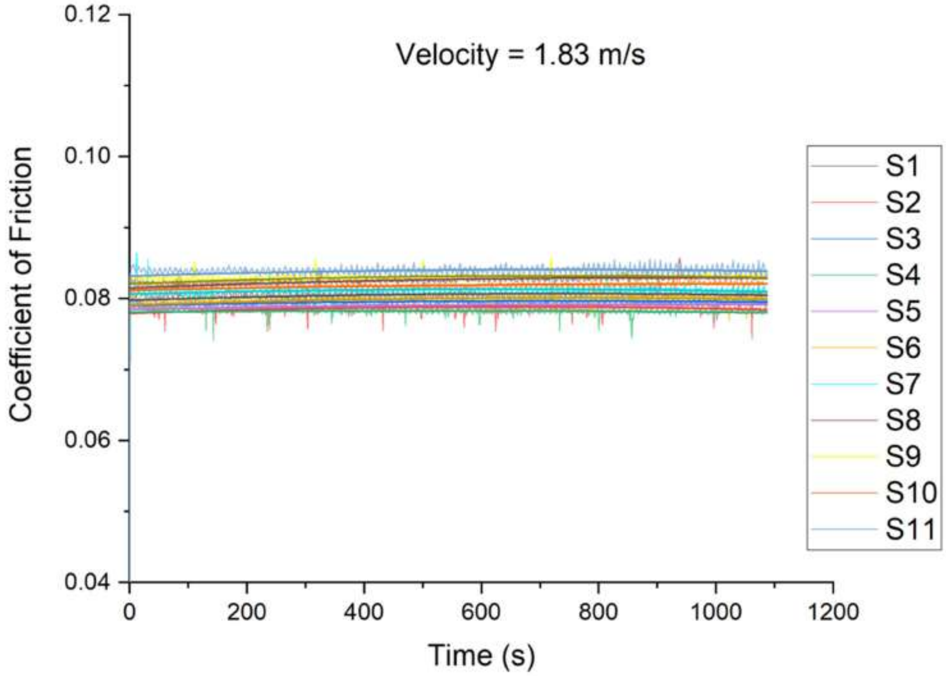
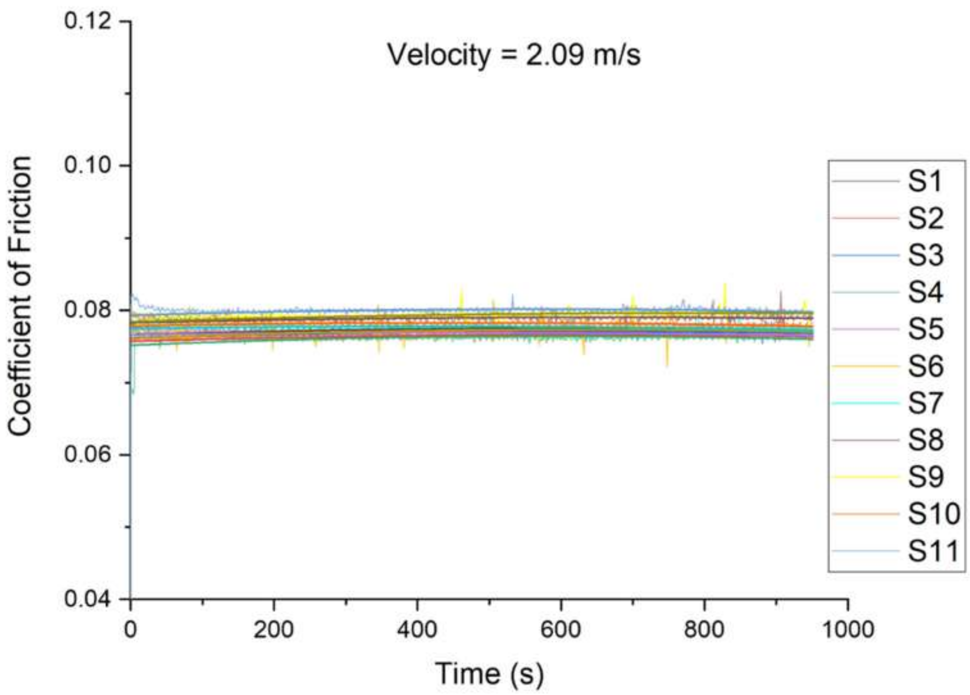
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

(b)

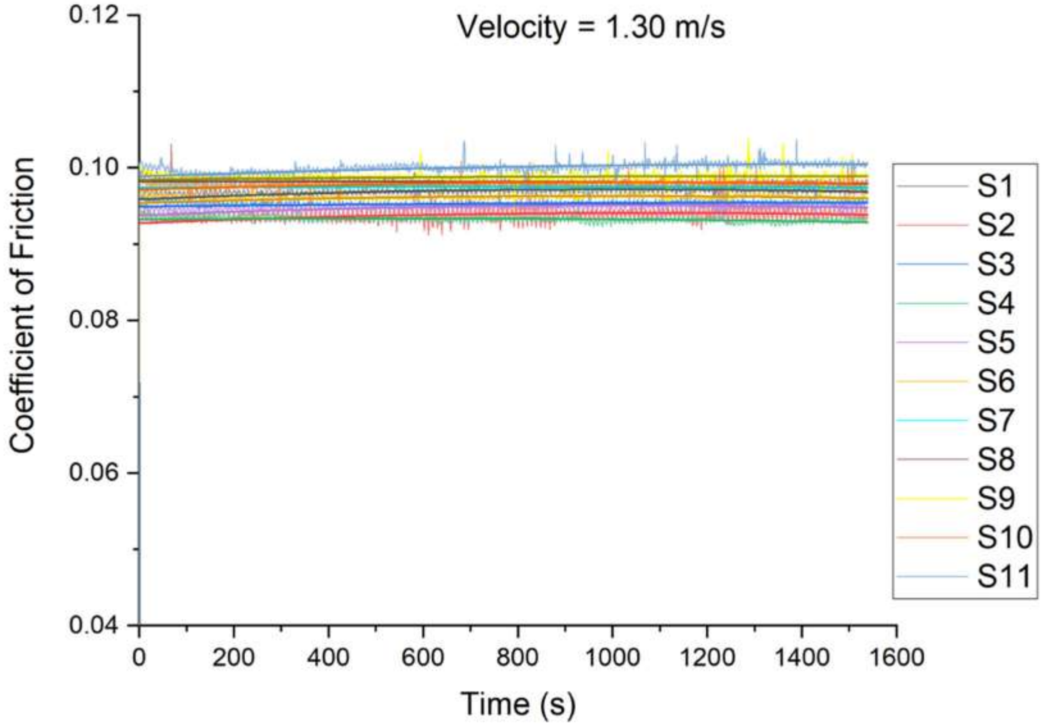


(c)

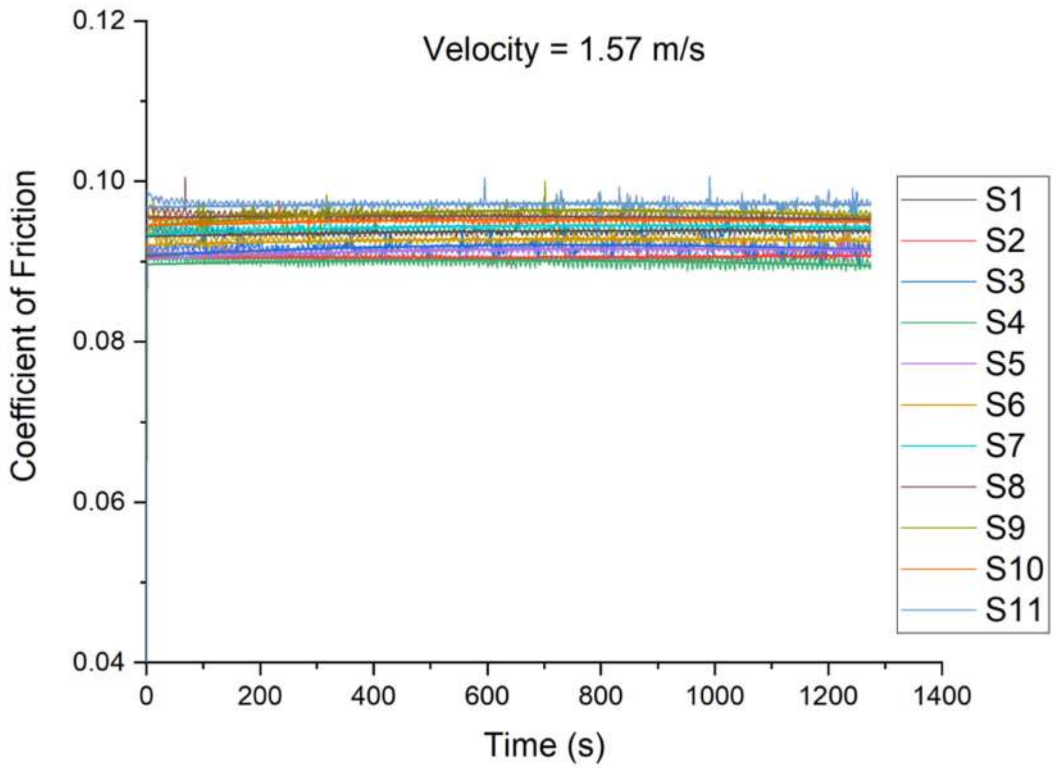


(d)

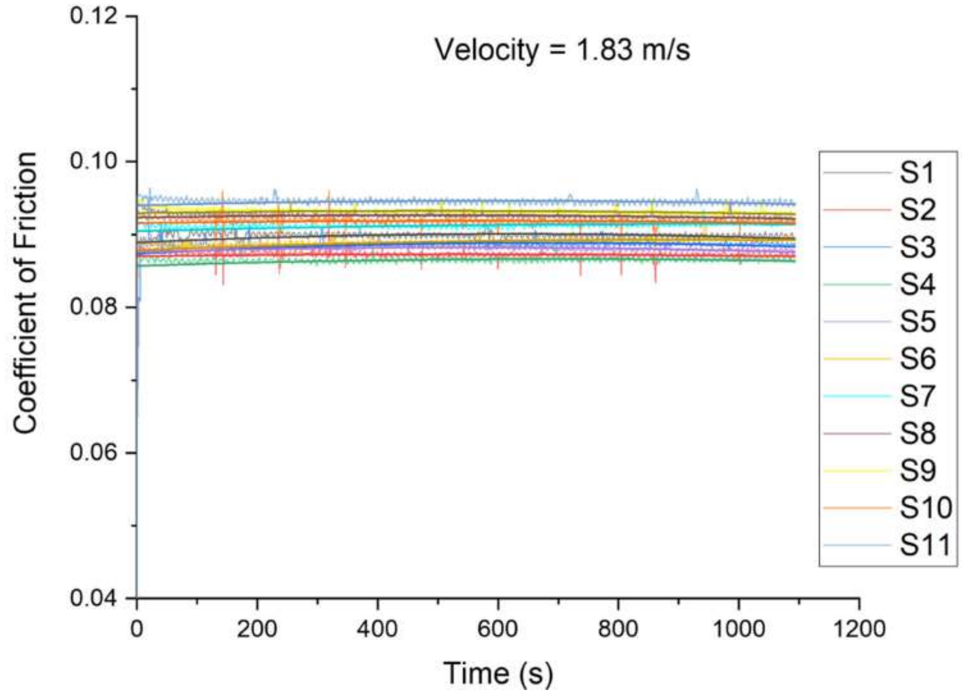
Figure 27: Variation of Coefficient of Friction against sliding time at 100°C, measured at different sliding speeds: (a) 1.30 m/s, (b) 1.57 m/s, (c) 1.83 m/s, and (d) 2.09 m/s.


Velocity = 1.30 m/s
Coefficient of Friction
Time (s)
0.12
0.10
0.08
0.06
0.04
0
200
400
600
800
1000
1200
1400
1600
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

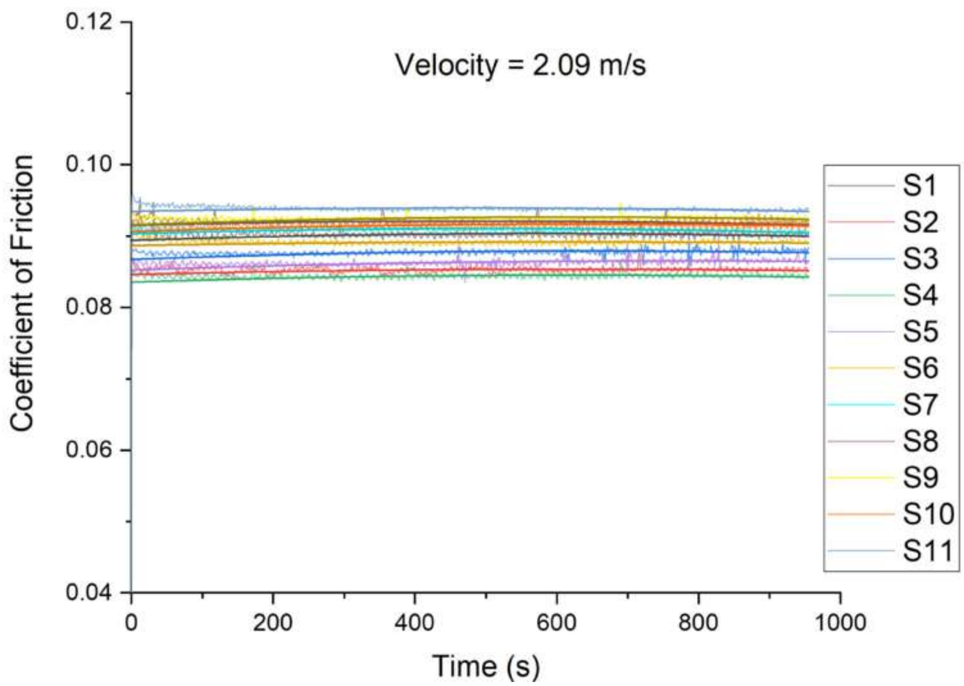
(a)


Velocity = 1.57 m/s
Coefficient of Friction
Time (s)
0.12
0.10
0.08
0.06
0.04
0
200
400
600
800
1000
1200
1400
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

(b)



(c)



(d)

Figure 28: Variation of Coefficient of Friction against sliding time at 150°C, measured at different sliding speeds: (a) 1.30 m/s, (b) 1.57 m/s, (c) 1.83 m/s, and (d) 2.09 m/s.

In the above shown graphs, a curve of best fit was drawn for each oil sample to make the analysis simpler. It is observed from the Figure 26, 27 & 28, that coefficient of friction peaks at the onset of the test run, gradually declining to its minimum value. This behaviour can be elucidated by the formation of an oxide layer on the sliding surfaces. The lubricant sample interacts with this oxide layer, leading to the development of a thick tribo-film on the sliding surface. Initially, the shear stress of this tribo-film is high, resulting in a correspondingly high coefficient of friction. As the sliding continues, the cast iron pin undergoes erosion, exposing fresh metal surfaces. Due to the reduced tendency of fresh metal surfaces to react with the lubricant, the rate of tribo-film formation decreases. Consequently, the shear stress diminishes compared to the initial phase, resulting in lower frictional forces. Thus, the coefficient of friction decreases with prolonged sliding time (Imran et al., 2013). It is further observed that the blend of 30% v/v of prepared bio-additive and 70% v/v of 15W40 mineral oil is the optimum blend as at this blend minimum coefficient of friction was observed. The coefficient of friction for S4 oil sample was reduced by 3.5% with respect to coefficient of friction for 15W40 mineral oil.

The apricot oil-based biolubricant exhibits tribological properties comparable to those of mineral oil. As the temperature rises, the coefficient of friction for the biolubricant also increases. This is because higher temperatures reduce the viscosity of the biolubricant, resulting in a thinner lubricating film between the contacting surfaces. The diminished film thickness reduces lubrication effectiveness and heightens friction. Conversely, as sliding velocity increases, the coefficient of friction decreases. At higher sliding speeds, the biolubricant spreads more uniformly over the contact area, enhancing the formation of a stable lubricating film. The increased velocity also drives the biolubricant into the contact zone, leading to better separation of the surfaces, reduced direct contact, and, consequently, a lower coefficient of friction.

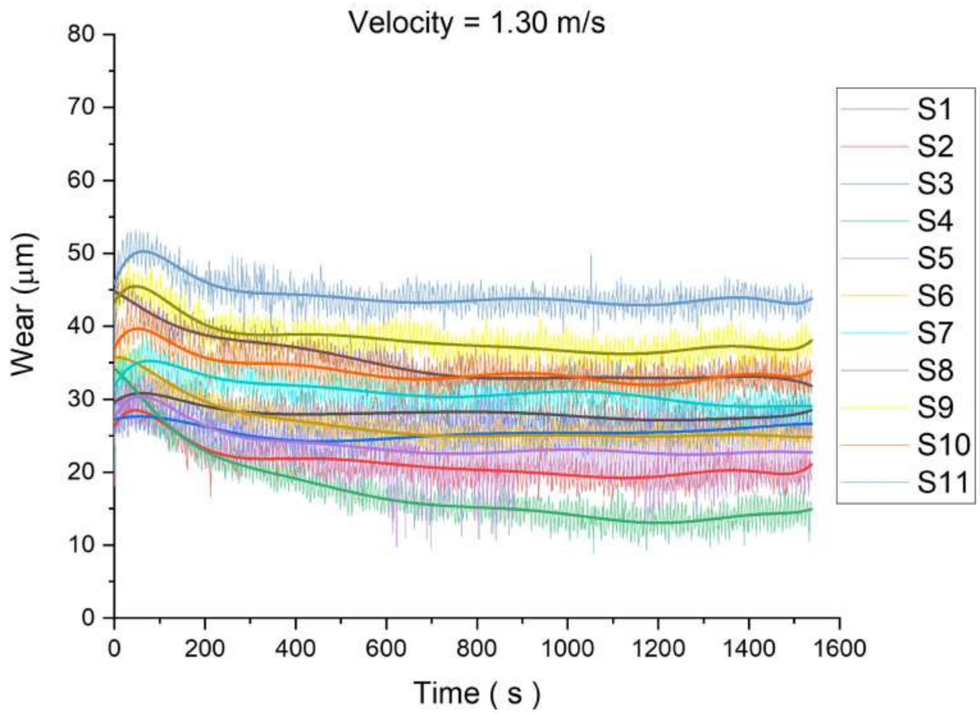
Figure 29, 30 and 31, shows the variation of wear of pin against sliding time for various oil samples subjected to different sliding speed and temperature condition. It is observed that the maximum pin wear occurs during the initial stage of testing, known as running-in period. Running-in phase refers to the initial test time when the wear rate is high. The sliding surface's asperities were eliminated during the running-in phase and the contact area to its equilibrium size. After some time passed, tribo-pair surfaces achieved an equilibrium wear condition, which caused the wear to stabilise. As the testing temperature rises, the wear on the pin also increases. This occurs because the viscosity of the test lubricant decreases at higher temperatures. A lower viscosity results in the thinning of the lubricant film, leading to more frequent asperity contacts, which in turn accelerates surface wear. The blending of apricot oil based biolubricant in 15W40 oil has better wear resistance compared to the individual content of a blend. This indicates that the fatty acid constituents of the biolubricant formed both singular and multiple layers on the rubbing zone's surface, generating a durable film that prevented contact between the tribo-pairs. The biolubricant may enhance the overall viscosity and film strength of the blend, hence creating a more robust lubrication film that reduces direct contact between surfaces, thereby minimizing wear between tribo-pairs.

4.2.2 Statistical Analysis

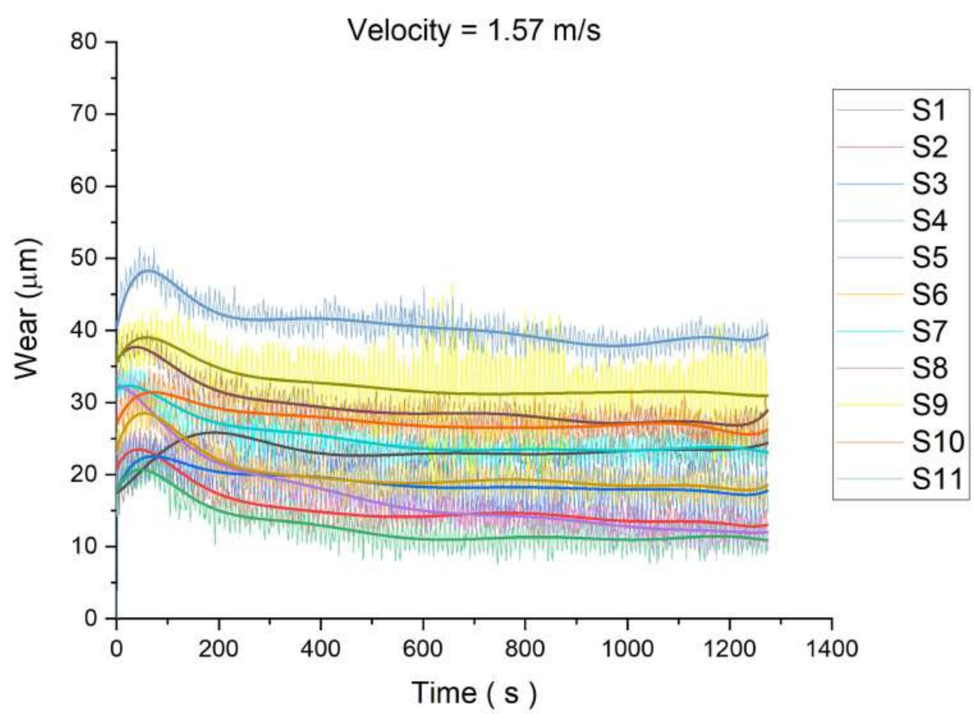
The primary goal of ANOVA analysis is to investigate how different combinations of mixing function levels, utilizing a full factorial design, impact the overall variance in the results, namely, wear and coefficient of friction.

Table 13 presents the results of ANOVA performed on wear data. The analysis highlights Velocity (V) as the primary factor influencing wear, contributing significantly with a 35.70%. Following closely is Temperature (T), which exerts a substantial effect on the coefficient of friction, contributing 32.80%. In contrast, the interaction term (T × V) demonstrates minor contributions, each accounting for less than 1.71% of the variance. Consequently, it is reasonable to assume that other factors do not hold significant importance in this context. Furthermore, the presence of p-values below 0.05 in the model suggests the adequacy of the models and the statistical significance of the terms in influencing the responses, which is a desirable outcome.

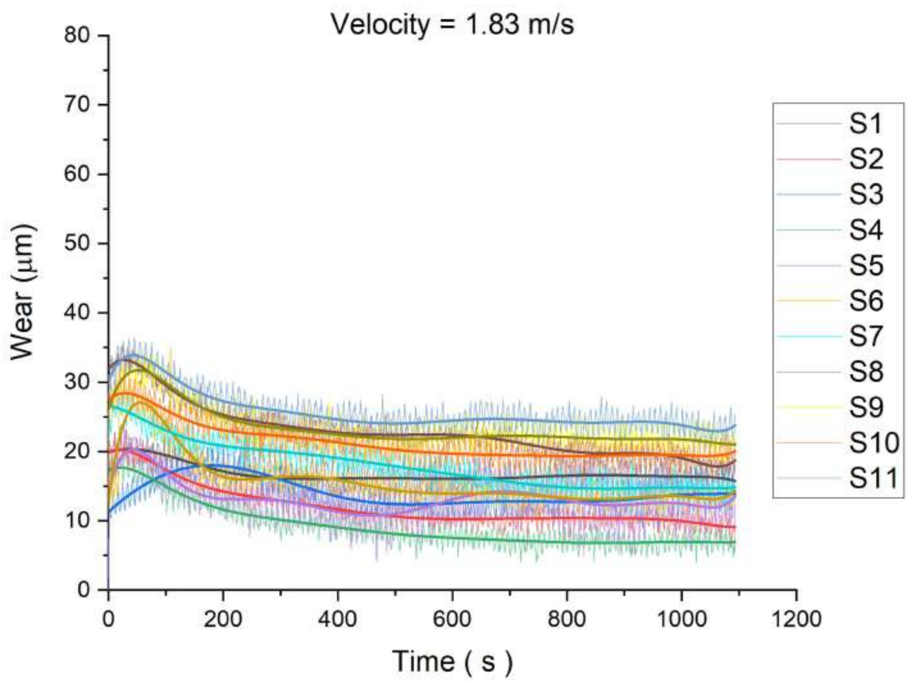
The model F-value of 271.21 suggests that the model is statistically significant, with only a 0.01% probability that such a high F-value could arise from random variation. P-values below 0.0500 demonstrate the significance of specific model terms. In this instance, the significant terms are A, B, C, AB, AC, A^2, and B^2. On the other hand, P-values above 0.1000 indicate that the model terms are not statistically significant.


Velocity = 1.30 m/s
Wear (μm)
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11
0
10
20
30
40
50
60
70
80
0
200
400
600
800
1000
1200
1400
1600

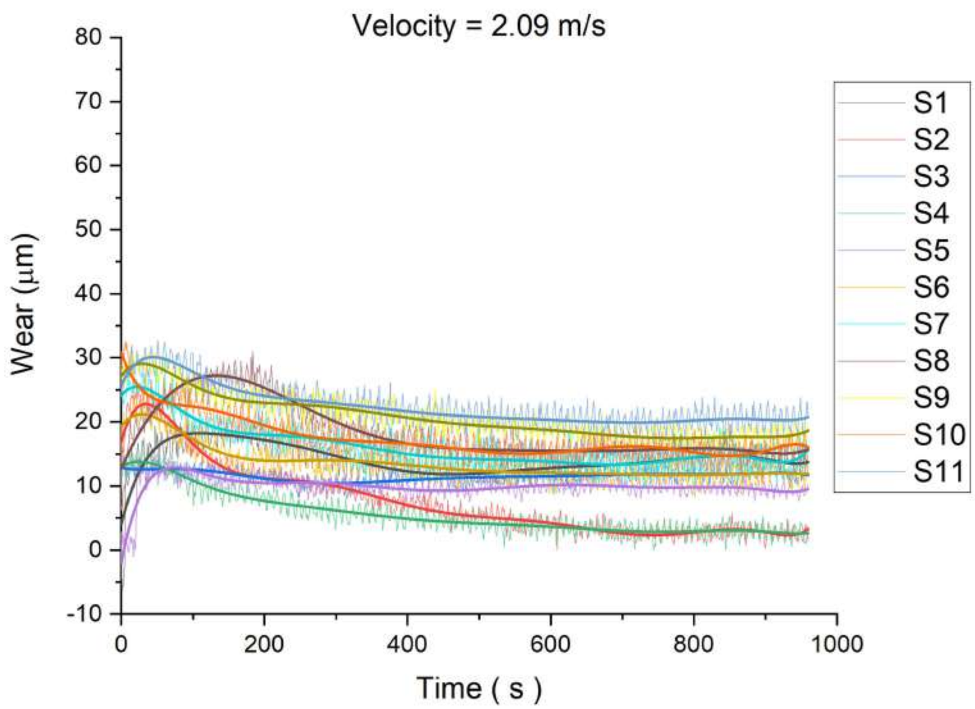
(a)


Velocity = 1.57 m/s
Wear (μm)
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11
0
10
20
30
40
50
60
70
80
0
200
400
600
800
1000
1200
1400

(b)

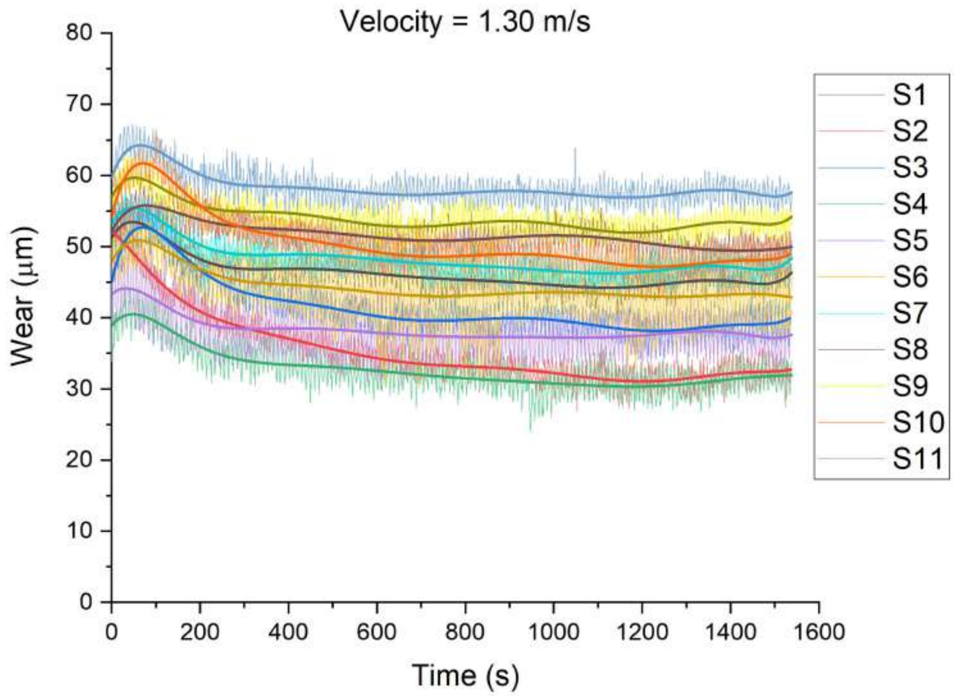


(c)

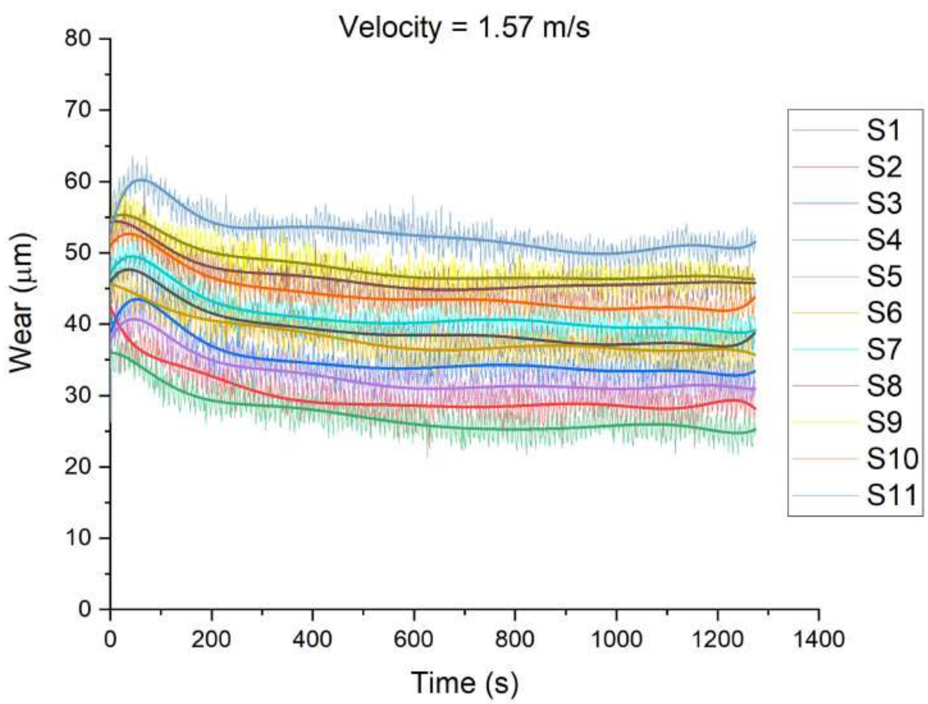


(d)

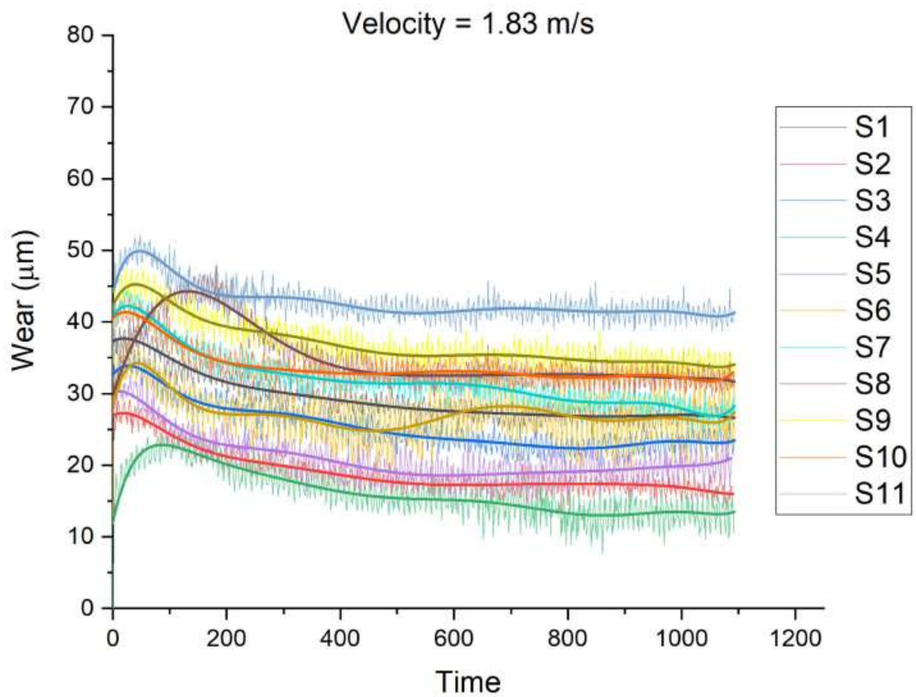
Figure 29: Variation in the wear over sliding time at 40°C, measured at different sliding speeds: (a) 1.30 m/s, (b) 1.57 m/s, (c) 1.83 m/s, and (d) 2.09 m/s.


Velocity = 1.30 m/s
Wear (μm)
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

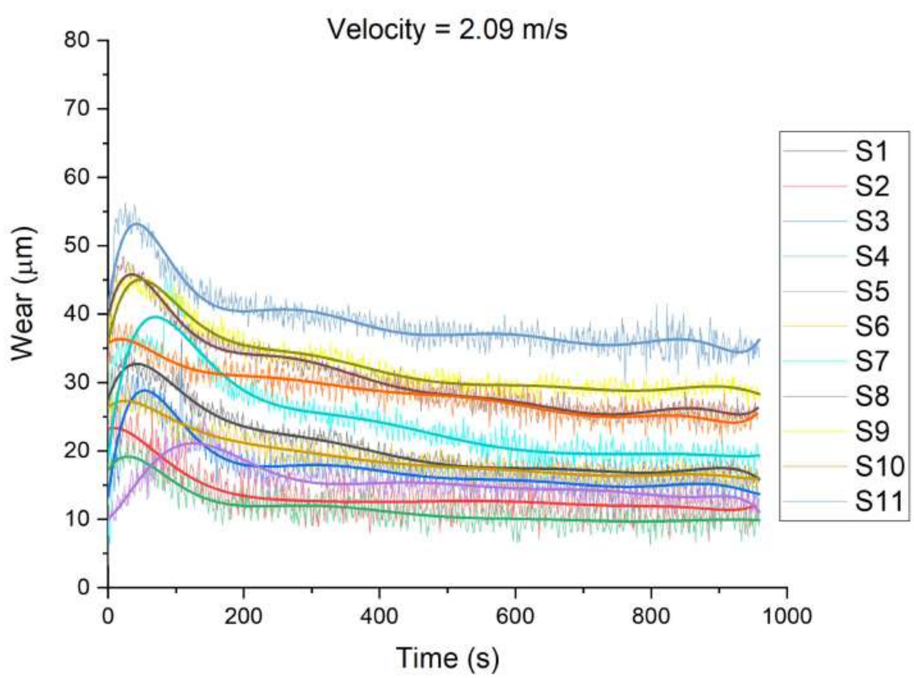
(a)


Velocity = 1.57 m/s
Wear (μm)
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

(b)

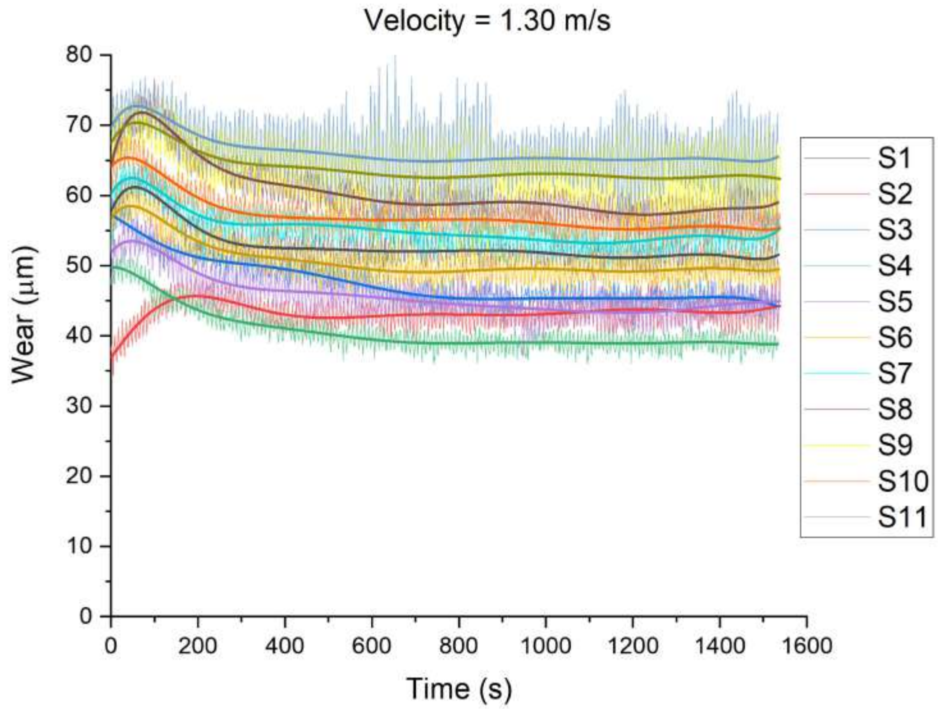


(c)

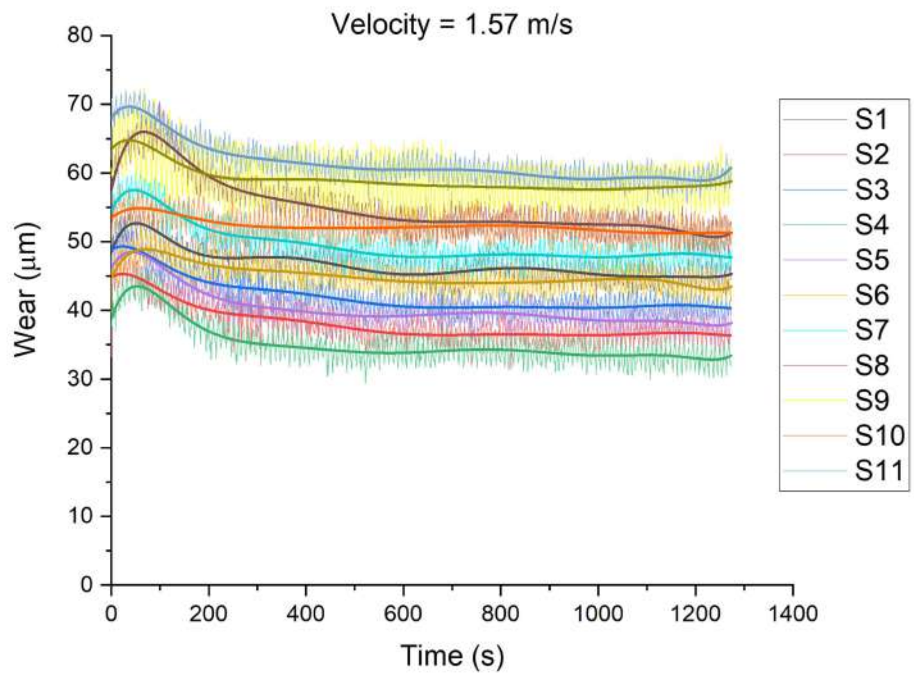


(d)

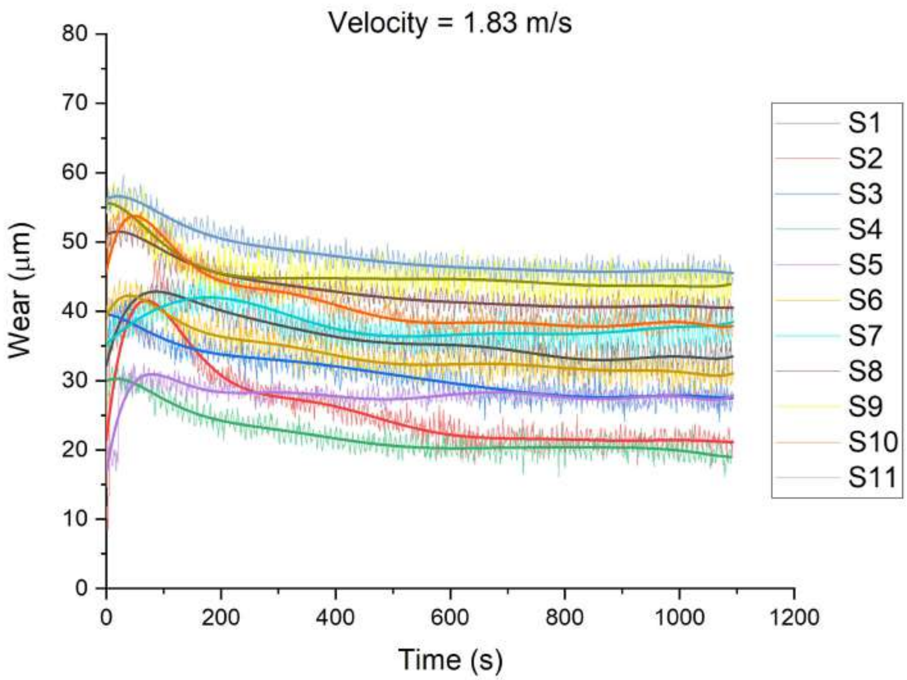
Figure 30: Variation in the wear over sliding time at 100°C, measured at different sliding speeds: (a) 1.30 m/s, (b) 1.57 m/s, (c) 1.83 m/s, and (d) 2.09 m/s.


Velocity = 1.30 m/s
Wear (μm)
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

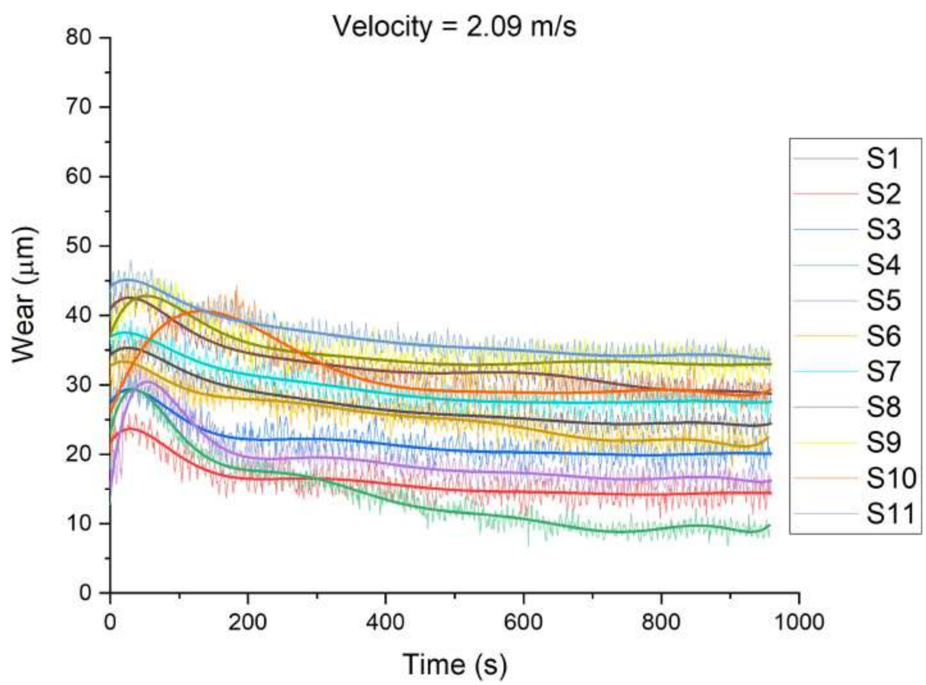
(a)


Velocity = 1.57 m/s
Wear (μm)
Time (s)
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11

(b)



(c)



(d)

Figure 31: Variation in the wear over sliding time at 150°C, measured at different sliding speeds: (a) 1.30 m/s, (b) 1.57 m/s, (c) 1.83 m/s, and (d) 2.09 m/s.

Table 13: ANOVA results of Wear

Source	Sum of Squares	df	Mean Square	F-value	p-value	% Contri	
Model	25391.82	35	725.48	271.21	< 0.0001		Significant
A-Temperature	8405.45	1	8405.45	3142.31	< 0.0001	32.80%	Significant
B-Velocity	9161.01	1	9161.01	3424.77	< 0.0001	35.70%	Significant
C-Sample	7087.83	10	708.78	264.97	< 0.0001	27.60%	Significant
AB	439.16	1	439.16	164.18	< 0.0001	1.71%	Significant
AC	72.19	10	7.22	2.70	0.0059	0.281%	Significant
BC	24.72	10	2.47	0.9241	0.5147	0.0964%	Not significant
A^2	168.45	1	168.45	62.97	< 0.0001	0.657%	Significant
B^2	33.01	1	33.01	12.34	0.0007	0.129%	Significant
Residual	256.79	96	2.67				
Total	25648.61	131					

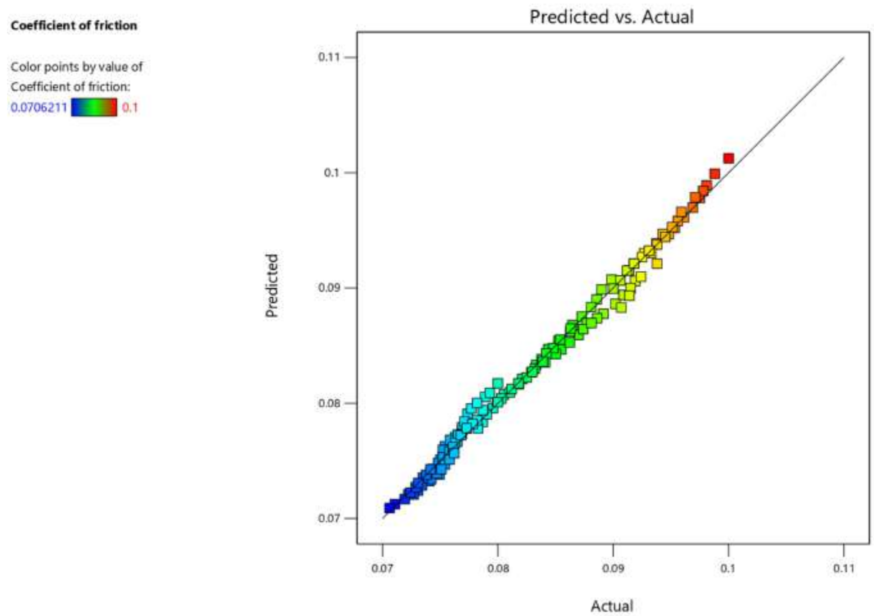
Table 14 displays the results of the ANOVA analysis concerning the coefficient of friction. Notably, the most influential factor impacting the coefficient of friction is Temperature (T), contributing a substantial 80.488%. Following closely, Velocity (V) emerges as the second most crucial factor, contributing a significant 10.976% to the variance in the coefficient of friction. In contrast, the interactions (T × V) exhibit minimal contributions, each accounting for less than 1.5% of the observed variation. This suggests that the other terms in the analysis may not hold significant weight. Crucially, the p-values associated with the model are all less than 0.05, signifying that the models are statistically robust and that the examined factors exert a significant influence on the responses, a highly desirable outcome in statistical analysis.

Table 14: ANOVA results of coefficient of friction

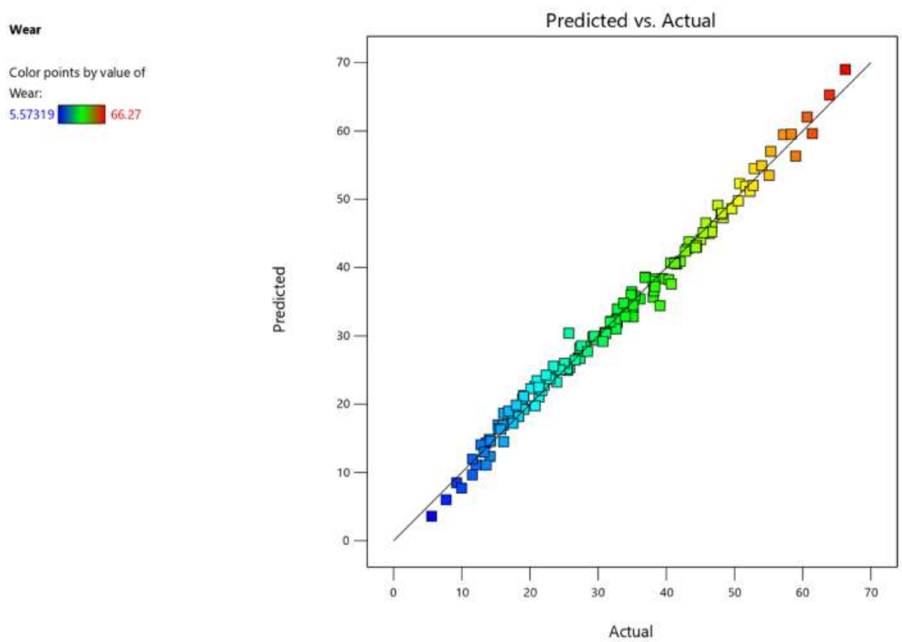
Source	Sum of Squares	df	Mean Square	F-value	p-value	% Contri	
Model	0.0082	35	0.0002	298.32	< 0.0001		Significant
A-Temperature	0.0066	1	0.0066	8476.95	< 0.0001	80.488%	Significant
B-Velocity	0.0009	1	0.0009	1146.53	< 0.0001	10.976%	Significant
C-Sample	0.0004	10	0.0000	52.54	< 0.0001	4.878%	Significant
AB	0.0000	1	0.0000	44.35	< 0.0001		Significant
AC	0.0000	10	3.825E-06	4.89	< 0.0001		Significant
BC	1.312E-06	10	1.312E-07	0.1679	0.9980	0.0161%	Not significant
A^2	0.0002	1	0.0002	194.84	< 0.0001	2.439%	Significant
B^2	1.902E-06	1	1.902E-06	2.43	0.1221	0.0232%	
Residual	0.0001	96	7.815E-07				
Total	0.0082	131					

Similarly, the model F-value of 298.32 confirms the model's significance, with a 0.01% likelihood of occurring due to noise. Significant terms in this case include A, B, C, AB, AC, and A^2. As before, terms with P-values over 0.1000 are considered insignificant.

In Figure 32, the graph illustrates the experimental values of both the coefficient of friction and wear, plotted against their corresponding predicted values. Upon careful analysis of the figure, it becomes evident that the points of convergence between the experimental data and the estimated values closely align with the central median line, which has a slope of 45 degrees. This alignment is particularly pronounced in the learning and validation sets, providing compelling evidence of the ANOVA model's efficacy.



(a)



(b)

Figure 32: Comparison between experimental and predicted values for (a) Coefficient of Friction and (b) Wear

Modelling by RSM

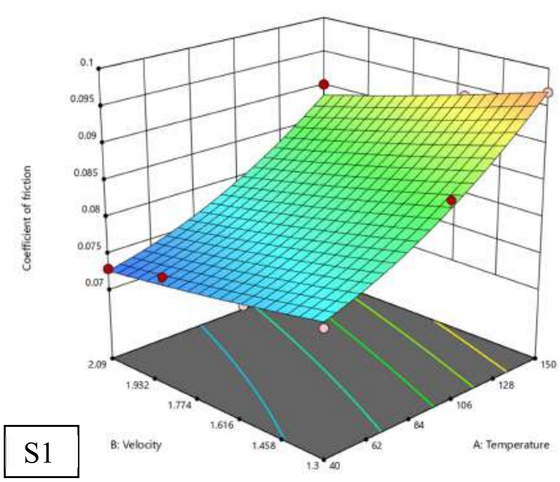
The relationships between the input factors and output parameters were represented using quadratic regression, as expressed in the equations 13 & 14 provided below.

$$\mathrm{CoF} = 0.086440 + 0.000080 * \mathrm{T} - 0.010963 * \mathrm{V} - 0.000038 * \mathrm{T} * \mathrm{V} + 7.60513 * 10^{-7} * \mathrm{T}^2 + 0.001801 * \mathrm{V}^2 \quad (13)$$

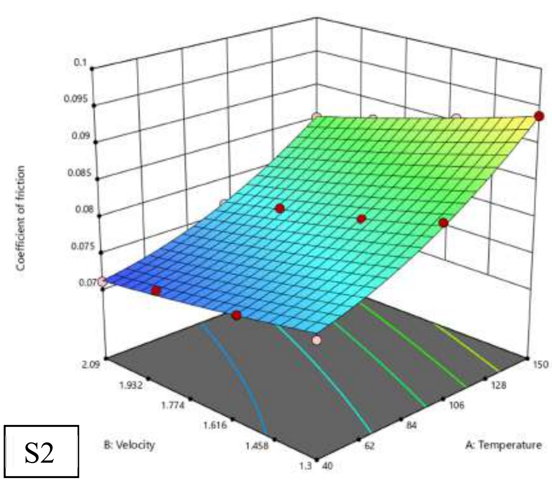
$$\mathrm{Wear} = 12.84003 + 0.566353 * \mathrm{T} + 10.64638 * \mathrm{V} - 0.135642 * \mathrm{T} * \mathrm{V} - 0.000800 * \mathrm{T}^2 - 7.50279 * \mathrm{V}^2 \quad (14)$$

Regression models are valuable tools for predicting how response parameters vary in relation to input control parameters. To gain deeper insights into the interplay between variables and their impact on response factors, 3D plots were generated representing the measured responses and contour graphs using the equations from models 1 and 2.

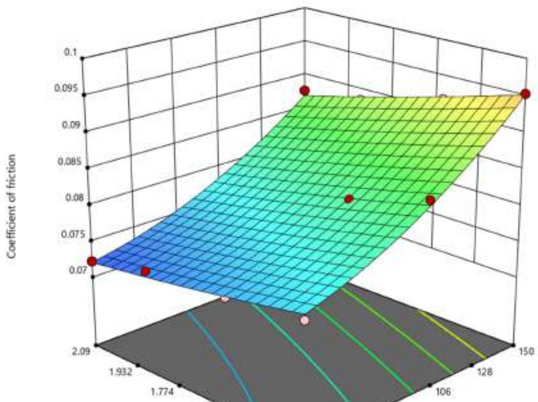
From Figure 33, it has been observed that the temperature has dominant effect on coefficient of friction as compared to velocity. This observation is true for all the lubricant samples. On the other hand, from Figure 34, it has been observed that, for all lubricant samples, velocity has dominant effect on wear of tribo-pair as compared to temperature.



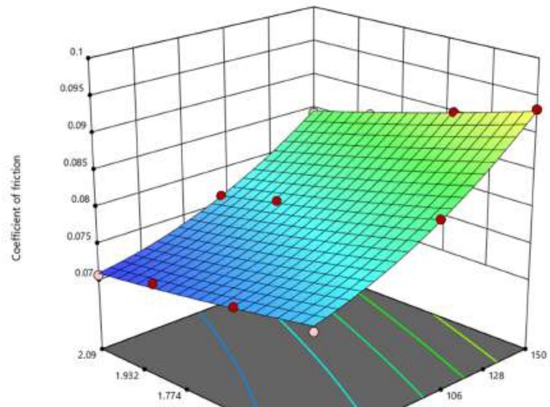
S1



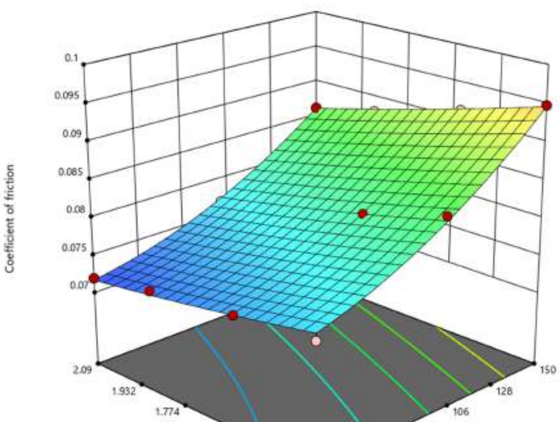
S2



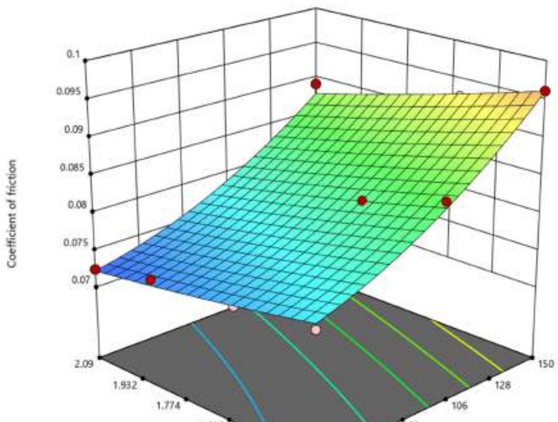
S3



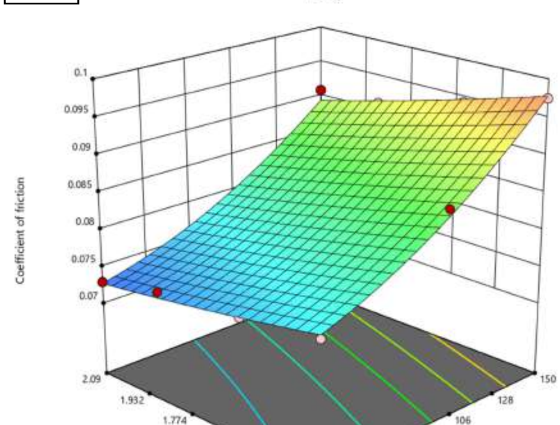
S4



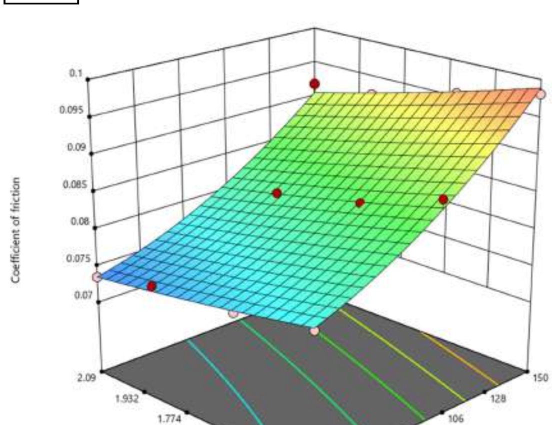
S5



S6



S7



S8

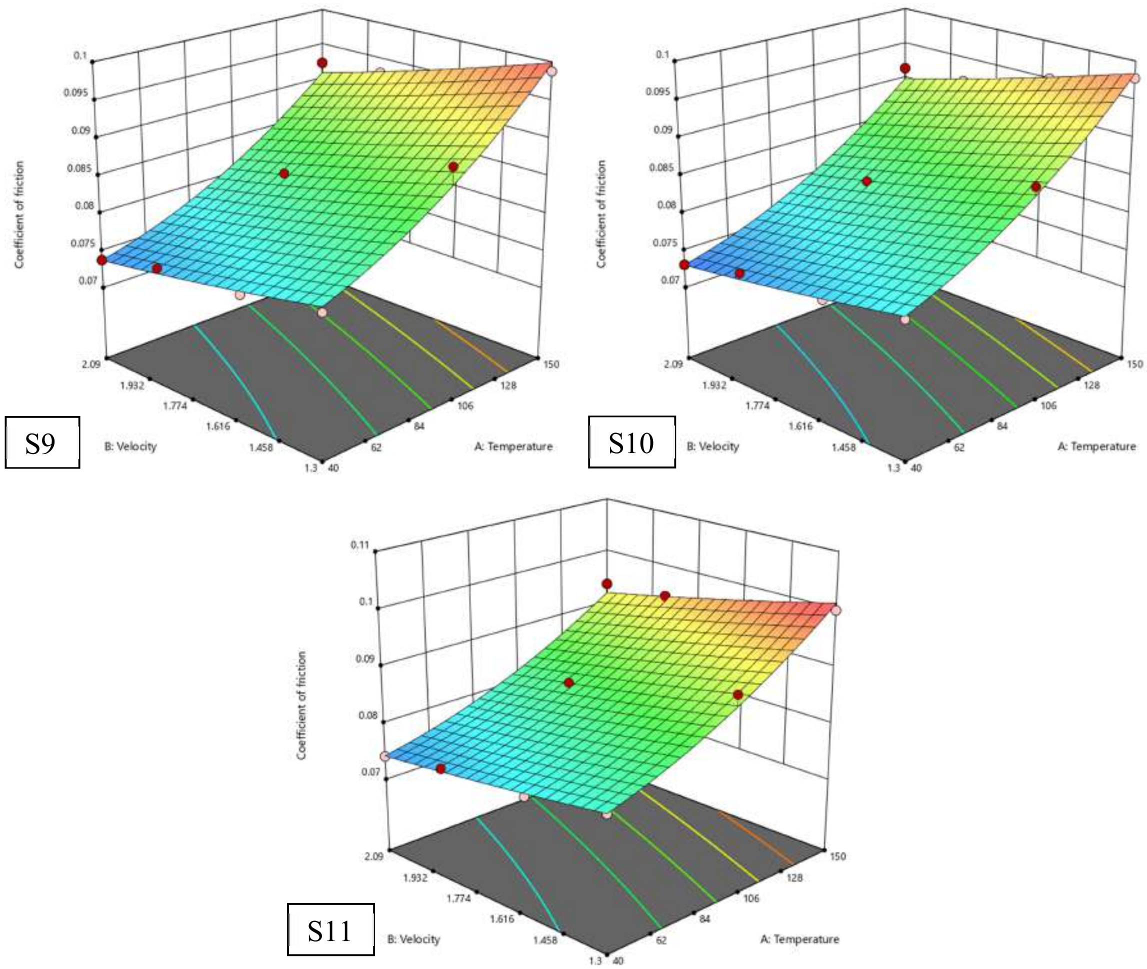
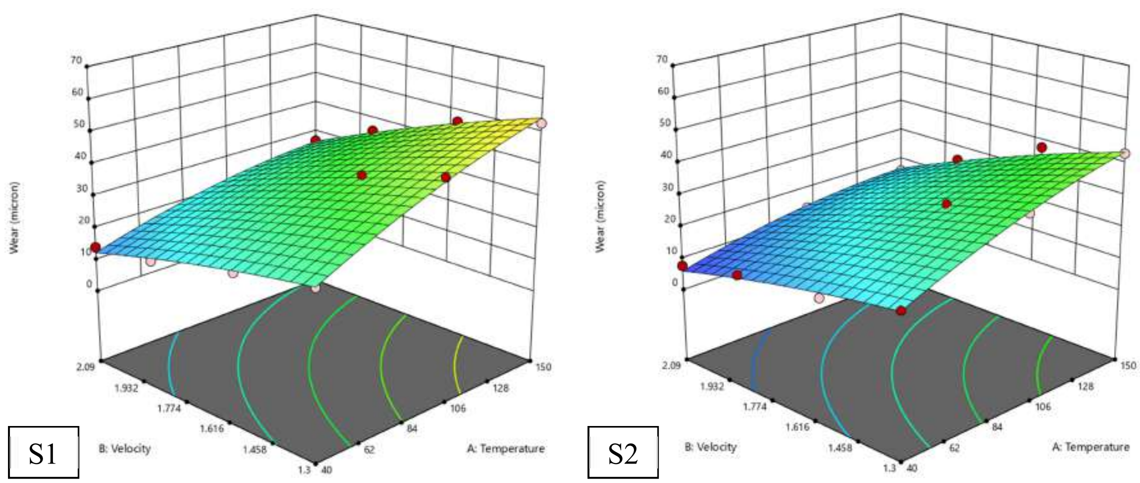
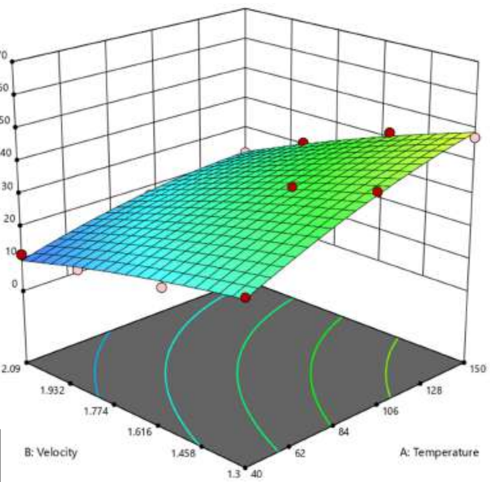
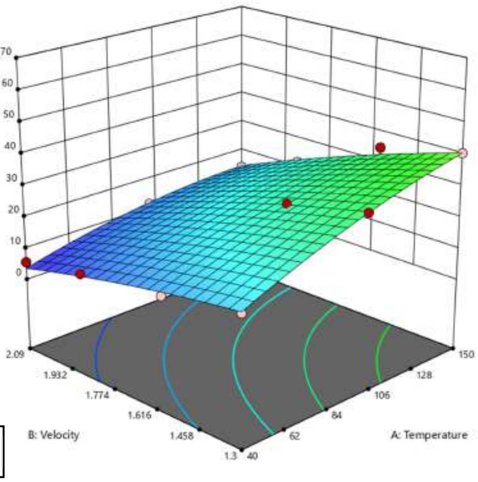


Figure 33: Response surface for coefficient of friction depending upon the temperature and velocity for different lubricant samples.

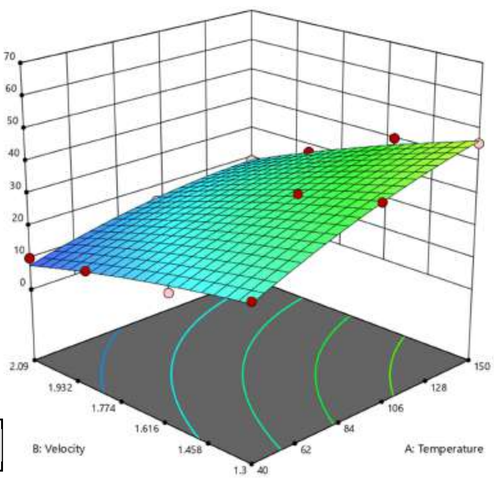




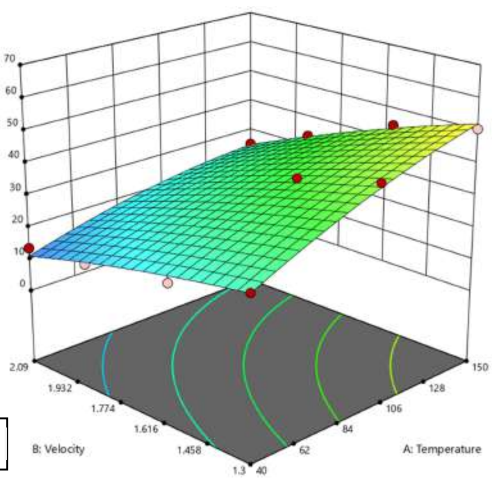
S3



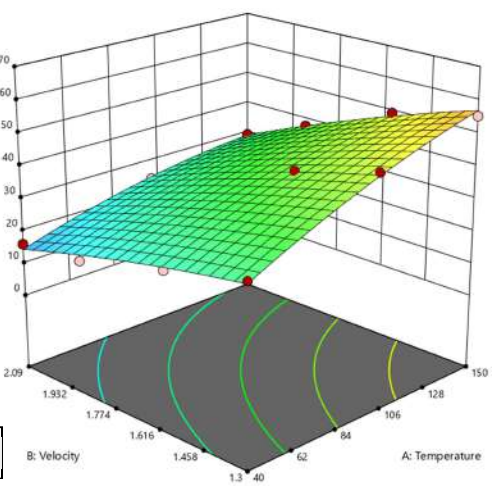
S4



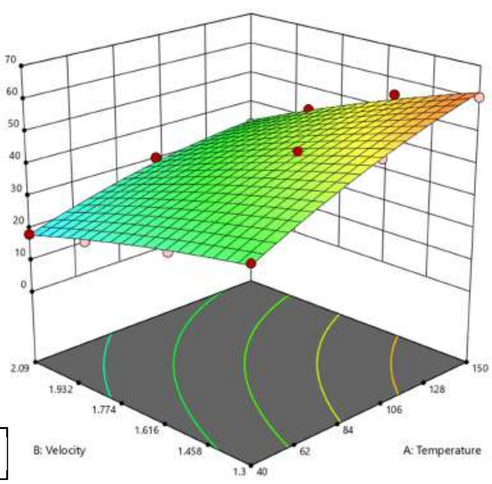
S5



S6



S7



S8

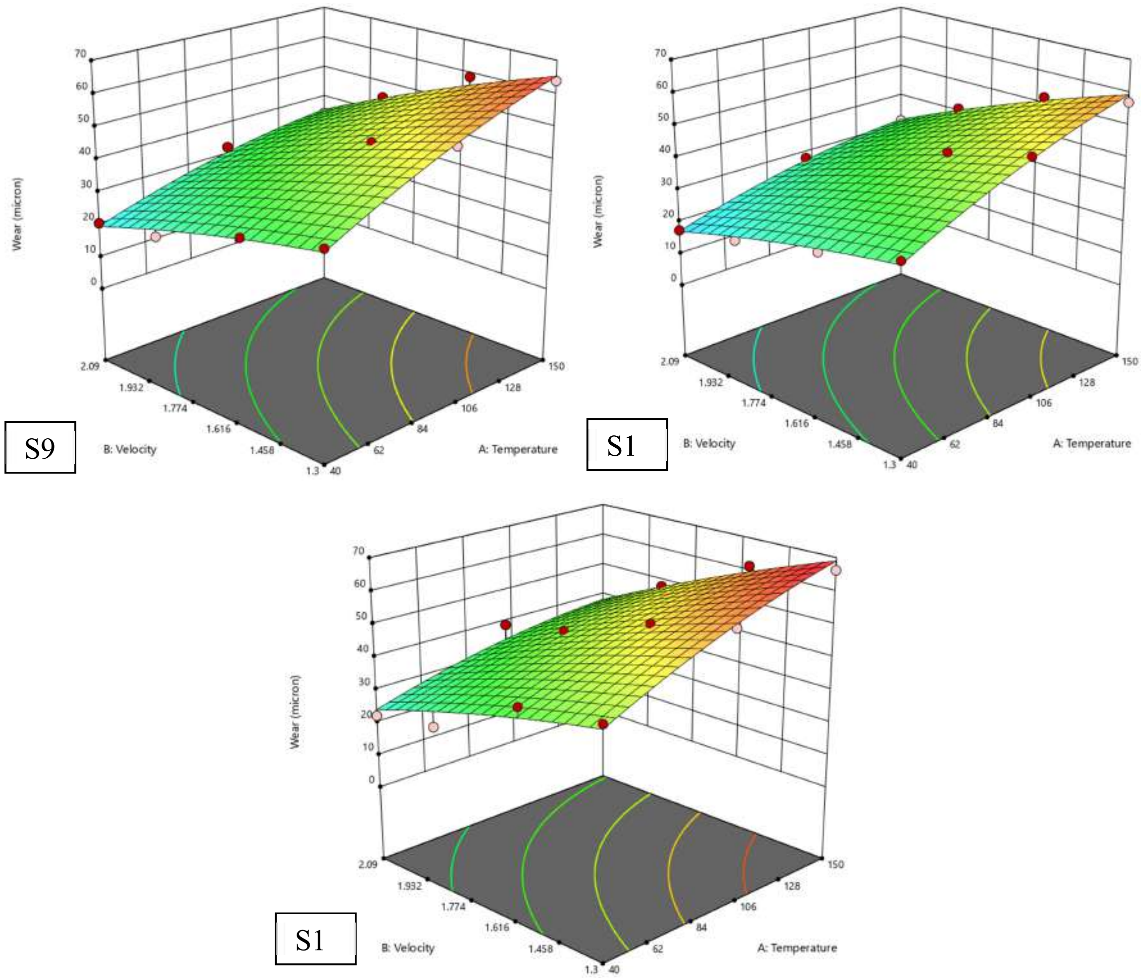


Figure 34: Response surface for wear depending upon the temperature and velocity for different lubricant samples.

4.2.3 Four Ball tester

Tribological testing of the oil samples was conducted using a four-ball tester in compliance with ASTM D4172 standards to analyse the friction and wear properties of the lubricants. The compositions of the oil samples are detailed in the Table 4. During testing, a load is applied vertically downwards and the top ball rotates at a constant speed. The assembly's temperature is maintained at the desired level with a temperature-controlled unit. Frictional torque was recorded through a load displacement sensor, with the results displayed on an online monitor. The experimental test conditions for various oil samples on a four-ball tester are shown in Table 8.

Study of coefficient of friction and wear

The applied loads for the test were 196 N and 392 N, and the experiment was carried out on samples labeled S1 through S11. From Figure 35(a), it is evident that under an applied load of

196 N, the coefficient of friction (CoF) for sample S4 i.e. comprising of 70% 15W40 oil and 30% apricot oil-based biolubricant, was approximately 12.93% lower compared to sample S1 i.e. 100% 15W40, and 43.19% lower compared to sample S11 i.e. 100% apricot oil based biolubricant.

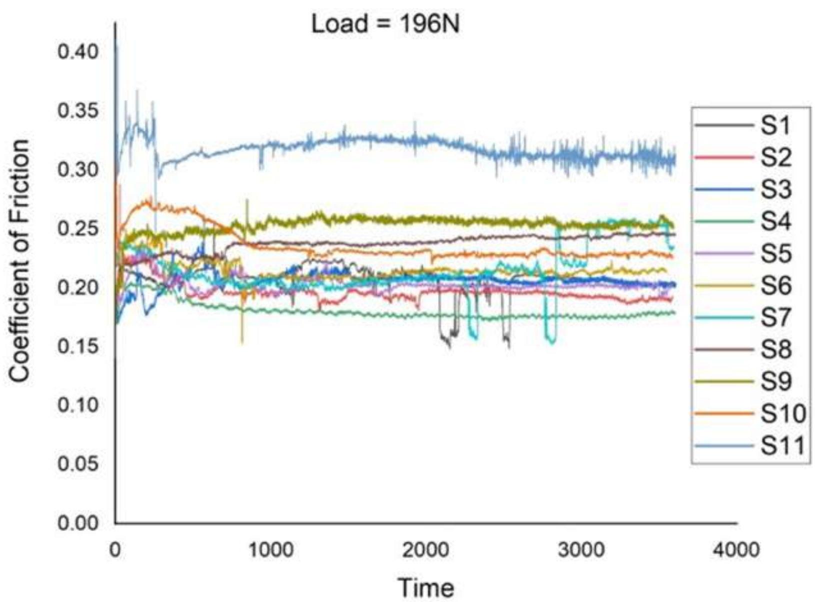
A similar trend was observed in Figure 35(b) under a higher applied load of 392 N. For this condition, the CoF for sample S4 was reduced by about 9.71% compared to S1 and 35.08% compared to S11. These results demonstrate that adding a specific proportion of apricot oil-based biolubricant as an additive to commercially available 15W40 oil effectively reduces the coefficient of friction. This finding underscores the potential of this mixture to enhance lubrication performance.

Figures 36 and 37 illustrate the variation in the volume wear rate and the wear scar diameter (WSD) of the test balls subjected to different oil samples, labelled S1 through S11, under two different normal loads: 196N and 392N. The data reveal that the average scar diameter observed on the test balls is smaller under the lower load (196N) compared to the higher load (392N). Notably, sample S4 exhibited the lowest average scar diameter under both loading conditions.

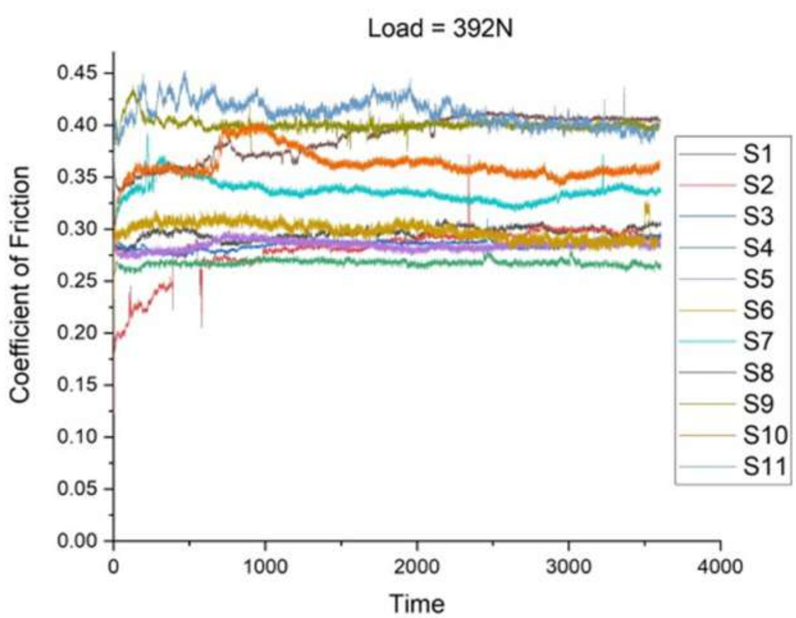
The volume wear rate, which quantifies the volume of metal lost per unit of time during testing, provides a crucial measure of wear resistance. The findings indicate that incorporating a bio-lubricant as an additive in 15W40 oil significantly reduces both the wear scar diameter and the volume wear rate. The wear scars on the test balls were analyzed using an optical microscope, as shown in Figure 38. This figure presents the microscopic views of test balls for samples S1 (100% 15W40 oil with no bio-lubricant), S4 (a blend of 70% 15W40 oil and 30% bio-lubricant), and S11 (100% bio-lubricant) under both 196N and 392N loading conditions.

The analysis demonstrates that the addition of 30% apricot oil-based bio-lubricant to 70% conventional SAE 40 oil (sample S4) substantially improves wear performance. Under a load of 392N, this formulation reduces the average wear scar diameter by 36.6% compared to pure SAE 40 oil (S1) and by 58.5% compared to pure apricot oil-based bio-lubricant (S11). Similarly, under a load of 196N, the average wear scar diameter decreases by 40.6% and 58.5% compared to S1 and S11, respectively. These results underscore the effectiveness of blending bio-lubricants with conventional oils in enhancing wear resistance.

The observed reduction in the coefficient of friction and wear when incorporating apricot oil-based biolubricant as an additive in 15W40 oil can be credited to the natural lubricating properties of vegetable oils. These oils contain long-chain fatty acid molecules that form a protective boundary layer on metal surfaces, minimizing direct metal-to-metal contact. Additionally, the polar nature of vegetable oil molecules enhances their ability to adhere firmly to metal surfaces, resulting in a durable and effective lubricating film. This reduces wear by minimizing surface asperity interactions under load. When blended with 15W40 oil, vegetable oil can enhance the overall performance by combining the thermal stability of mineral oil with the superior boundary lubrication properties of vegetable oil.



(a)



(b)

Figure 35: Behaviour of coefficient of friction for various oil samples under normal load of (a) 196N and (b) 392N

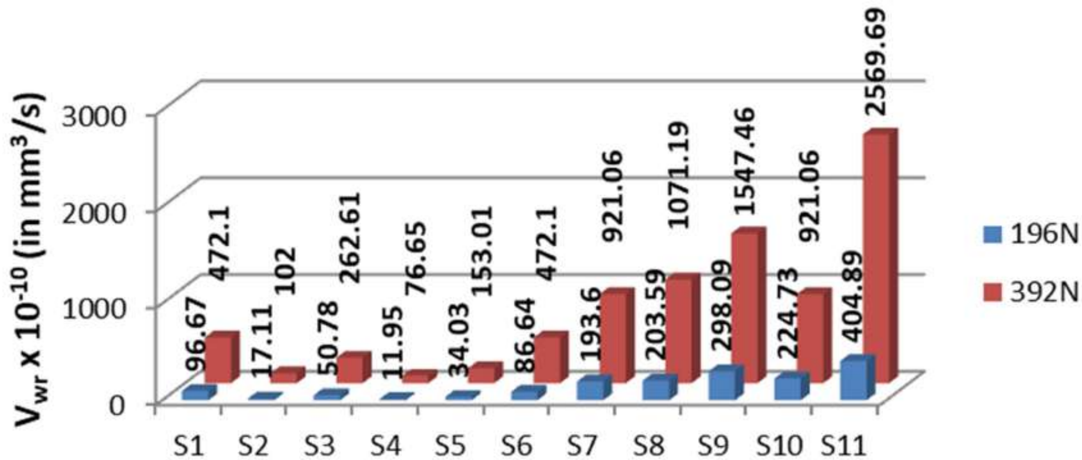


Figure 36: Volume wear rate for different oil samples at 196N and 392N load

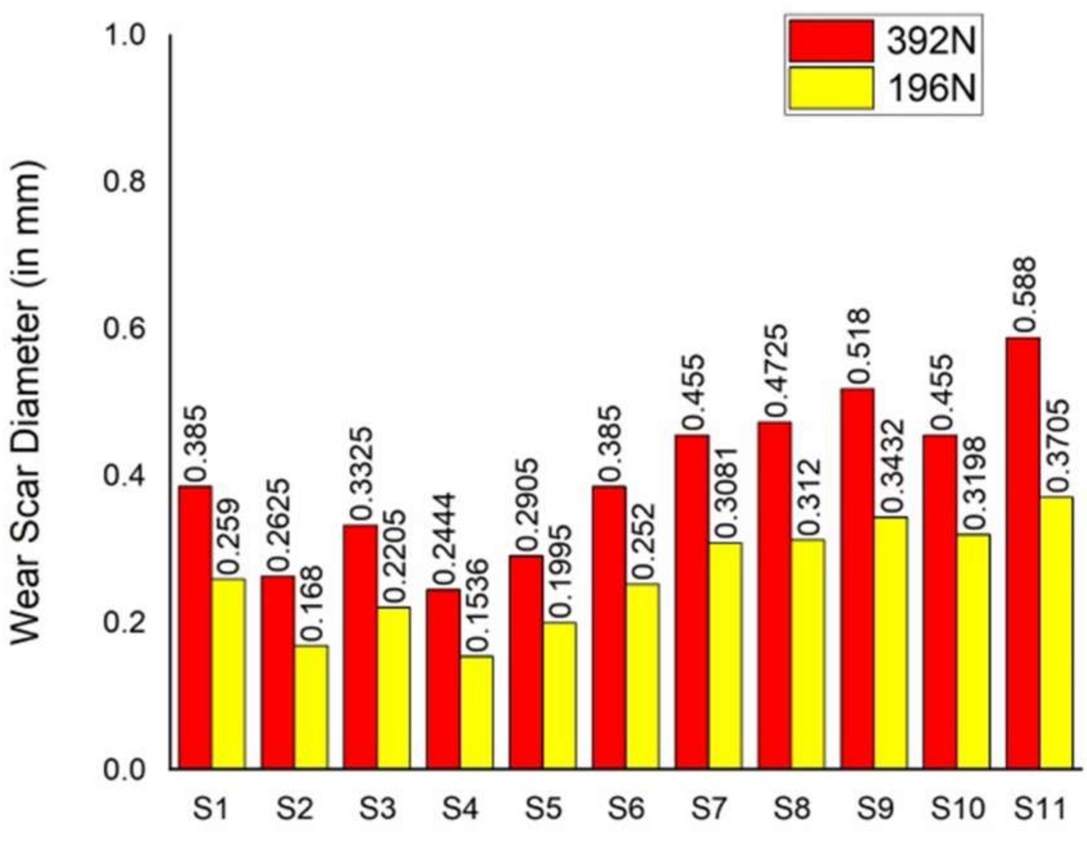


Figure 37: Average wear scar diameter for different oil samples under load of 196 N and 392 N

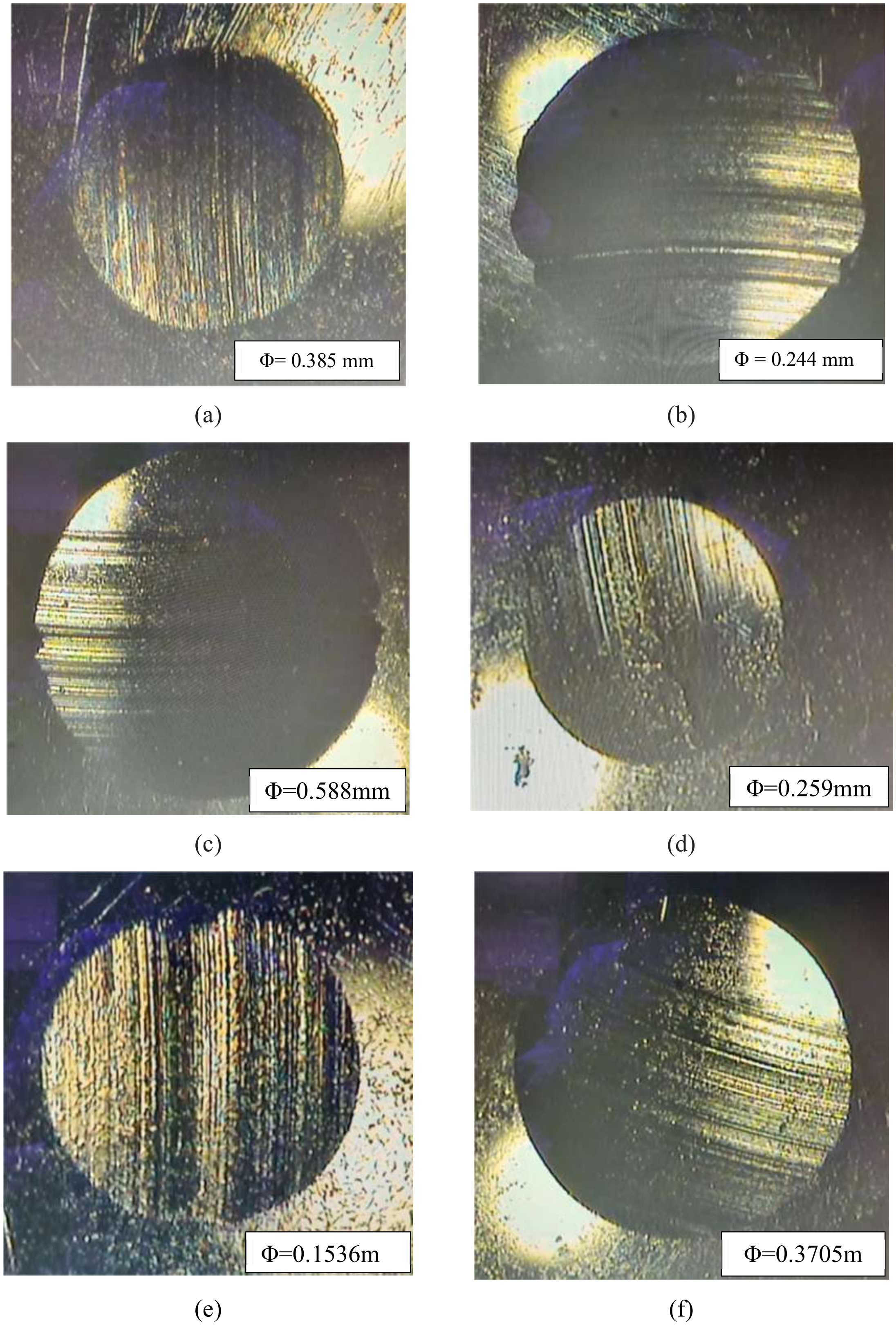


Figure 38: Optical image of wear scar diameter on test balls (a) S1 at 392N, (b) S4 at 392N, (c) S11 at 392N and (d) S1 at 196N, (e) S4 at 196N and (f) S11 at 196N.

Flash temperature parameter (FTP) is the numerical parameter used to menifest critical temperature above which a lubricant layer under testing will fail under a given load condition. The FTP value increases as the load increases, as shown in Figure 39. It is also inferred that a higher value of FTP indicates better stability of lubricant under given loading conditions. Sample S4 i.e. 70% of 15W40 + 30% of biolubricant, has the highest FTP value under both 196N and 392N loading conditions; hence it has better lubrication stability. Therefore sample S4 shows minimum wear scar diameter (WSD) under both loading conditions. Whereas Sample S11 i.e. 0% of 15W40 + 100% of biolubricant, has a minimum value of FTP under both loading conditions, indicating the breakdown of lubricant film thickness, hence maximum wear scar diameter for sample S11.

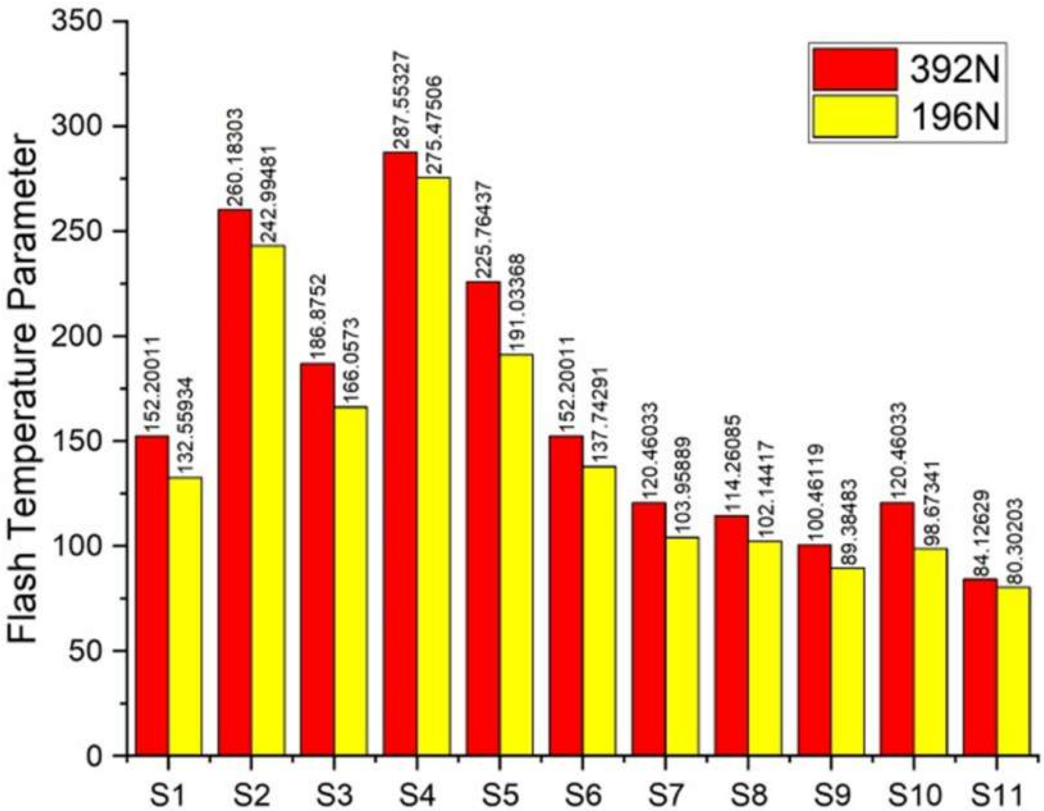


Figure 39: Flash Temperature Parameter for different oil samples under load of 196N and 392N

4.3 Effect of Lubricant blends with bio-additive in the Journal Bearing

The performance of a journal bearing system is significantly influenced by factors such as the type of lubricant oil used, the load applied to the shaft, and the shaft rotational speed. In the present analysis, the performance of the journal bearing was assessed using various lubricant samples, as detailed in Table 15. In this section of the chapter, the friction torque and maximum pressure have been evaluated for hydrodynamic lubrication regime.

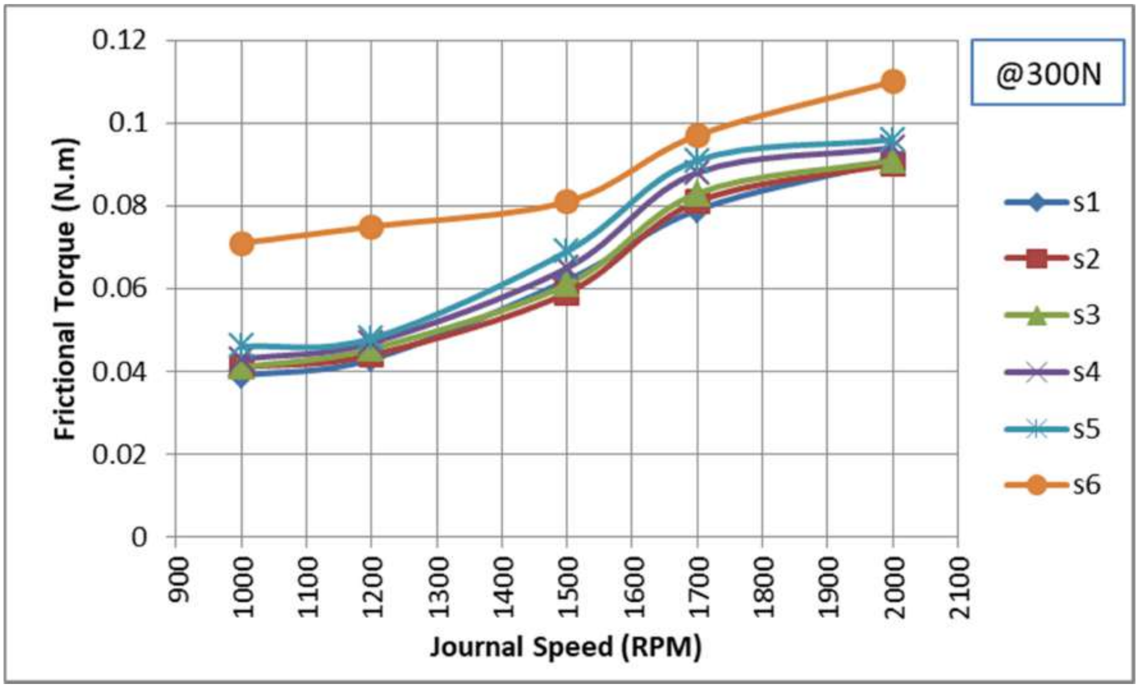
Table 15: Composition of oil samples for Journal bearing test

	S1	**S2**	**S3**	**S4**	**S5**	**S6**
Bio-Lube %	0	10	20	30	40	100
15W40 oil %	100	90	80	70	60	0

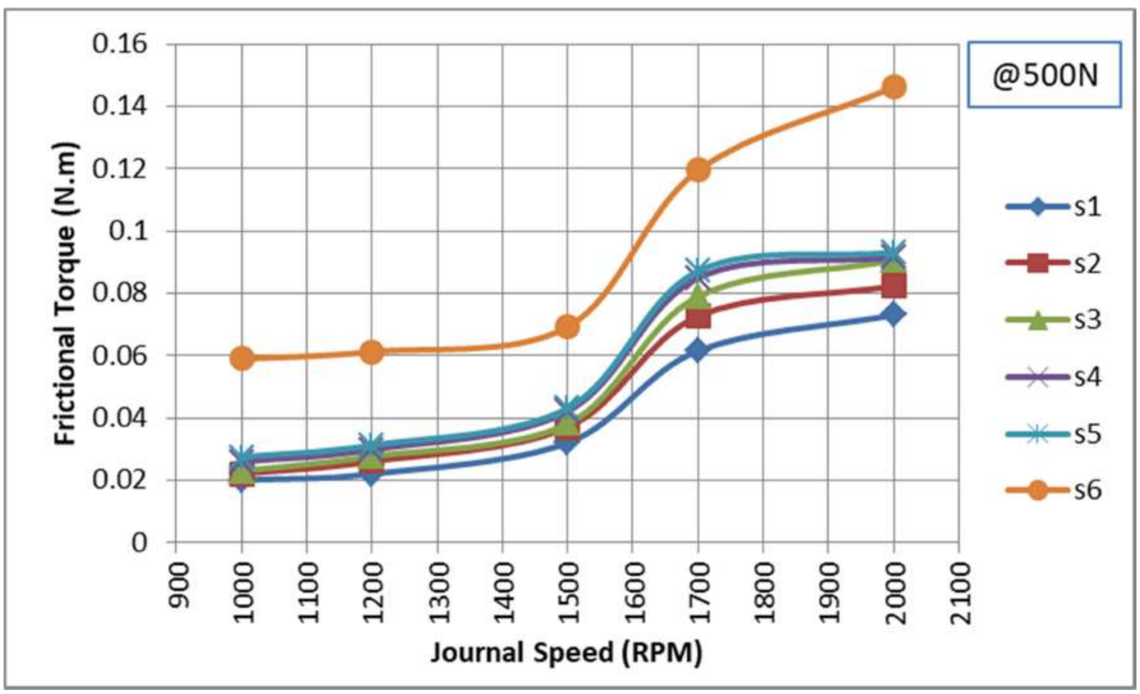
4.3.1 Friction torque in hydrodynamic lubrication

The Figure 40 shows the variation of frictional torque for different oil samples relative to journal speed under varying load conditions. At a lower speed of 1000 RPM, the average frictional torque was observed to be minimal compared to the torque levels recorded at higher speeds of 1500 RPM, 1700 RPM, and 2000 RPM. At elevated speeds, the fluid film thickness between the bearing and shaft diminishes significantly, leading to increased frictional torque. This pattern was consistent across all tested oil samples. However, it is observed that value of frictional torque gets reduced for a blend of apricot oil based biolubricant with 15W40 oil. The blend of apricot based oil biolubricant with 15W40 oil has shown better performance compared to the individual contents of the blend. The polar molecule of the apricot oil adhere strongly to metal surfaces creating a robust lubricating film, hence reducing the frictional torque for a blend of test oils.

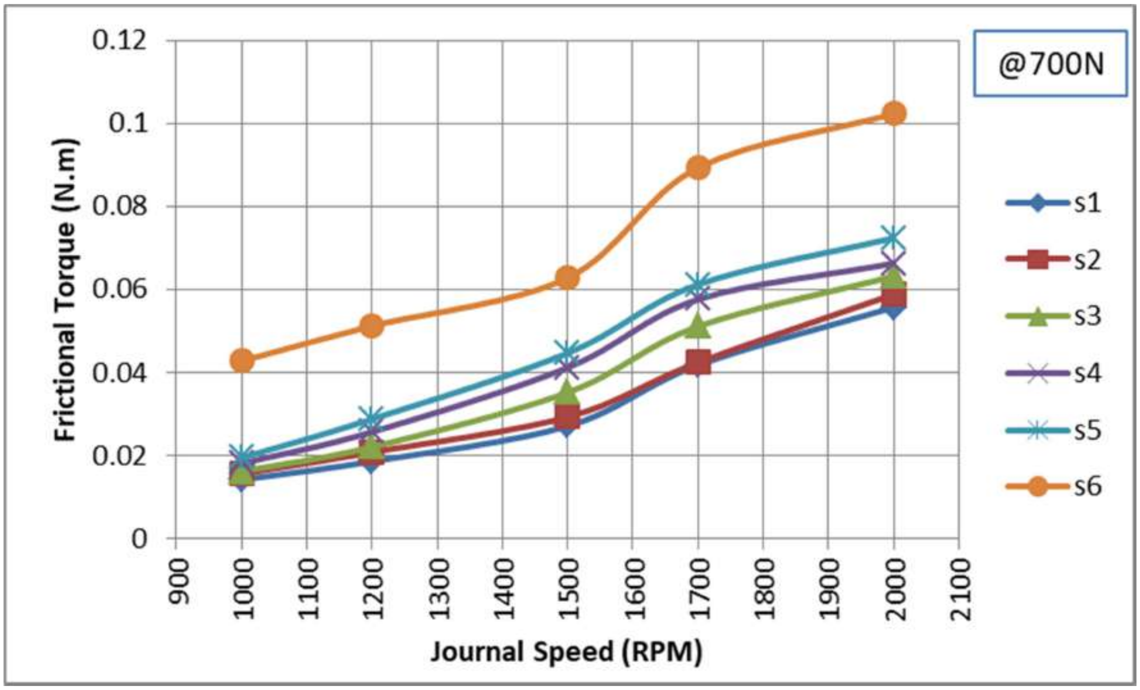
As the load on the rotating shaft rises, there is a corresponding increase in frictional torque. This phenomenon occurs due to higher contact pressure between the journal and the bearing surface. As the applied load increases, the pressure within the lubricated contact zone also raises, which results in greater shear forces within the lubricant film. The increased shear resistance contributes to higher energy dissipation as frictional heat and raising the overall frictional torque. Also, at higher loads, there is chance of localised surface asperity interactions due to reduced lubricant film thickness which further leads to rise in frictional torque.


@300N
Frictional Torque (N.m)
0.12
0.1
0.08
0.06
0.04
0.02
0
900
1000
1100
1200
1300
1400
1500
1600
1700
1800
1900
2000
2100
Journal Speed (RPM)
s1
s2
s3
s4
s5
s6

(a)


@500N
Frictional Torque (N.m)
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
900
1000
1100
1200
1300
1400
1500
1600
1700
1800
1900
2000
2100
Journal Speed (RPM)
s1
s2
s3
s4
s5
s6

(b)

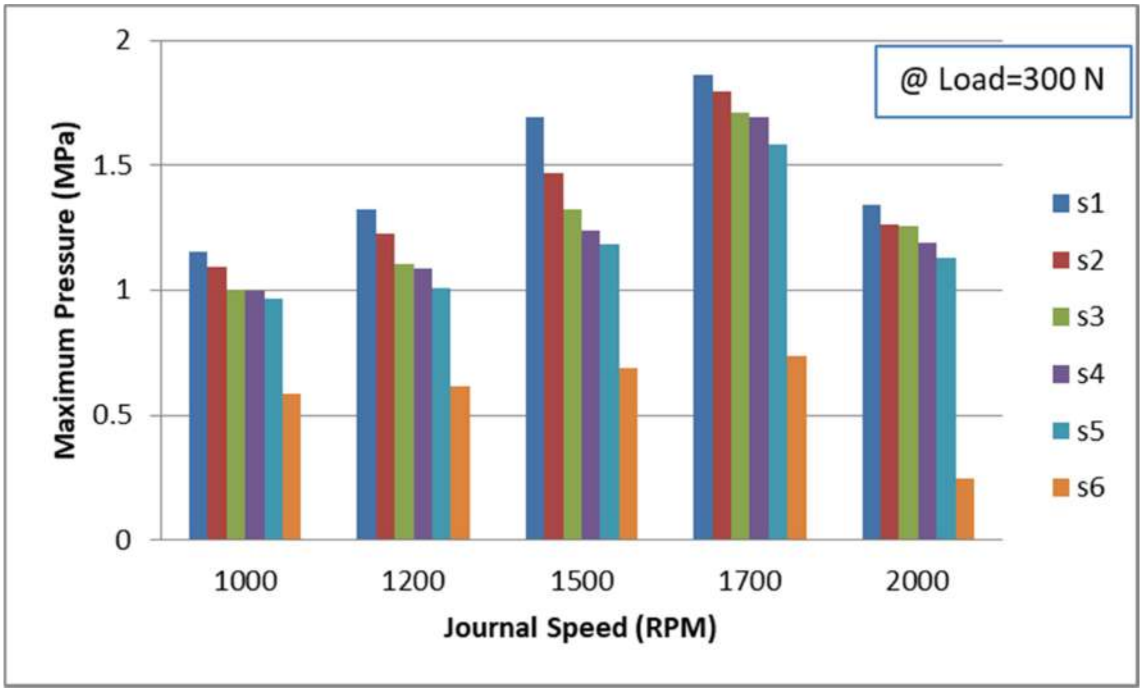


(c)

Figure 40: Variation of Frictional torque of various oil samples with respect to Journal speed under the load of (a) 300N, (b) 500N, and (c) 700N

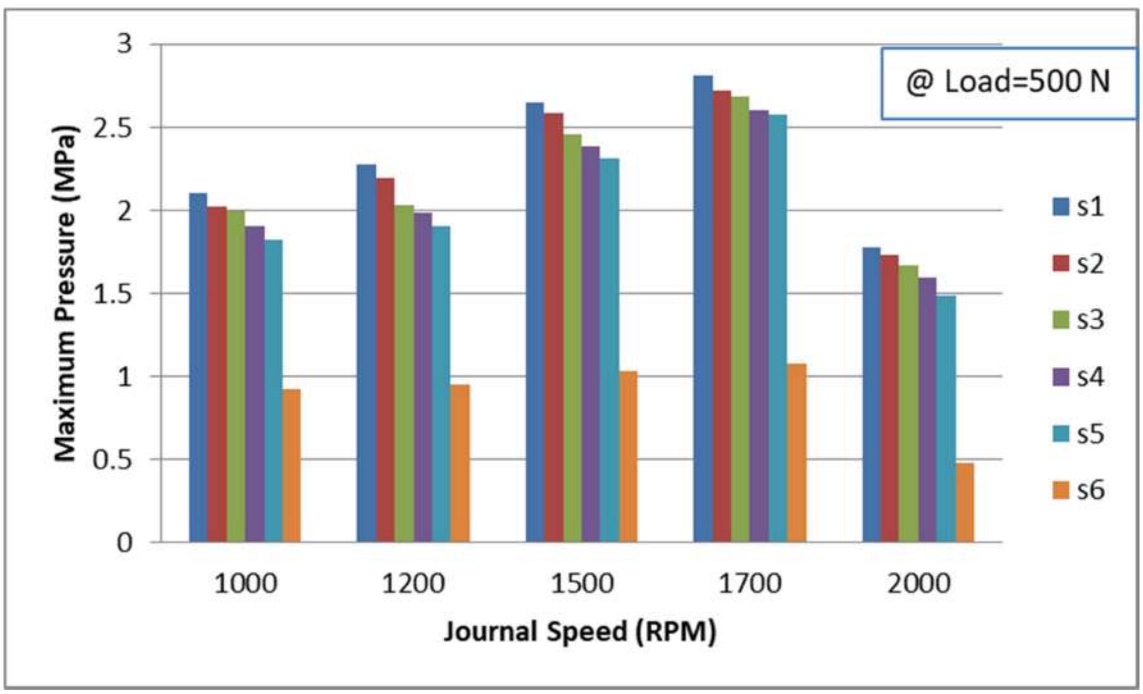
4.3.2 Circumferential Oil film pressure

The pressure distribution along the bearing's surface was measured using pressure sensors. The Figure 41 depicts the maximum pressure generated under specific load conditions and journal rotational speeds. The results indicate that maximum pressure tends to increase with rising rotational speed. However, beyond 1700 RPM, a slight decrease in maximum pressure was observed. The higher journal speed promotes the formation of thicker lubricant oil film. The converging wedge shaped geometry between journal and bearing surface generates a dynamic pressure gradient within the lubricant. The developed pressure gradient increases with the journal speed. Hence, the maximum pressure increases with the journal speed. Also, at higher journal speeds, the thick lubricant film effectively separates the journal and bearing surfaces. This separation reduces the metal-to-metal contact and allows the lubricant to sustain higher pressure without breaking down. The generated high pressure at high speeds supports the external load on the journal, hence improving the load carrying capacity of the system. The trend of increasing maximum pressure was observed for all the test oil samples.



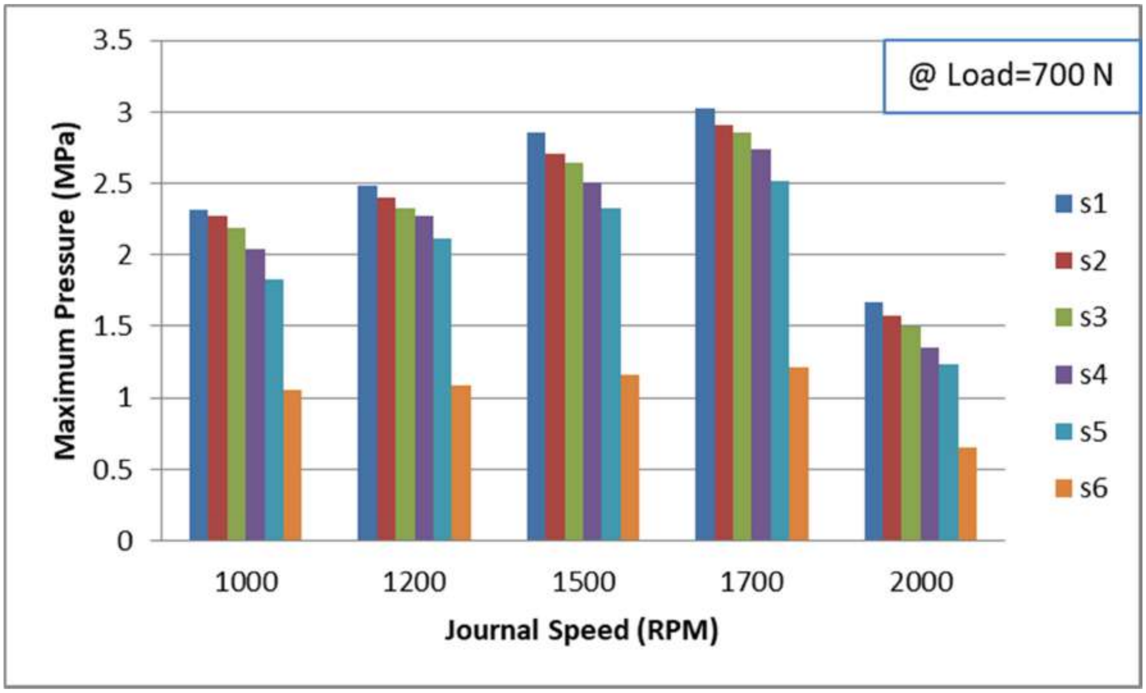
@ Load=300 N
Maximum Pressure (MPa)
2
1.5
1
0.5
0
1000
1200
1500
1700
2000
Journal Speed (RPM)
s1
s2
s3
s4
s5
s6

(a)



@ Load=500 N
Maximum Pressure (MPa)
3
2.5
2
1.5
1
0.5
0
1000
1200
1500
1700
2000
Journal Speed (RPM)
s1
s2
s3
s4
s5
s6

(b)



(c)

Figure 41: Maximum pressure generated for different oil samples at different journal speed under the load of (a) 300N, (b) 500N, (c) 700N

But beyond the journal speed of 1700RPM, the value of maximum pressure declines a little. The reduction of maximum pressure at high journal speed i.e. 2000RPM, is attributed to the fact that as the journal rotates, heat generation occurs within the lubricant layers due to friction and shear forces. The heat generation rises the temperature of the lubricant and hence lowers the viscosity of the lubricant. At higher journal speed, the heat generation is large which further reduces the viscosity of lubricant oil. The reduction of lubricant oil's viscosity leads to the reduction of maximum pressure generation.

The blends of apricot oil based biolubricant with 15W40 oil result in lesser value of maximum pressure compared to the 15W40 oil alone under all load conditions. The lower value of maximum pressure for blends of oil was the result of reduction in their viscosity. The viscosity of a blend reduces with the increase in volume % of apricot oil based biolubricant in 15W40 oil.

The Figure 42 shows the circumferential pressure distribution between the journal shaft and bearing. The pressure generated for 15W40 oil is larger than that of apricot oil based biolubricant. This is due the fact that 15W40 oil has larger viscosity than the prepared biolubricant.

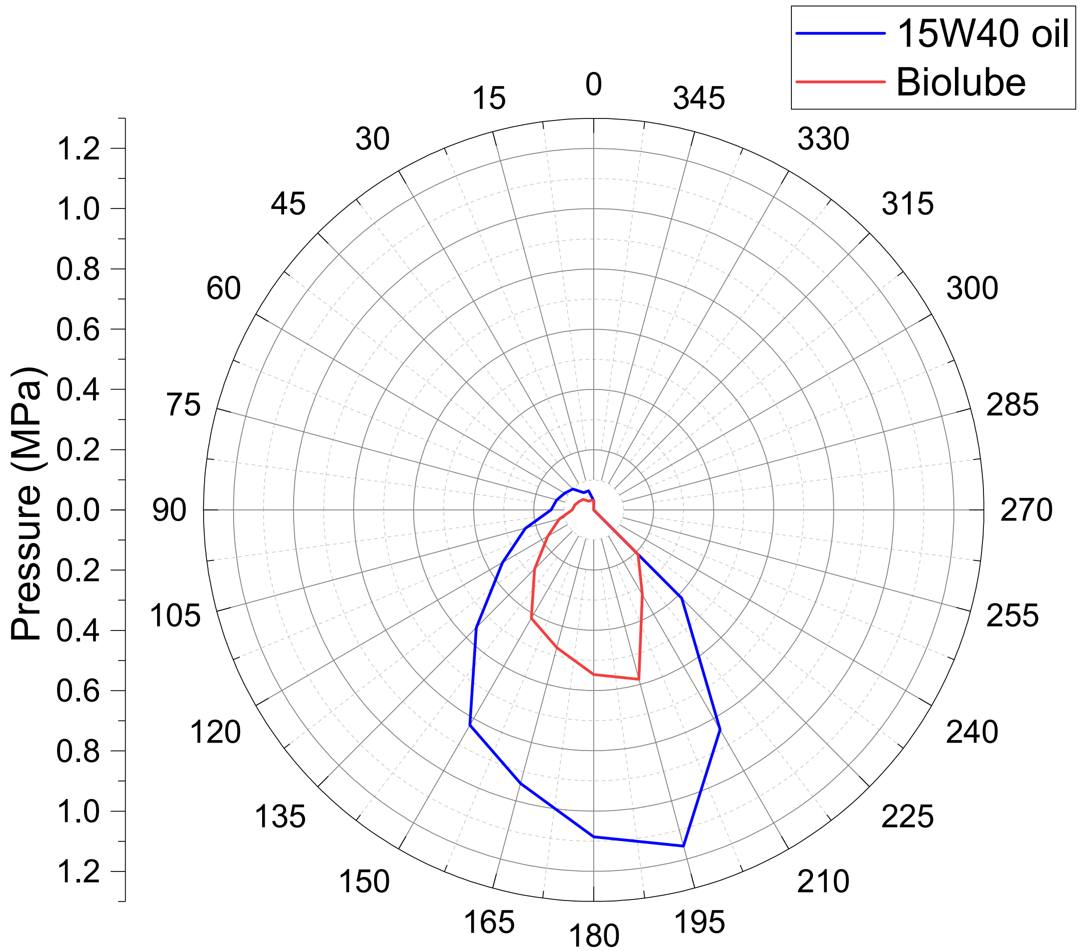


Figure 42: Circumferential pressure distributions in a journal bearing for 15W40 oil and prepared biolube of apricot oil under journal rotational speed of 1000RPM and load of 300N.

CHAPTER 5: CONCLUSIONS & FUTURE SCOPES

This chapter presents an overview of the conclusions drawn from the research study on "Synthesis, Characteristics of Bio-additives for performance study of Bio-lubricants" and gives the future scope of the research work.

5.1 Conclusions

In the presented thesis work, a novel Apricot oil based biolubricant was synthesis for its characterisation and tribological performance testing. A chemical process called transesterification was utilised for converting a vegetable oil into a biolubricant. The physio-chemical characterisation of novel biolubricant was done as per the ASTM standards and FTIR analysis. The tribological performance of the synthesised biolubricant was determined under different conditions by using High Temperature Tribometer and Four-Ball Tester. The performance of apricot oil based biolubricant as bio-additive was determined in the field of journal bearing by using a Journal bearing test rig. The following conclusions were drawn from the presented research work:

1. **Synthesis & Chracterisation of Apricot oil based Biolubricant/Bio-additive.**

a. Apricot kernel oil was extracted using the traditional method of oil extraction. However, the oil yield for mechanical extraction method is better than the traditional method.

b. The vegetable oil can be converted to suitable biolubricant through a method of chemical conversion. The chemical conversion method called Transesterification was utilised to convert Apricot oil into biolubricant. Transestrification method has higher yield rate of feedstock into desired product compared to selective hydrogenation and epoxidation. Transesterification method requires less energy and milder reaction conditions compared to other methods, hence reducing the overall production cost. Also, transestrification process is equally beneficial for both small scale and large scale production of biolubricant, whereas selective hydrogenation and epoxidation are suited for large scale production.

c. The physical characterisation of the apricot oil based biolubricant was conducted using the ASTM standard methods, and the chemical characterisation was performed using a Fourier Transform Infrared spectroscopy. The kinematic viscosity of the prepared biolubricant is lower than 15W40 oil. The lower kinematic viscosity of biolubricant is due its molecular structure and base vegetable oils. Vegetable oil molecule has triglycerides which are converted into esters using transesterification. The biolubricant contains esters that contribute to a lower viscosity. Also, the Viscosity index of apricot oil biolubricant higher than 15W40 mineral oil allowing less variation in viscosity of biolubricant with temperature. The chemical characterisation of apricot oil based biolubricant concluded that apricot oil biolubricant is a complex molecule having a long chain linear aliphatic compound.

2. Tribological performance of Bio-additive in lubricant.

a. The optimum blend of biolubricant and 15W40 oil shows better tribological behaviour than individual lubricants. The blending of 30% (v/v) biolubricant into 15W40 oil i.e. sample S4 has shown minimum wear between the tribo-pair. This observation was applicable under all test temperature conditions.

b. The optimum mixture of 15W40 oil and apricot oil-based biolubricant, i.e. S4 sample, has formed a stable, protective layer between the tribopair, which prevents the metal-to-metal contact and maintains consistency throughout the operation. This ability of the S4 sample has shown a minimum friction coefficient.

c. The fatty acids in the biolubricant lubricate most effectively only when they react with the metal to form soap. This soap film has strong cohesion. A single molecular layer of soap may provide good lubrication even on a rough surface, whereas the metallic film at a greater thickness shows the same effect.

d. The possible reason for pure biolubricant to have maximum wear and friction coefficient is attributed to removing a metallic soap film formed after the chemical reaction with a metallic surface. The optimum blend of biolubricant with mineral oil lubricant does not remove the metallic soap film and maintains a stable lubricating layer over the metal surface.

e. The ideal mixture of 15W40 and apricot oil-based biolubricant demonstrated effective anti-friction and wear-preventive characteristics.

f. The composition of 30% (v/v) apricot oil-based biolubricant with 70% (v/v) 15W40 oil has formed a suitable boundary layer lubricant between the contacting surfaces, and this lubricating film was able to withstand loading conditions during the tribo-testing.

g. The addition of 30% (v/v) biolubricant in 70% (v/v) of 15W40 oil was the optimum combination as minimum volume wear rate, and the Coefficient of friction occurs at this combination.

h. The maximum volume wear rate and Coefficient of friction have been observed for pure apricot oil-based biolubricant, subjected to tribo-testing on a four-ball tester. Hence, it concludes that pure apricot oil-based biolubricant could not create a stable, protective layer between two planes, leading to contact between them.
i. The results of ANOVA analysis of coefficient of friction proved that temperature is the most important factor affecting coefficient of friction followed by velocity. The contribution of temperature is 80.48%.
j. The results of ANOVA analysis of wear proved that velocity is the most important factor affecting wear followed by temperature. Their contributions are 35.70% and 32.80% respectively.

3. Effect of Lubricant blend with Bio-additive in the Journal Bearing.

a. It is concluded that as the journal rotational speed increased, the oil film pressure also increases and reaches maximum value at 1700RPM, beyond 1700RPM, the max. pressure value drops. The possible reason behind the drop of max. pressure is that the lubricant layer was not able to sustain high journal speed and the lubricant layer ruptures.
b. In the field of journal bearing, the biolubricant performed inferior to the 15W40 oil as the load carrying capacity of the apricot oil based biolubricant was lesser compared to 15W40 oil. This attributed to the fact that the viscosity of 15W40 oil is higher than the apricot oil based biolubricant which results in higher pressure generation in a journal bearing system.

Overall it can be concluded that the apricot oil based biolubricant has tribologically performed inferior compared to 15W40 oil. But the optimum blend of 15W40 oil and apricot oil based biolubricant had shown better performance compared to the individual contents of the blend. At the end of the presented research work, it is concluded that the optimum blend of oils is 70% volume of 15W40 oil and 30% volume of Apricot oil based biolubricant. Also, with the growing dependency of humankind on Petroleum based products, the reserve of petroleum stock is depleting at a fast rate. Hence, to attain a sustainable future, vegetable or animal oil-based lubricants should be considered an alternative to mineral oil-based lubricants.

5.2 Future scopes of work

- This thesis presents the synthesis of a biolubricant from Apricot kernel oil and studies its tribological behaviour under different conditions. Since the prepared biolubricant cannot be used alone due to its high wear rate, a nano-additive could be added in biolubricant for better tribological performance.
- A cryogenic tribometer could be utilised to analyse the behaviour of apricot oil based biolubricant under sub-zero temperature conditions.
- The formulation and performance of apricot oil based biolubricant could be optimised by testing its blend with other natural oils or eco-friendly additives to improve lubrication properties and reduce production costs.
- The performance of Apricot oil based biolubricant could be analysed for agricultural machinery which aligns with the sustainable farming practices. It could support eco-friendly operations, particularly in equipment used in organic farming.

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LIST OF PUBLICATIONS

International Journals/Proceedings

1. **Anshul Kumar,** Rajiv Chaudhary, Ramesh Chandra Singh, *Tribological Analysis of Novel Apricot oil based Biolubricant against 15W40 oil tested on High Temperature Tribometer.*, Journal of Engineering Research, vol. 2021, pp. 205–214, 2021, DOI: 10.36909/jer.ICARI.15269. **(SCIE indexed)**
2. **Anshul Kumar,** Rajiv Chaudhary, Ramesh Chandra Singh, *Tribological Performance of various blends of commercial SAE 40 oil and Novel Apricot oil based Bio-lubricant using Four Ball tester Tribometer.,* International Journal of Materials and Product Technology. vol. 67, no. 2, pp. 166–177, 2023, DOI: 10.1504/IJMPT.2023.133049. **(SCIE indexed)**
3. **Anshul Kumar,** Rajiv Chaudhary, Ramesh Chandra Singh, *Experimental and statistical analysis of apricot (prunus armeniaca) oil based biolubricant under various temperature conditions.* International Journal of Materials and Product Technology. DOI: 10.1504/IJMPT.2024.10067245

   ACCEPTED & UNDER PUBLICATION. (SCIE indexed)

International/National Conferences

1. **Anshul Kumar,** Rajiv Chaudhary, Ramesh Chandra Singh (2022). *An Experimental Investigation of Influence of Coated Piston Ring Surface under Different Lubrication Condition against Uncoated Surface on Diesel Engine.* Advances in Mechanical and Materials Technology. International Conference on Energy, Materials Sciences & Mechanical Engineering (EMSME), 30 Oct 2020 - 01 Nov 2020. Lecture Notes in Mechanical Engineering. Springer, Singapore. https://doi.org/10.1007/978-981-16-2794-1_93
2. **Anshul Kumar,** Rajiv Chaudhary, Ramesh Chandra Singh, *Performance of Apricot oil based bio-lubricant under extreme pressure conditions using Four-Ball Tester.* International Conference on Optimization Techniques in Engineering and Technology (ICOTET), 21 June 2022.
3. **Anshul Kumar,** Rajiv Chaudhary, Ramesh Chandra Singh, *Tribological analysis of a Prunus armeniaca biolubricant under various temperature conditions and its performance in the field of Journal Bearing.* International Conference on Green Technology and Sustainability (ICGTS), 30-31 Jan 2024.