Chapter 1

General Introduction

1.1: Overview

Oils, complex mixture of hydrocarbons, play a pivotal role in the current global landscape, significantly impacting both industrial and daily life sectors [1]. Its composition primarily consists of long-chain alkanes, cycloalkanes, aromatic hydrocarbons, and various heteroatomic compounds. The physicochemical properties of oil, such as viscosity, density, and thermal stability, are governed by its molecular structure and the interactions between its constituent molecules [2, 3]. In the realm of energy, mineral oils and their derivatives are indispensable, serving as primary sources for fuel, lubricating oil, and energy generation. The demand for alternate sources has also brought vegetable oils to the forefront, with biodiesel production offering a cleaner alternative to fossil fuels. In the chemical industry, oils are crucial feedstocks for the synthesis of a vast array of compounds, including polymers, pharmaceuticals, and agrochemicals. The unique properties of oils, such as their hydrophobicity, viscosity, and thermal stability, make them essential in formulating products ranging from cosmetics to industrial lubricating oils. Furthermore, advancements in nanotechnology and material science are leveraging oils as carriers for nano-additives, enhancing the thermal and flow properties of various systems. As environmental point of view challenges, the role of oils in developing eco-friendly lubricating oils and energy solutions becomes need with the aim of reducing carbon footprints and promoting sustainability. The multifaceted applications and the evolving importance of oils underscore their vital role in advancing global technological and environmental objectives [4-6].

Industrial oils play a crucial role as essential fluids employed across diverse applications in various industries [7]. These oils are formulated to meet the specific requirements of different machinery and equipment, providing necessary lubrication, protection, and performance enhancement [8, 9]. From heavy-duty manufacturing to automotive applications, industrial oils play a critical role in ensuring the smooth operation of industrial manufacturing [10]. Industrial oils are used as lubricating oils in the manufacturing industry for variety of equipments, such as presses, pumps, and machine tools. They help to reduce friction and wear, thereby improving the longevity and performance of machinery [11]. The use of industrial oils also helps to minimize downtime, maintenance costs, and energy consumption in manufacturing operations, thereby increasing productivity and profitability. Another important application of

industrial oils is in the automotive industry. EOs, transmission fluids, and hydraulic fluids are examples of industrial oils used in automotive applications. These oils help to protect engine parts from wear and tear, reduce friction, and ensure the smooth operation of various components in the vehicle. Industrial oils also play a vital role in maintaining the fuel economy and emission control systems of modern vehicles. Industrial oils are also used in the construction industry for various applications, such as in heavy equipment, cranes, and hydraulic systems. These oils are specifically designed to provide high levels of lubrication and protection in extreme operating conditions, such as high temperatures and heavy loads. The industrial oils in construction equipment also helps to reduce wear and tear, improve equipment longevity, and minimize downtime due to maintenance [12, 13].
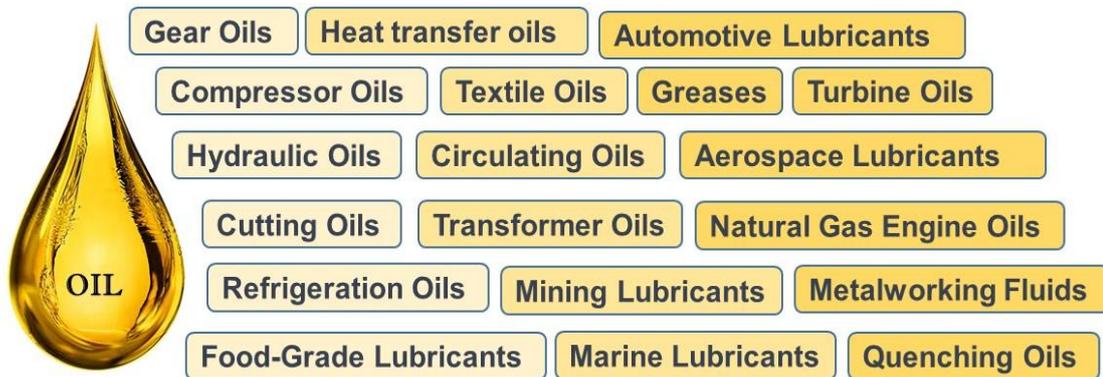

Fig. 1.1: Types of industrial oils based on various applications [14].

In addition to the above applications, industrial oils are also used in other industries, such as the food and beverage industry, pharmaceuticals, and electronics manufacturing [15]. In the food and beverage industry, it is applied for lubrication and corrosion protection in food processing and packaging equipment. In the pharmaceutical industry, they are utilized for smoothening of pharmaceutical processing equipment. In the electronics manufacturing industry, industrial oils are used for cooling and lubrication of cutting and drilling machines used in printed circuit board manufacturing [16, 17]. Overall, the application of industrial oils is diverse and essential in various industries (Fig. 1.1). These oils provide critical lubrication and protection for machinery and equipment, thereby ensuring the smooth and efficient operation of industrial processes. With advancements in technology, the demand for more specialized industrial oils with specific properties is very

much increasing, and research and development in this area will play a critical role in fulfilling the demand of the industries [4].

1.2: Base Oil (BO)

1.2.1: Classification based on chemical structure

BOs are the starting material for producing lubricating oils and other products, and their properties greatly influence the performance of the finished lubricating oil [18]. They are typically derived from crude oil but can also be made from synthetic or bio-based sources. It serves as the foundation to which various additives are added to enhance performance characteristics, like viscosity, thermal stability, and oxidation resistance [17, 19, 20].

BOs define the inherent properties of the finished product, including viscosity, stability/longevity, and load-bearing capacity (the ability to withstand extreme pressures). For example, a lubricating oil made with a high-quality, highly refined BO may have better thermal and oxidative stability than a lubricating oil made with a lower-quality BO but not have the same degree of load-bearing capacity as a result of the molecular structure and purity. As a result, the choice of BO is an essential consideration in producing lubricating oils [21, 22]. BOs can be derived from either crude oil (mineral oils), synthesized through chemical processes (SOs) or naturally derived (NOs). Based on the chemical structure and source, there are three types of liquid oil: mineral, synthetic and NO (Fig. 1.2) [23-25].
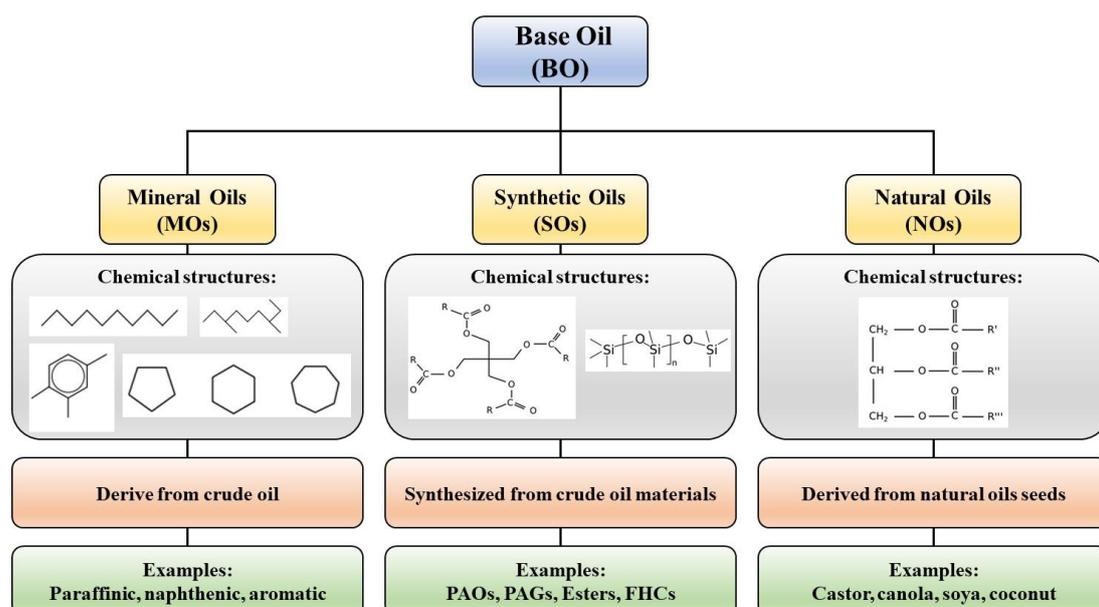
Fig. 1.2: BO classification based on chemical structure [25].

Mineral oils (MOs)

MOs are refined petroleum-based hydrocarbons. MOs are classified according to their refining process and viscosity, such as paraffinic, naphthenic, or aromatic. MOs are acquired from petroleum products which are obtained from oil wells (oilers) [26].

Once the petroleum is extracted, it goes through various fractionation processes. During these processes, oils and fuels of different properties such as viscosity are obtained. The products with higher viscosity and lubricating properties are used as lubricating agents or oils [22]. It is important to mention that MOs are very sensitive to changes in temperature and that they generally have a viscosity index up to 120. Their pour point is in the range of -6°C to -60°C, however, most MOs have a -20°C pour point [27]. Additionally, MOs burn easily means they have lower flash points. As they originate from natural resources, they contain other elements such as sulphur, phosphorous, nitrogen, and others. These elements are considered as impurities that both affect lubrication and contaminate the environment. They can cause sludge and deposit formation which occur as products of oxidation during the lubrication process. For those reasons, MOs need to go through the purification process. Once they are purified, MOs are complemented with additives to compensate for their shortcomings. Nevertheless, MOs are used in the industry in the largest amount [28, 29].

Synthetic oils (SOs)

SOs, derived from hydrocarbons or other chemicals, are designed to exhibit specific properties, such as high temperature stability, making them superior to MOs in many applications [30]. Although more expensive, SOs are purer and offer advantages in performance due to their chemical structure. They include types such as organic esters, silicones, and halogenated organic compounds [31].

These man-made BOs, such as PAOs (poly alpha olefins), PAGs (polyalkylene glycols), and various esters (diesters, polyol esters, phosphate esters), can be tailored for specific properties like high viscosity index and superior thermal stability [32]. Unlike MOs, SOs lack impurities, reducing the risk of deposit formation and enhancing stability. They also have a higher viscosity index, maintaining stability across different temperatures, and their low pour points (-18°C to -74°C) improve flowability in cold conditions [33]. Their high flash points make them ideal for applications where fire hazards are a concern. SOs excel in high and low-temperature applications, situations

requiring low flammability, and scenarios demanding compatibility with materials like rubber or plastic. They are particularly useful in industries where contamination by sulfur and phosphorus is a serious concern, such as the food industry [34]. Despite their advantages, SOs are less effective as boundary lubricating oils compared to MOs, meaning they do not reduce friction and wear as efficiently. However, this drawback is mitigated by incorporating additives, enhancing their performance as industrial lubricating oils [35, 36].

Natural oils (NOs)

NOs, derived from sources such as animal fats and vegetables like rapeseed, castor, soybean, sunflower, and palm oils, undergo minimal processing to retain their natural properties [37, 38]. These bio-based oils, sourced from renewable materials, are favoured for their biodegradability and lower environmental impact compared to traditional mineral BOs. Bio-based oils are classified according to their feedstock, with common categories including vegetable oil-based and animal fat-based oils. Despite their renewable origins, these oils often require extensive chemical processing to enhance their stability and performance, leading to higher costs. Bio-based oils can exhibit similar properties to MOs, such as lubrication and thermal stability, but with the added benefit of being more environmentally sustainable [18, 24, 39].

NOs are inherently unstable in oxidative and thermal environments. They oxidize easily at high temperatures, leading to the degradation of their lubricating properties. This instability limits their use in high-performance machine lubrication, where stability and long-term performance are crucial [40]. For example, while rapeseed and soya oils are excellent in some applications, they can deteriorate quickly under high thermal stress. In contrast, bio-based oils, although derived from natural sources, are engineered through sophisticated chemical processes to improve their resistance to oxidation and thermal degradation. These processes include the addition of antioxidants, thermal stabilizers, and other performance-enhancing additives. As a result, bio-based oils can provide reliable lubrication in a wide range of applications, from industrial machinery to automotive engines [24, 28].

Furthermore, bio-based oils are gaining traction in industries focused on sustainability and environmental responsibility [41]. Their ability to reduce dependence on non-renewable resources and lower the environmental footprint of lubricating oils makes them an attractive choice for companies aiming to meet stringent environmental

regulations and corporate sustainability goals. Despite these advancements, one of the challenges with bio-based oils remains their cost. The complex processing and sourcing from renewable materials make them more expensive than traditional MOs. However, ongoing research and development are expected to reduce these costs over time, making bio-based oils a more competitive option in the lubricating oil market [28, 42].

1.2.2: Classification based on American Petroleum Institute (API) standard

The American Petroleum Institute (API) classifies lubricating oils based on their production processes and performance characteristics, facilitating clear communication about the performance differences among various BO categories [7, 35, 43]. The API categorizes these oils into five main groups i.e., Group I. II, III, IV, and V.
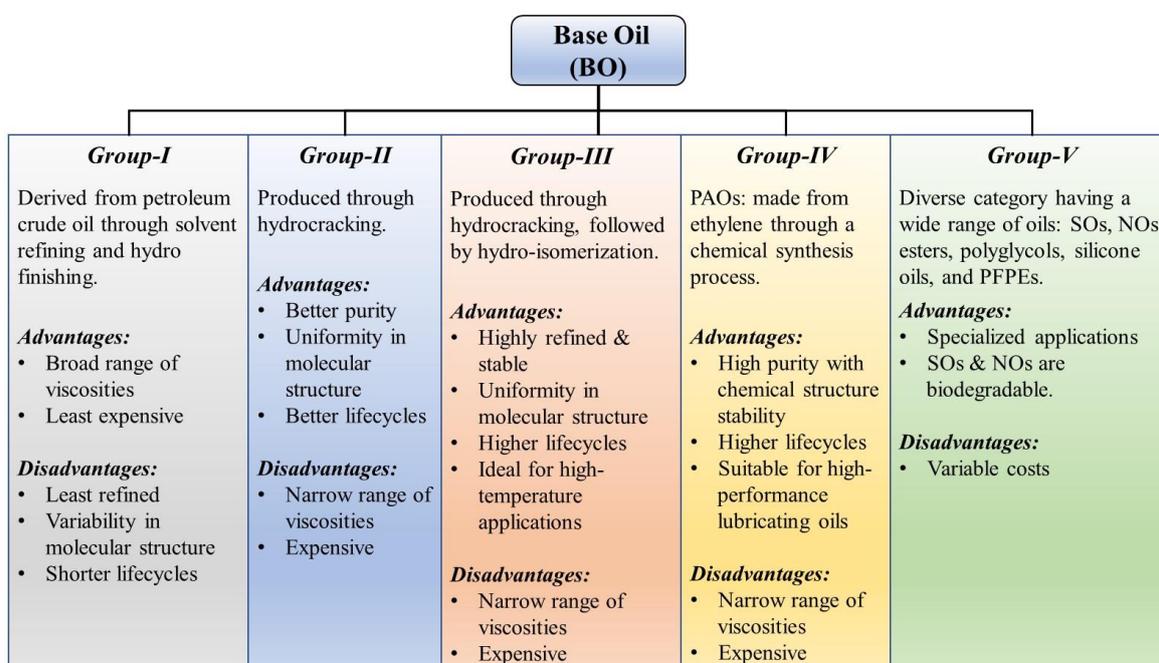
	Base Oil (BO)			
Group-I	*Group-II*	*Group-III*	*Group-IV*	*Group-V*
Derived from petroleum crude oil through solvent refining and hydro finishing.	Produced through hydrocracking.	Produced through hydrocracking, followed by hydro-isomerization.	PAOs: made from ethylene through a chemical synthesis process.	Diverse category having a wide range of oils: SOs, NOs, esters, polyglycols, silicone oils, and PFPEs.
Advantages: • Broad range of viscosities • Least expensive	*Advantages:* • Better purity • Uniformity in molecular structure • Better lifecycles	*Advantages:* • Highly refined & stable • Uniformity in molecular structure • Higher lifecycles • Ideal for high-temperature applications	*Advantages:* • High purity with chemical structure stability • Higher lifecycles • Suitable for high-performance lubricating oils	*Advantages:* • Specialized applications • SOs & NOs are biodegradable.
Disadvantages: • Least refined • Variability in molecular structure • Shorter lifecycles	*Disadvantages:* • Narrow range of viscosities • Expensive	*Disadvantages:* • Narrow range of viscosities • Expensive	*Disadvantages:* • Narrow range of viscosities • Expensive	*Disadvantages:* • Variable costs

Fig. 1.3: BO classification based on API standard.

Group I BOs are derived from petroleum crude oil through solvent refining and hydro finishing. They are the least refined and exhibit significant variability in molecular structure. This variability leads to shorter lifecycles due to the presence of benzene rings that degrade easily, but these oils also provide high load support because of naphthene ring structures. Group I oils offer a broad range of viscosities, allowing for diverse lubricating oil formulations. Historically the least expensive, their prices have risen due to market shifts towards more refined Group II and III oils.

Group II BOs, also mineral oil-based, are produced through hydrocracking, a process that breaks down larger, complex molecules into smaller, more uniform ones. This results in a purer, more durable BO compared to Group I. Commonly used in engine, hydraulic, turbine, and compressor oils, Group II oils offer higher performance and purity. These oils are generally more expensive than Group I and have a narrower viscosity range, favouring low to medium viscosities.

Group III BOs are also derived from crude oil through hydrocracking, followed by hydro-isomerization, which further refines the molecular structure for improved uniformity and stability. This results in BOs with superior durability and temperature response. Group III oils are ideal for high-temperature applications and are used in EOs, compressor oils, and transmission fluids.

Group IV BOs, known as poly alpha olefins (PAOs), are fully SOs made from ethylene through a chemical synthesis process. These oils are highly pure, uniform, and stable, making them suitable for high-performance lubricating oils such as motor oils and transmission fluids. Commercially, they are referred to as synthetic hydrocarbon (SHC) oils.

Group V includes all BOs not classified in Groups I-IV. This diverse category encompasses a wide range of oils, including SOs, NOs, esters, polyglycols, silicone oils, and PFPEs. Group V oils vary widely in properties and performance and are used in specialized applications across automotive and industrial sectors. Their costs can vary significantly depending on their specific composition and intended use.

Owing to the need of various oils to develop best lubricating oils, our present research is focused on castor oil (natural) & engine oil (synthetic) as the base oils.

1.3: Castor oil (CO)

CO has long been utilized commercially as a highly renewable resource in the chemical industry. It is a vegetable oil obtained by pressing the seeds of the CO plant (Ricinus communis L.), which is predominantly cultivated in Africa, South America, and India. Major producers of CO include Brazil, China, and India. Historically, CO is believed to have been domesticated in Eastern Africa and introduced to China from India approximately 1,400 years ago. India stands as a net exporter of CO, accounting for over 90% of global exports. The primary importers are the United States, the European Union, and China, which collectively account for 84% of the imported CO [44, 45].

India is recognized as the world leader in castor seed and oil production, dominating the international CO trade. The country's CO production typically ranges between 250,000 and 350,000 tons annually. Approximately 86% of India's castor seed production is concentrated in Gujarat, followed by Andhra Pradesh and Rajasthan. The regions of Mehsana, Banaskantha, and Saurashtra/Kutch in Gujarat, along with the districts of Nalgonda and Mahboobnagar in Andhra Pradesh, are the primary areas for CO production. The economic success of castor crops in Gujarat since the 1980s can be attributed to an effective breeding program, a robust extension model, and access to well-developed national and international markets [46].
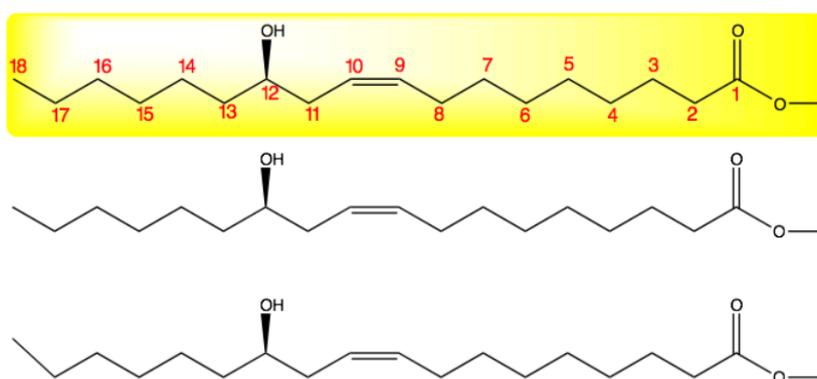
Fig. 1.4: Chemical structure of CO [45].

The structure of CO consists of triglycerides without glycerin, characterized by an 18-carbon chain with a double bond (Fig. 1.4). Its chemistry is predominantly based on the ricinoleic acid structure, which includes a carboxyl group, a hydroxyl group, and a single point of unsaturation [47]. The carboxyl group in CO molecule enables the preparation of various esterification products. The hydroxyl group on the 12th carbon can be acetylated or removed through dehydration to increase unsaturation, resulting in a semi-drying oil. High-temperature pyrolysis and caustic fusion can split the reactive hydroxyl group, producing useful shorter-chain products. Additionally, the hydroxyl group enhances the molecular structure, preventing the formation of hydroperoxides. The double bond can be modified via carboxylation, epoxidation, or hydrogenation, and the single point of unsaturation can also be altered through epoxidation and hydrogenation. Hydrogenated CO, a wax-like substance, is produced through hydrogen reduction of the oil [46-48].

The unique structure of CO imparts properties that make it suitable for various industrial applications. CO is composed of up to 90% ricinoleic acid, 4% linoleic acid, 3%

oleic acid, 1% stearic acid, and less than 1% linolenic acid. Its high ricinoleic acid content is particularly valuable for numerous applications in the chemical industry [49].

CO has a wide range of industrial applications due to its unique chemical composition and properties. In lubricating applications, CO is valued for its high viscosity, excellent lubricity, and ability to perform well under extreme temperatures. It is often used as a BO in high-performance lubricating oils for engines, machinery, and hydraulic systems. Additionally, its biodegradability and non-toxic nature make it better choice for lubricating oils. In the realm of dielectric applications, CO serves as an effective insulating fluid in transformers and capacitors due to its high dielectric constant and electrical insulating properties. This helps in the efficient operation and cooling of electrical equipment. Furthermore, CO is used as a cooling fluid in various industrial processes where its thermal stability and high boiling point ensure effective heat dissipation. Its versatility extends to its role as a raw material in the production of biopolymers, surfactants, and other chemical derivatives used in the manufacturing of coatings, plastics, and cosmetics. Overall, CO's multifunctional nature makes it a valuable resource across diverse industrial sectors [44-48].

To develop CO as an effective lubricating oil, several key improvements are essential. Enhancing its rheological properties is crucial to ensure optimal viscosity and flow characteristics across a wide temperature range, reducing friction and wear on engine components. Improving its thermal conductivity is also vital for efficient heat dissipation, preventing overheating, and maintaining stable engine temperatures. Additionally, increasing CO's oxidative stability will prevent degradation and extend its service life. Future research should focus on incorporating nano-additives to further enhance these properties, exploring synthetic modifications to improve performance under extreme conditions, and developing environmentally friendly formulations. These advancements will position CO as a competitive and sustainable alternative in the lubricating oils market.

1.4: Engine Oil (EO)

EO, also known as motor oil or lubricating oil, is essential for the efficient and long-lasting operation of internal combustion engines [18]. Its primary functions include lubricating moving parts to reduce friction and wear, dissipating heat generated during engine operation, and preventing damage to engine components. Additionally, EO acts as a cleaning agent, removing sludge, dirt, and contaminants, and provides a protective barrier

against corrosion and rust. EOs are typically formulated from BOs derived from petroleum (MOs), synthesized from chemical compounds (SOs), or a blend of both, combined with additives that enhance performance characteristics such as viscosity, oxidation stability, detergency, and anti-wear properties [50, 51].

The Society of Automotive Engineers (SAE) classification is a standardized system used to grade EOs based on their viscosity, which are important for proper lubrication under different operating conditions. Viscosity, the measure of an oil's resistance to flow, varies with temperature. The SAE classification system uses a numerical code to indicate the oil's viscosity at cold (winter) temperatures, denoted by a "W," and at high operating temperatures. For example, in the classification "10W30," the number before the "W" (10) represents the oil's viscosity at low temperatures, indicating better flow in cold conditions. The number after the "W" (30) indicates the oil's viscosity at high temperatures, signifying that it remains thick enough to provide proper lubrication under heat [52, 53].
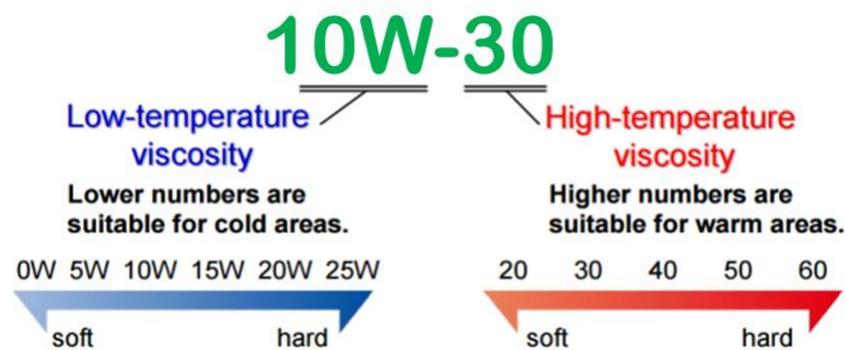

Fig. 1.5: SAE viscosity notation [54].

Single-grade oils, such as SAE 30 or SAE 40, have a fixed viscosity rating and are suitable for specific temperature ranges. In contrast, multi-grade oils, like 10W30 or 5W40, are designed to perform effectively across a wider temperature range. Multi-grade oils contain viscosity index improvers, allowing the oil to remain fluid at low temperatures while retaining sufficient thickness at high temperatures [55, 56]. This versatility makes multi-grade oils more suitable for modern engines, which often operate under varying temperature conditions. The SAE classification helps consumers and manufacturers choose the right oil for their engines, ensuring optimal performance, protection, and longevity.

EOs must meet specific specifications to perform their functions effectively. Appropriate density and viscosity are crucial for forming a lubricating film between

moving surfaces, which reduces friction and wear. A high flash point ensures the oil cannot igniting even at high temperatures, while a high viscosity index (VI) indicates the oil's ability to maintain consistent viscosity across a wide temperature range, ensuring reliable lubrication [57].
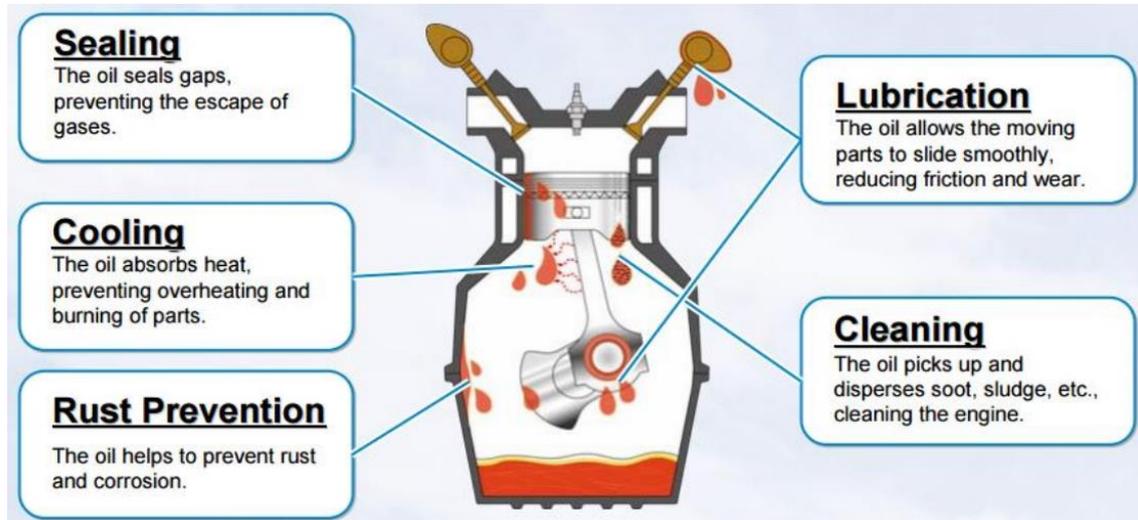

Fig. 1.6: Function of EO [58].

High oxidation stability is essential to prevent the oil from oxidizing, which can degrade its physical properties and cause damage to engine parts [59]. The pour point of the oil, the lowest temperature at which it remains fluid, is particularly important for engines operating in very cold environments. If the oil's pour point is too high, it can freeze and fail to flow into the tubes and engine parts, preventing proper lubrication and potentially leading to engine failure. Therefore, EO must have a pour point suitable for the work environment to ensure reliable performance in all conditions [60]. Therma conductivity is another important property of EO. High conductivity allows the oil to transfer heat efficiently away from critical engine components, helping to prevent overheating and ensuring that the engine operates within a safe temperature range. Efficient heat dissipation contributes to the overall durability and performance of the engine, making thermal conductivity a key consideration in the formulation of high-quality EOs [60-63]. Regardless of application, Fig. 1.6 shows the essential functions of EOs.

The study of rheology and thermal conductivity improvements in EO is crucial in today's automotive industry due to the increasing demands for enhanced engine performance, efficiency, and longevity. Understanding and optimizing the flow properties (rheology) of EOs ensures better lubrication, reduced friction, and improved fuel

efficiency, especially under varying temperature conditions. Enhancing thermal conductivity permits more effective heat dissipation, preventing overheating and maintaining optimal engine temperature. These improvements collectively contribute to the engine's overall durability and performance, addressing the needs of modern high-performance and fuel-efficient engines while meeting stringent environmental regulations.

1.5: Nano-additives: Newer class of oil additives

Additives are substances specifically formulated to enhance the anti-friction, physical, and chemical properties of BOs-whether mineral, synthetic or natural. These enhancements result in improved oil performance. The combination and quantity of different additives are determined by the type of oil and the specific operating conditions such as temperature, loads, materials of machine parts, and the environment. The maximum proportion of additives in lubricating oils can be as high as 30% [4, 64-66]. Fig. 1.7 depicts the research articles published with the topic "*Nano-additives*" at the Web of Science (Clarivate Analytics) up to the present year 2024.
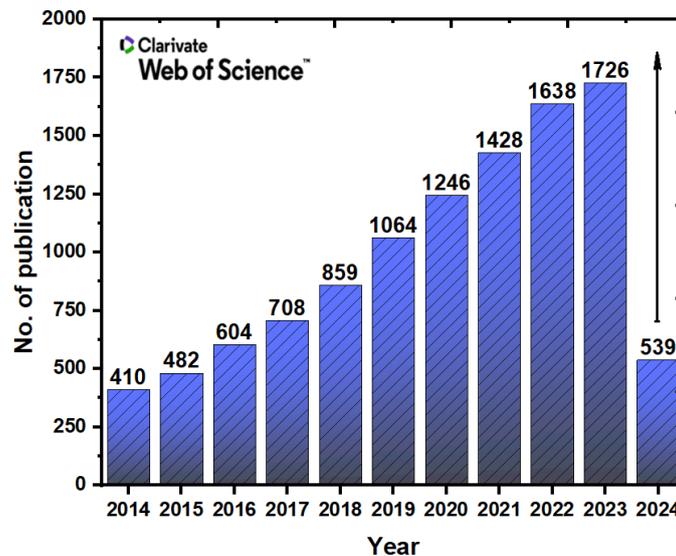

Fig. 1.7: Research publications with "*Nano-additives*" as the key word against year. (*Source: WoS*)

Lubricating oils are essential for reducing friction and protecting mechanical parts from wear. Traditionally, mineral oil has been used as a lubricating oil. However, its disposal poses significant environmental risks, affecting both aquatic and terrestrial ecosystems. Bio- lubricating oils offer a promising alternative as they are biodegradable and non-toxic.

- **Extreme pressure (EP) additives,**
- **Rust and corrosion inhibitors**
- **Friction modifiers,**
- **Anti-wear additives,**
- **Anti-oxidants,**
- **Detergents,**
- **Dispersants,**
- **Pour point depressants,**
- **Viscosity index improvers,**
- **Anti-foaming agents**

Fig. 1.8: Traditional oil additives used for lubrication [58].

Compared to MOs, bio- lubricating oils provide several advantages, including excellent lubricity, higher flash points, appropriate viscosity, and lower volatility [67]. To further reduce wear and friction, small percentages of additives are incorporated into the lubricating oil base stock to enhance its properties. These additives can be categorized in to friction modifiers (FMs), anti-wear (AW) additives, extreme pressure (EP) additives, rust and corrosion inhibitors (R&CIs), anti-oxidants (AOs), detergents, dispersants, pour point depressants (PPDs), viscosity index (VI) improvers, and anti-foaming agents (AFAs) [24, 68].

FMs are substances that reduce the coefficient of friction, thereby decreasing fuel consumption. The crystal structure of most friction modifiers consists of molecular platelets (layers) that can easily slide over one another. Common solid lubricants used as friction modifiers include: graphite, molybdenum disulfide, boron nitride (BN), tungsten disulfide (WS2), polytetrafluoroethylene (PTFE), etc [69].

AW additives prevent direct metal-to-metal contact between machine parts when the oil film breaks down. Their use results in longer machine life due to the higher wear and score resistance of the components. The mechanism involves the additive reacting with the metal on the part surface to form a film, which slides over the friction surface. Common anti-wear additives include: zinc dithiophosphate (ZDP), zinc dialkyldithiophosphate (ZDDP), tricresylphosphate (TCP), etc [70].

EP additives prevent seizure conditions caused by direct metal-to-metal contact under high loads. Similar to anti-wear additives, EP additives form a coating on the part surface, protecting it from direct contact with other parts and reducing wear and scoring.

Materials used as EP additives include: chlorinated paraffins, sulfurized fats, esters, zinc dialkyldithiophosphate (ZDDP), molybdenum disulfide, etc [70].

R&CIs form a barrier film on the substrate surface, reducing the corrosion rate. They also absorb onto the metal surface, protecting it from oxygen, water, and other chemically active substances. Common inhibitors include: alkaline compounds, organic acids, esters, amino acid derivatives, etc [71].

AOs inhibit the oxidation process of oils. MOs react with air oxygen to form organic acids, increasing oil viscosity, forming sludge and varnish, corroding metal parts, and causing foaming. Most lubricating oils contain anti-oxidants, such as: zinc dithiophosphate (ZDP), alkyl sulfides, aromatic sulfides, aromatic amines, hindered phenols, etc [72].

Detergents neutralize strong acids in the lubricating oil (such as sulfuric and nitric acids from internal combustion engines) and remove the neutralization products from metal surfaces. They also form a film on the part surface to prevent high-temperature sludge and varnish deposition. Detergents are commonly added to EOs and include phenolates, sulfonates, and phosphonates of alkaline and alkaline-earth elements such as calcium (Ca), magnesium (Mg), sodium (Na), or barium (Ba) [73].

Dispersants keep foreign particles in a lubricating oil finely divided and uniformly dispersed, preventing the formation of sludge and varnish. Long-chain hydrocarbons such as polyisobutylene succinimides are used as dispersants [73].

PPDs inhibit the formation and agglomeration of wax particles in MOs at low temperatures, maintaining the lubricating oils fluidity. Co-polymers of polyalkyl methacrylates (PMAs) are commonly used as pour point depressants [74].

VI improvers maintain the oil's viscosity at acceptable levels even at increased temperatures, ensuring a stable oil film. These improvers are widely used in multigrade oils, with acrylate polymers being a common choice [75].

AFAs, such as dimethylsilicones (dimethylsiloxanes), are used in lubricating oils to prevent the formation of air bubbles that cause foaming, enhancing oil oxidation, and reducing the lubrication effect, leading to oil starvation [76].

Traditional additives used in oils have been restricted due to environmental protection concerns. To address this issue and the performance limitations of existing lubricating oils, researchers are investigating "***Nano-additives***" [77, 78]. Nano-additives,

composed of nanoparticles, are the finest form of materials with at least one structural dimension less than 100 nm and a large surface area to volume ratio. These nanoparticles have reduced surface imperfections, spatial confinement, a large fraction of surface atoms, and high surface energy. They can be categorized based on their physical and chemical properties, shape, size, morphologies, and materials, such as carbon-based, ceramic, metal, semiconductor, polymeric, and lipid-based nanoparticles [79]. When these nano-additives are designed to form colloidal suspensions in BOs, the resulting product is referred to as a "*Nanofluid*", which is an enhanced version of the BO [80, 81]. Fig. 1.9 depicts the research articles published with the topic "*Nanofluid*" at the *Web of Science (Clarivate Analytics)* up to the present year 2024.
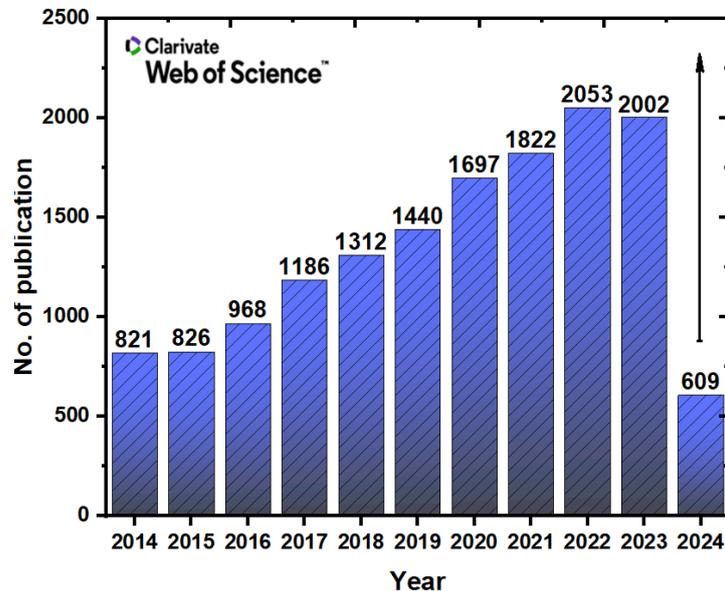

Fig. 1.9: Research publications with "*Nanofluid*" as the key word against year. (*Source: WoS*)

Most nano-additives are environmentally friendly and improve the tribological properties of oils without requiring harmful trioactive elements such as chlorine, phosphorus, and sulfur [82]. Their small sizes allow them to easily enter friction contact areas and form a protective tribofilm, preventing wear on friction surfaces. Additionally, their high surface activity enhances the stability of the tribofilm through physical and/or chemical adsorption. Nano-additives serve multiple functions: they act as AW and EP additives, FMs, and rheology modifiers. They also improve thermal conductivity, provide high thermal stability, and interact with friction surfaces without an induction period [83, 84]. Mainly basic 3 types of nano-additives: metal-based nano-additives, carbon-based nano-additives, and nanocomposite-based nano-additives, as shown in Table 1.1 [65].

Table 1.1: Types of nano-additives for oils.

Types of nano-additives		Examples	References
Metal-based nano-additives	Pure metal	Au, Ag, Pd, Pt, Cu, Ni, Fe, Ti, Zn	[85-90]
	Metal oxide	TiO_2, ZnO, Fe_2O_3, Fe_3O_4, Al_2O_3, CuO, SiO_2, CeO_2, MgO, NiO	[91-94]
	Metal sulfide	ZnS, CdS, PbS, CuS, NiS, FeS, MoS_2, SnS_2, Ag_2S, Sb_2S_3	[95-98]
	Metal hydroxide	$Al(OH)_3$, $Fe(OH)_3$, $Mg(OH)_2$, $Ni(OH)_2$, $Cu(OH)_2$, $Ca(OH)_2$, $Zn(OH)_2$, $La(OH)_3$, $Sn(OH)_2$, $Mn(OH)_2$ and layered double hydroxide (LDH)	[99-102]
	Metal salt	AgCl, $AuCl_3$, $FeCl_2$, $FeCl_3$, $AlCl_3$, $MgCl_2$, $CaSO_4$, $CuSO_4$, $NiSO_4$, $ZnSO_4$ $Zn_3(PO_4)_2$, $FePO_4$, $Ca_3(PO_4)_2$, $AlPO_4$ CaB_4O_7, ZnB_2O_4, $Cu(BO_2)_2$, $Mg_3(BO_3)_2$	[103, 104]
Carbon-based nano-additives	Pure carbon	CNTs, graphene, fullerenes (such as C60), carbon quantum dots, carbon nanofibers, nano diamond	[105-108]
	Polymer	PS, PEG, PLGA, PVA, PMMA, PTFE, PSS, PVP	[23, 109, 110]
Nanocomposite-based nano-additives		α-Fe_2O_3@GO, FeS_2@G, Ag@G, Cu@GO, Mn_3O_4@G, Al_2O_3@MWCNT, Fe_3O_4@SiO_2, TiO_2@CNTs, ZnO@SiO_2, and CuO@ZnS	[111-114]

The synthesis methods for nanofluids can be classified into two categories: one-step methods and two-step methods.

One-step method

The one-step method of synthesizing nanofluids involves the direct preparation and dispersion of nanoparticles into the BO. This process eliminates the requirement for additional steps like vaporization drying, storage, and transportation, leading to improved stability of the particles and reduced nanoparticle agglomeration. The one-step approach can be relatively expensive and may not be easily scalable for large-scale production. Furthermore, the presence of residual reactants and impurities in the nanofluids can have a negative impact on their overall quality and performance [115, 116].

Two-step method

Two-step methods are commonly employed technique for synthesizing nanofluids. Initially, the chemical or physical methods are employed for preparation of solid

nanoparticles, and then they are dispersed into the BO using different techniques like magnetic stirring, ultrasonic treatment, high-shear mixing, or ball milling. These methods are preferred due to their cost-effectiveness, compatibility with various nanoparticles, and potential for large-scale production. However, nanoparticle agglomeration can occur during both stages of the two-step process because of nanoparticles' large surface area and high surface activity. Despite this challenge, researchers favour the two-step methods for their ability to lower production costs and facilitate scalability [117].

The role of nano-additives in reducing friction has been extensively investigated by researchers, and the mechanisms involved can be categorized as follows: rolling effect, protective film formation, mending effect, and polishing effect. The first two mechanisms directly improve lubrication. Spherical nano-additives can roll between frictional surfaces, acting as ball bearings. Additionally, nano-additives form a thin protective film on the surface, reducing friction between the surfaces. The latter two mechanisms enhance the surface. Nano-additives can deposit on the frictional surface, forming a tribo-film that compensates for material loss (mending effect). Furthermore, the abrasiveness of hard nano-additives reduces the roughness of the rubbing surfaces (polishing effect) [65, 118, 119].
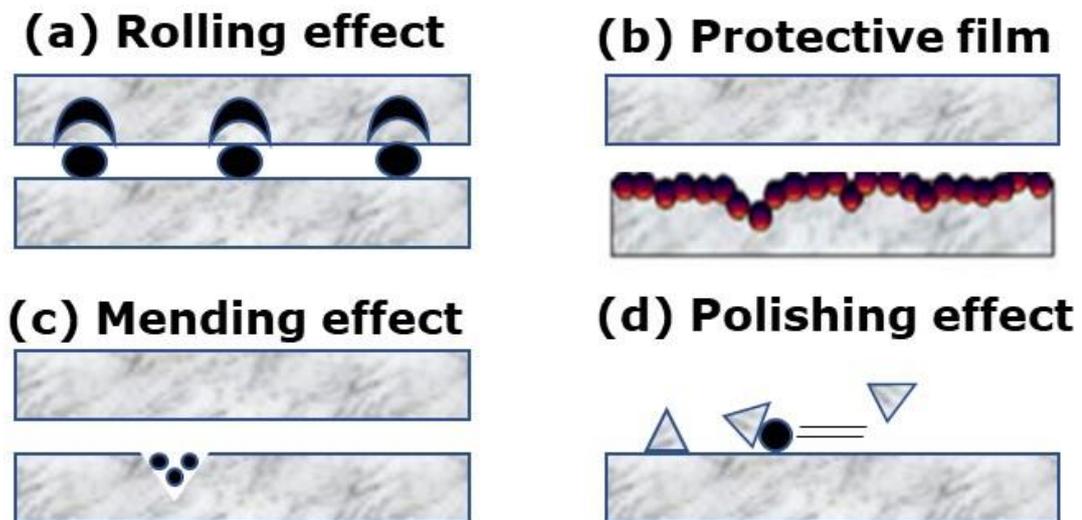

Fig. 1.10: Lubrication mechanisms of nanofluid containing nano-additives: (a) rolling effect, (b) protective film, (c) mending effect, and (d) polishing effect [118, 119].

1.6: Influence of nano-additives on the rheology of base oil and its nanofluids

1.6.1: Rheology

Rheology is the science that studies the flow and deformation of matter. The term 'Rheology' was coined in the 1920s by Professor E. C. Bingham at Lafayette College in Indiana [120]. It originates from the Greek words 'rheo', meaning 'flow', and 'logia', meaning 'the study of' [121]. Rheology encompasses the deformation of solid-like materials as well as the flow of liquid-like materials, focusing on the behaviour of complex viscoelastic materials that exhibit properties of both solids and liquids in response to force, deformation, and time. Flow is considered a special case of deformation, and the relationship between stress and deformation is a fundamental property of materials [122].

Viscosity is a crucial property in analysing fluid behaviour. When a fluid is disturbed from equilibrium by a velocity u, the dynamic viscosity η describes the fluid's tendency to dissipate energy [122-124]. Dynamic viscosity is related to the tangential force per unit area required to slide one layer (A) against another layer (B) with the fluid in between. In Fig. 1.11, the force F causing plate A moving with the velocity U in x-direction, creating a velocity profile in the liquid depending on y, is schematically shown below.
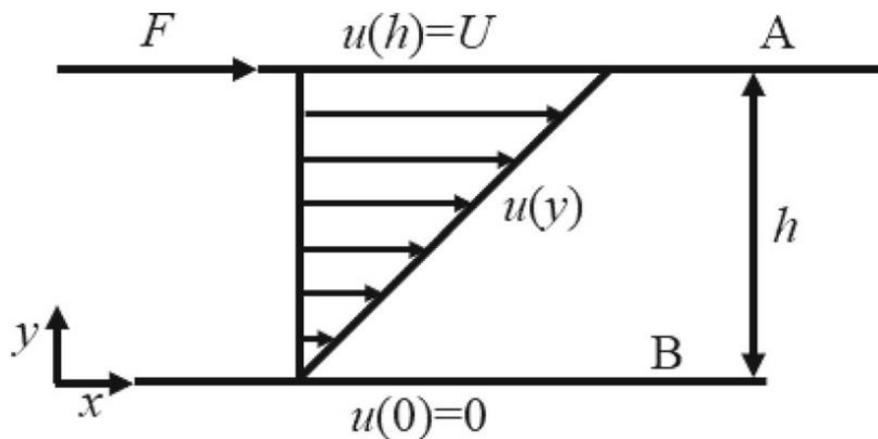

Fig. 1.11: Simple shear of a liquid film between two plates [125].

So, the viscosity of a liquid can be seen as the measure of how resistive the fluid is to flow, and it can be defined by the mathematical expression:

$$\eta = \frac{\tau}{\dot{\gamma}}\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\text{Eq. (1.1)}$$

Where,

η = dynamic viscosity in Pa·s,

τ = shear stress in N·m^{-1}, and

$\dot{\gamma}$ = shear rate in s^{-1}

In one direction flow, the shear rate is generally expressed as:

$$\dot{\gamma} = \frac{du}{dy}$$…………………………Eq. (1.2)

Where,

y = height,

t = time, and

u = dx/dt i.e., velocity

1.6.2: Newtonian and non-Newtonian flow behaviour of fluids

Fluid behaviour can be categorized as Newtonian and non-Newtonian (including pseudoplastic, Bingham plastic, and dilatant behaviours) [126]. For Newtonian behaviour, the viscosity remains constant with shear rate, and the stress exhibits a linear relation with shear rate. Typical Newtonian fluids include water, simple hydrocarbons, and dilute colloidal dispersions. In contrast, non-Newtonian fluids exhibit viscosity that varies versus the applied shear rate or shear stress [124, 127].
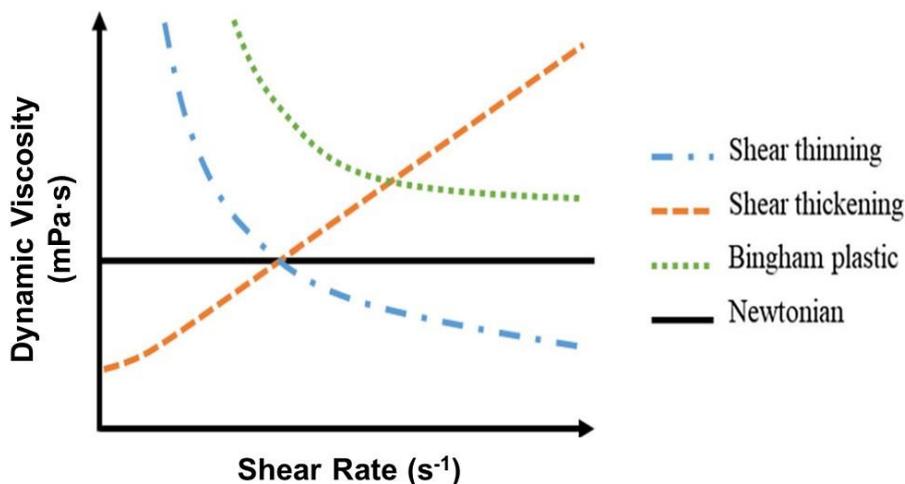

Fig. 1.12: Different models for the change of viscosity with shear rate [128].

As displayed in Fig. 1.12, a fluid is classified as a ***Newtonian fluid*** when its viscosity remains constant with varying shear rate. An example of a Newtonian fluid is water. Regardless of the shear rate applied, whether you stir it gently or vigorously, the

viscosity of water remains constant. This means water flows consistently and predictably under various conditions without changing its viscosity based on the force or stress applied [129]. In contrast, non-Newtonian liquids exhibit viscosity that varies with applied shear force and time. Common types of non-Newtonian fluids include shear-thinning fluids, shear-thickening fluids, and Bingham plastic fluids [130].

Shear-thinning fluids i.e., pseudoplastic fluids which indicates fluids viscosity, or the resistance to flow, reduces as the shear rate increases. In other words, the more you stir or squeeze, the easier it flows. However, if you stop applying the force, it thickens up again over time [131-133]. An example of a pseudoplastic fluid is paint. When you stir or brush paint, it becomes less viscous and flows easily, allowing for smooth application. This property arises because the viscosity of pseudoplastic fluids reduces as shear rate increases. Once the stirring or application force is removed, paint returns to its original viscosity, which prevents dripping and ensures an even coat on surfaces.

Dilatant fluids become thicker and more viscous under stress [134]. These fluids dilate or expand when force is applied, causing them to resist flow more strongly. This behaviour is often described as "shear thickening" because the viscosity increases as the shear rate or force applied increases [135-137]. When subjected to shear stress, such as stirring or squeezing, fluid behaves like a solid and becomes more viscous. If you apply a sudden force, it can even resist the impact. However, when the force is removed and it's allowed to sit quietly, it returns to its liquid state and flows freely again [138].

Bingham plastic fluids are a type of non-Newtonian fluid that behaves as a solid until a certain yield stress or force is exceeded [139]. Below this yield stress, Bingham plastics do not flow and maintain their shape like a solid. However, once the yield stress is surpassed, they begin to flow like a viscous fluid with a constant viscosity. This behaviour distinguishes Bingham plastic fluids from other non-Newtonian fluids, where viscosity varies continuously with shear rate or stress. Common examples of Bingham plastics include toothpaste, certain paints, and some types of drilling muds used in engineering applications.

It has been found that nano-additives in nanofluids often aggregate, which reduces the mobility of the liquid within and around these aggregates, thereby increasing the viscosity of the nanofluid. As the shear rate increases, these aggregates break down to

small structures, releasing some of the immobilized liquid and causing a decrease in suspension viscosity. Numerous studies have focused on developing models to predict the viscosity of nanofluids as a function of shear rate. In this study, traditional power-law model also known as "Ostwald–de Waele" (OdW) model have been employed to describe the flow behaviour of nanofluids [140]. This power-law model with the relationship between the shear stress with the shear rate can be written as:

$$\tau = m \cdot \gamma^n \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\text{Eq. (1.3)}$$

Where,

τ = Shear stress,

γ = Shear rate,

m = Consistency index, and

n = Power law index

In Equation (1.3), the power law index (n) characterizes the rheological aspects of fluids, while the consistency index (m) describes the fluid's flow resistance. Fluids can be classified into three types based on the value of n: Newtonian behaviour (n=1), non-Newtonian dilatant behaviour (n>1), and non-Newtonian pseudoplastic behaviour (n<1). In this analysis, a power law curve fitting was applied to the shear stress vs shear rate curve to find the values of m and n.

Understanding the flow behaviour is crucial in formulating nanofluids for different applications due to its direct impact on performance and efficiency. Nanofluids, which are suspensions of nanoparticles in base fluids, exhibit unique flow characteristics influenced by factors like nanoparticle concentration, size, and surface chemistry. The flow behavior determines key properties such as viscosity, shear-thinning or shear-thickening tendencies, and stability under varying conditions of temperature and pressure. Tailoring the flow behavior of nanofluids ensures optimal performance in applications ranging from advanced cooling systems in electronics to enhanced lubricants in automotive and industrial settings. By controlling flow properties through careful formulation, nanofluids can achieve desired thermal conductivity, viscosity, and stability profiles, thereby enhancing heat transfer efficiency, reducing energy consumption, and improving overall system reliability and longevity. Fig. 1.13 depicts the research articles published with the topic "***Nanofluid rheology***" at the Web of Science (Clarivate Analytics) up to the present year 2024.
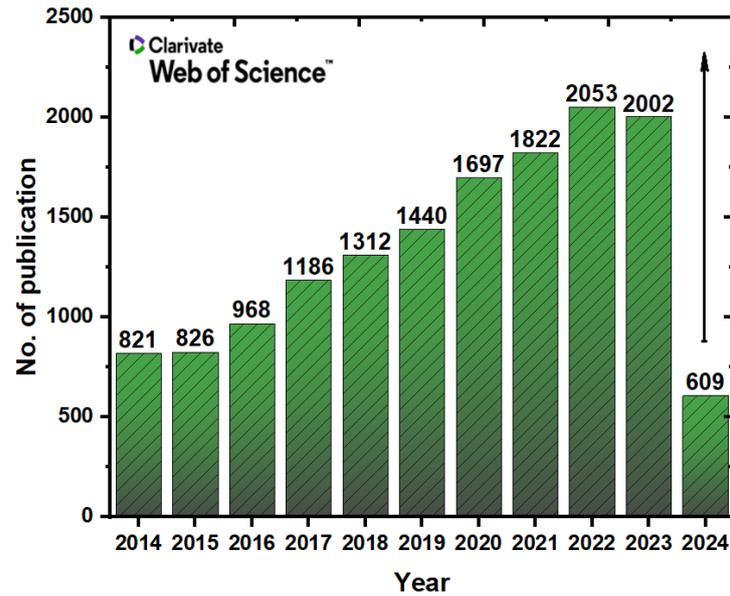
Fig. 1.13: Research publications with "*Nanofluid rheology*" as the key word against year. (*Source: WoS*)

1.6.3: Factors influencing rheology of nanofluids

Concentrations of nano-additives

In most cases, the addition of nano-additive in the lubricating oil is useful in reducing friction and wear [84]. With the changes in nano-additive concentration, the viscosity of nanofluid varies and high concentrations leads to abrupt viscosity rises.

Temperature

The viscosity of nanofluids is strongly affected by temperatures. The research findings have shown that viscosity changes and in general decreases with high temperature [141].

Type, size and shape of nano-additives

The viscosity of nanofluid is influenced by the size and shape of nano-additives. Many research findings have reported that with decreasing the size of nano-additives resulted the enhancement of viscosity of nanofluids [142].

Dispersion stability of nano-additives

To develop the stable nanofluids, it is essential that nano-additives have been homogeneously dispersed in BO. Various surfactants are utilized for homogeneous dispersion of nano-additives in to BO, which provides stability to nanofluids [80]. Surfactant molecules covers the surface of nano-additives and resist the aggregation, which execute stabilization of nanofluids [143].

1.7: Influence of nano-additives on the thermal conductivity of base oil and its nanofluids

1.7.1: Thermal conductivity

Thermal conductivity refers to the ability of a matter to conduct heat. It is a crucial parameter for evaluating the heat transfer performance of a fluid. Higher thermal conductivity in a fluid indicates better heat absorption and dissipation, which is necessary for maintaining optimal performance and reducing heat loss. Fluids with high thermal conductivity are much better at cooling, ensuring the efficient operation of systems and enhancing their overall performance [144-146].

Thermal conductivity is a vital property in several oil applications, where efficient heat transfer is essential for optimal performance and longevity of systems.

- *Lubrication:* In automotive and industrial machinery, high thermal conductivity oils help dissipate heat generated by friction, reducing wear and tear on moving parts and enhancing the lifespan of equipment [14].

- *Dielectric Fluids:* In transformers and other high-voltage equipment, dielectric high thermal conductivity oils effectively manage heat, preventing overheating and ensuring reliable operation [147].

- *Cooling Systems:* EOs and coolants with good thermal conductivity are critical in internal combustion engines, where they help in maintaining appropriate operating temperatures, preventing overheating, and ensuring efficient engine performance [148].

- *Hydraulic Fluids:* In hydraulic systems, high thermal conductivity oils facilitate effective heat dissipation, ensuring smooth operation and preventing system failures due to overheating [149].

- *Heat Transfer Fluids:* In applications such as heat exchangers, high thermal conductivity oils are used to efficiently transfer heat between different parts of a system, optimizing energy use and system efficiency [150].

Vegetable oil-based nanofluids are gaining attention due to their biodegradability, sustainability, and excellent thermal properties. When nano-additives are added to vegetable oils, the thermal conductivity of the base fluid can be significantly enhanced. These nanofluids are particularly useful in applications requiring efficient heat dissipation, such as in cooling systems for electronics, renewable energy systems, and other industrial

processes [151]. The use of NOs as a base fluid also offers environmental benefits, reducing reliance on non-renewable petroleum-based oils and minimizing ecological impact [152].

In EOs, thermal conductivity is a key factor for maintaining engine health and performance. High thermal conductivity oils ensure effective heat transfer from engine components to the cooling system, preventing overheating and maintaining optimal operating temperatures [148]. This is essential for the efficient functioning of the engine, reducing the risk of thermal degradation of the oil, and prolonging the lifespan of both the oil and engine components. Enhanced thermal conductivity in EOs also contributes to improved fuel efficiency and reduced emissions, aligning with environmental standards and performance requirements [153]. The addition of nano-additives to EOs can significantly enhance their thermal conductivity. Nanoparticles such as aluminium oxide (Al_2O_3), copper oxide (CuO), and carbon nanotubes (CNTs) improve the heat transfer capabilities of the oil, further optimizing engine performance and durability [154]. Fig. 1.14 depicts the research articles published with the topic "*Nanofluid thermal conductivity*" at the Web of Science (Clarivate Analytics) up to the present year 2024.
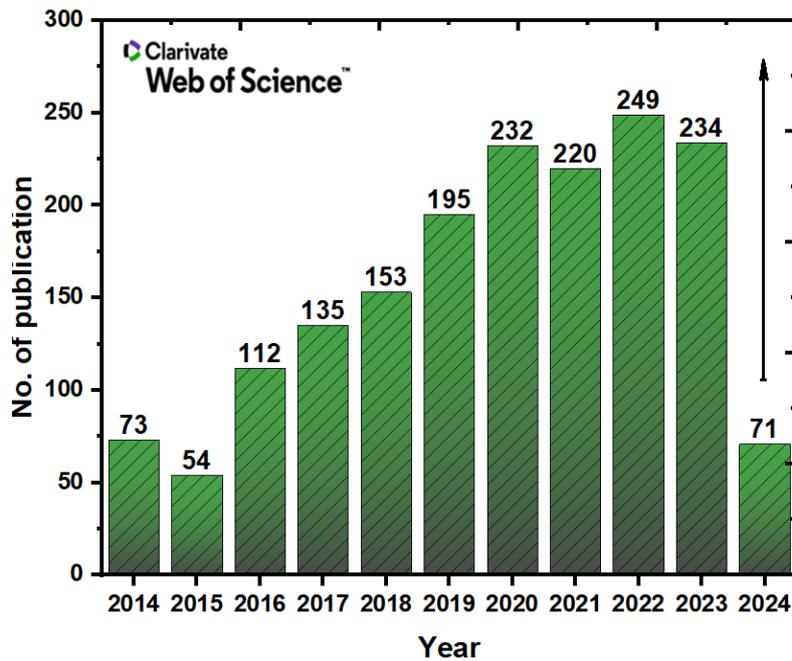
Fig. 1.14: Research publications with "*Nanofluid thermal conductivity*" as the key word against year. (*Source: WoS*)

1.7.2: Thermal conductivity mechanism of nanofluids

Brownian motion and micro convection effect of nano-additives

According to Brownian motion theory, nano-additives suspended in a liquid are influenced by the surrounding liquid molecules, causing the particles to move randomly. The smaller the particle size, the faster and more frequent the movement. This increased movement results in a higher frequency of energy exchange between the nanoparticles and the fluid, thereby enhancing the thermal conductivity of the fluid [155].

Agglomeration and percolation structure of nano-additives

In the energy transfer process within nanofluids, the optimal condition is for nano-additives to be uniformly dispersed in the base fluid at high concentrations without agglomeration. However, due to irregular Brownian motion, nano-additives often collide and aggregate into larger clusters. These agglomerates move more slowly than individual particles, and the increased vertical force accelerates particle precipitation. This agglomeration reduces the rate of energy transfer within the nanofluid, diminishing the enhanced heat transfer performance [156].

Theoretical Studies on Liquid Layer Formed by Nanoparticles and Base Liquid

Experimental observations have shown that an ordered liquid layer nanostructure forms around nano-additives in nanofluids. This solid-like layer plays a crucial role in thermal conduction, enhancing the thermal conductivity of the nanofluid. The thickness of this layer can be expressed by the following formula:

$$d_s = \frac{1}{\sqrt{3}}\left(\frac{4M}{\rho_f N_A}\right)^{\frac{1}{3}} \dots\dots\dots\dots\dots\dots\dots\dots\dots\text{Eq. (1.4)}$$

Where,

M = Mass fraction of the liquid,

ρ_f = Density of the liquid, and

N_A = Avogadro's constant (6.022×1023/mol).

According to this formula, the thickness of the nanostructure depends only on the properties of the base fluid. Other influencing factors need further investigation to determine whether the primary heat conduction mechanism of the liquid layer is more akin to solid or liquid heat conduction [157].

Ballistic transport and nonlocal effect

Ballistic transport occurs when the nano-additives diameter is smaller than the phonon's mean free path and there is no scattering within the molecule. In this scenario, the nanoparticle's internal thermal conductivity is insufficient, making the diffusion capacity at the liquid interface predominant. However, previous reports have identified that this local effect only cannot be the reason for the observed improvement in the thermal conductivity of fluids. Further research is needed to fully understand the mechanisms behind the enhanced heat transfer performance of nanofluids [158].
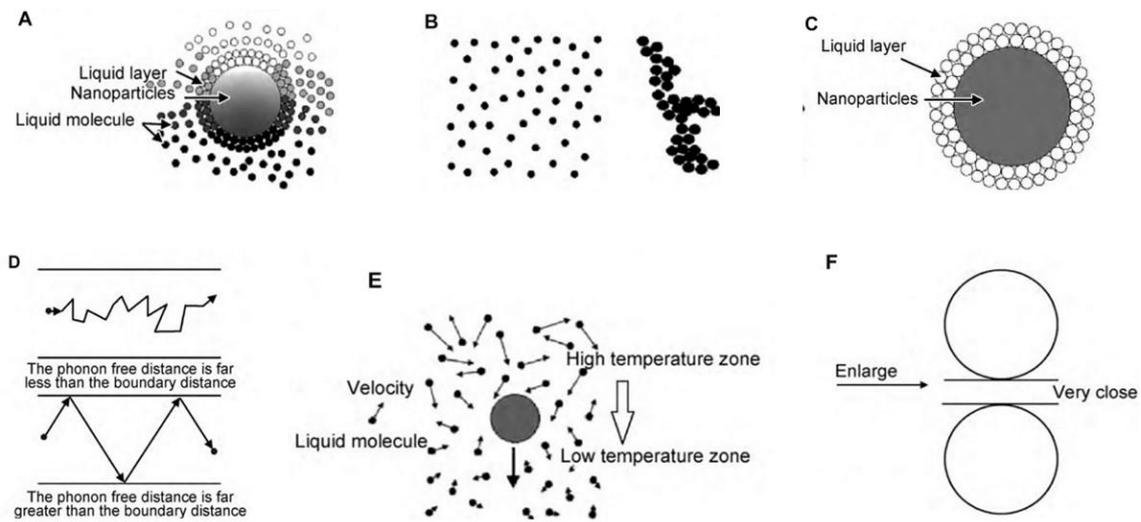

Fig. 1.15: Nanofluid heat conduction mechanism: (a) nano-additive brownian motion and micro convection, (b) nano-additive agglomeration and penetration, (c) nano-additive liquid layer, (d) ballistic transport and nonlocal effects, (e) thermophoresis theory, and (f) radiation heat transfer [157].

Thermophoresis Theory

Thermophoresis occurs due to the presence of a temperature gradient. Molecules on the high-temperature side experience stronger impacts than those on the low-temperature side, causing a directional movement of the molecules. However, experiments have shown that thermophoresis has minimal influence on the thermal conductivity of nanofluids, being several orders of magnitude less significant than the effects produced by Brownian motion [159].

Radiation Heat Transfer

Molecular dynamics simulations have shown that the energy transfer speed between two particles increases sharply when the distance between them is less than 1 µm. This suggests that near-field radiation might significantly impact thermal conductivity.

However, some authors have reported that near-field radiation does not substantially enhance the heat conduction of nanofluids, even when its intensity exceeds that of blackbody radiation [160]. Fig. 1.15 illustrates the schematic representations of each theory discussed:

1.7.3: Factors influencing thermal conductivity of nanofluids

Type of base fluid

Many studies demonstrate that the enhancement of heat conductivity in nanofluids using identical nanoparticles is inversely related to the thermal conductivity of the fluid, regardless of nanoparticle shape [161]. It has been observed that achieving significant improvements in thermal conductivity is more challenging in highly conductive base fluids compared to less conductive ones when incorporating nano-additives.

Nano-additive type and concentration

Nano-additives that are type of nanoparticles used for specific applications for improving thermal conductivity in nanofluids have been researched across various base liquids like water, ethylene glycol, coconut oil, EO, etc. Metallic nano-additives like Cu or Fe enhance base fluid thermal conductivity even at low concentrations. However, metallic nano-additives are less accessible and prone to oxidation during handling. Metal oxide-based nano-additives with lower thermal conductivity, such as Al_2O_3, CuO, and TiO_2, exhibit modest increases in thermal conductivity at concentrations below 2 vol. %. Carbon based nano-additives, such as graphene, CNT and fullerene have highest impact on enhancing thermal conductivity of base fluid [161, 162].

Nano-additive size

Studies with nano-additives diameters ranging from 30 to 60 nm in base fluids such as ethylene glycol, vegetable oil, pump oil, and water have generally shown that larger nano-additives lead to greater increases in thermal conductivity. However, the tendency of larger nano-additives to agglomerate, which was not always accounted for by researchers, could influence these findings [162].

Nano-additive shape

Research suggests that cylindrical nano-additives may enhance base fluid thermal conductivity more effectively than spherical nano-additives. Moreover, the increase in length-to-diameter ratio of the dispersed nanotubes leads to the increase of the thermal conductivity enhancement [157, 161].

Temperature

Experimental studies on the temperature-dependent thermal conductivity of nanofluids have revealed diverse trends in variation. Some reports indicate an increase in relative thermal conductivity (k_{eff}/k_f) with temperature, while others show a reverse trend with a decrease. Additionally, certain studies suggest no significant variation in relative thermal conductivity with temperature. The factors contributing to these varied trends remain unclear. Temperature changes affect the Brownian motion of particles, altering solvent-surfactant interactions at the interface. Furthermore, temperature fluctuations influence the density of molecules adsorbed on nanoparticle surfaces, directly impacting the thickness and thermal conductivity of the interfacial layer [163].

1.8: Present research

The use of nano-additives in different oils has significant potential to enhance their performance properties and extend the life of mechanical systems. Researchers are exploring their applications in areas such as heat exchangers, electronics cooling, and automotive systems to address challenges related to flow, thermal management and energy efficiency. The versatility and promising performance of nanofluids have propelled them into the forefront of scientific investigation and practical implementation [164].

This research aims to study the performance characteristics of different oils by focusing on flow properties through rheology study and thermal property i.e., thermal conductivity. The approach involves the synthesis and evaluation of various nano-fluids, wherein nano-additives are dispersed within BOs. The investigation places emphasis on understanding the effects of specific nano-additives, namely Alumina, Zinc oxide, Graphene nanoplatelets, and Multiwalled carbon nanotubes, when introduced into distinctive BOs like CO and EO. Notably, CO, a non-edible fatty oil with a unique hydroxyl group structure, serves as a distinctive base fluid for synthesizing CO-based nanofluids. Recognizing its distinct properties, such as lubricative qualities, dielectric functionality, and heat transfer potential, CO proves to be an intriguing candidate for nanofluid development. In parallel, India's expansive two-wheeler automotive market underscores the critical role of EO, elevating the significance of SOs. Notably, 10W-30 EO emerges as a standout choice due to its remarkable versatility, delivering optimal performance across a diverse temperature range. The selection of this SO for nanofluid research is motivated by its inherent compatibility with nanoparticle dispersion.

The study employs sophisticated characterization techniques, such as scanning electron microscopy (SEM), x-ray diffraction (XRD), and energy dispersive x-ray spectroscopy (EDX), to analyse the structural and chemical composition of the developed nano-additives. Hydrothermal method was employed to synthesize metal oxide-based nano-additives. The nanofluid development process involved the probe sonication method, ensuring the effective dispersion of nano-additives in the BOs. Rigorous evaluation of the synthesized nanofluids was conducted through rheological analysis using a rheometer and thermal conductivity analysis using a thermal conductivity meter. These analyses provide insights into the impact of nano-additives on the flow and thermal properties of the oils, contributing valuable data to the broader understanding of industrial oil enhancement.

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