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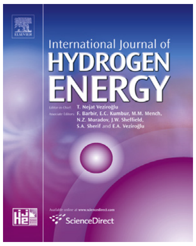

Review Article

Enhancing lubricant properties by nanoparticle additives

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ABSTRACT

A lubricant is derived from mineral oils or synthetic hydrocarbon blends. However, in order to fulfill the requirements set by the Original Equipment Manufacturers (OEMs), they lack properties of their own. A common solution to this problem is to include relatively small but effective additives in the formulation of the base stock, which leads to remarkable improvements to the attribute of the lubricants, such as introducing anti-oxidation capability, tribological characteristics, and thermal properties. Nanotechnology offers the opportunity to improve the performance of lubricant oil via the utilization of nano-additives. The addition of nanoparticles to common base oils is a promising approach towards enhancing certain characteristics, such as friction and wear resistance. This communication overview works on nano-additives in the lubricant industry. It encompasses general base fluids and common oil additives, and more narrow focus such as the application of nano-scale particles. Finally, this communication will highlight the future prospects of nanoparticles in the context of the lubricant industry.

Contents

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Introduction

Tribology is made up of two Greek words; “tribos” and “logos”, the former means “rubbing”, while the latter means “word”. Tribology, in practice, is the science of controlling and managing wear, friction, and lubrication [1–3]. Wear and friction in systems are some of the major reasons for:

Failure of machineries; in engines, gears, bearings, etc., all of which are vital for the smooth operation of mechanical systems in various industries, including automotive, aerospace, mining, and machinery [4], and:

Energy loss; which is an important tribological issue arising from high frictions [5–7]. In a study conducted in 2012 by Holmberg et al. [8], it was concluded that up to one third of energy from fuel is wasted due to frictional losses from engines and other moving parts such as the transmission, brakes, and tires. Their calculations also revealed that if a new friction reduction technologies could be applied to passenger cars, friction loss would decrease by 18% in 5–10 years (results in saving 174,000 million euros globally), and by 61% in 15–25 years (results in saving EUR 576,000 million globally). This trend is reflected in heavy duty vehicles as well [9,10]. Holmberg et al., [11] analyzed trucks and buses and proved that 33% of fuel energy is spent on friction loss in their engines. Moreover, they showed that globally, 180,000 million L of fuel was consumed to overcome friction in heavy duty vehicles in 2012.

In order to overcome the aforementioned issues, the most effective approach is lubricating machineries. Lubricants are broadly used in industry and manufacturing units to protect products and tools from wear and maintain their respective surface quality. Moreover, lubricants optimize the coefficient of friction (COF) of fabrication processes and excess heat accumulating in mechanical systems. As a result of this, the enhancement of lubricant oil properties are of great importance in the context of protecting machineries from highly probable damages and decrease energy consumption [12].

Today, lubricants having various properties are formulated and synthesized for application in various mechanical units and operations. Emerging technologies require intense and varied requirements from lubricants, due to fact that the appropriate formulation of hydrocarbon blends for lubricants being a complex process. Novel lubricants are composed of a wide range of base oils and essential additives. Base oil has a number of crucial roles, but mainly, it is a lubricant fluid that separate the surfaces of moving parts by providing fluid films [13]. In addition to minimizing friction, it removes heat and wear particles from the system. Numerous lubricant properties are improved and formed by adding special additive species to the base stock. Lubricants could be derived from two diverse sources; biological and non-biological. Therefore, an enormous collection of hydrocarbon mixtures is available through a combination of the aforementioned groups [14].

Lubricant additives -in a few weight percent- are added to the base stock in order to completely enhance the lubricative characteristics. They are essential towards maintaining the overall performance of lubricant and is capable of manipulating particular features, for example, friction and wear, clotting, oxidation, foaming, and corrosion tendency [15].

A new lubricant additive that was recently introduced to the industry are nano-additives. Some of the advantages of using nano-additives are possessing suitable size to enter contact asperities, thermal stability, variety of particle chemistries, and reaction rate with the surface without an induction period, which is an important factor for conventional lubricant additives [16–19]. Over the past decades, many studies have stated that the addition of nanoparticles, such as metal [20], metal oxide [21], metal sulfides [22] and [23], carbonate [23], borate [24], carbon materials [25], organic material [26] and rare-earth compound [27] to lubricants is effective in decreasing both friction and wear [28]. The friction-reduction and anti-wear behaviors are improved due to individual features of the nanoparticles, for example, their size, shape, and physicochemical nature [29].

This work will review all known nano-additives used in the lubrication industry. It will also analyze emerging sciences in the context of base oil and additives.

Base oil

Lubricants are synthesized from three different base oils; mineral, synthetic and biological; , all showing dissimilar assets and suitable for diverse applications. In the industry, the most commonly used lubricant is mineral oil. Mineral oil are petroleum-based fluids and utilized for machineries, which requires its temperature be moderated. They are commonly used in turbines, engines, gears, and bearings. Synthetic oils are specially formulated to produce lubricants with superior properties than mineral oils, for example lubricating at high or low temperatures [15]. Biological lubricants are typically utilized in food or pharmacological industry, such as bakery ovens or kilns, where the risk of contamination needs to be minimized. Sources for biological oil are vegetables and animals. Palm, rape-seed, and castor oils are examples of vegetable oil, while sperm, fish oil, and lanolin are sourced from animals. In the following section, a number of the key characteristics of minerals and synthetic oils will be discussed.

By selecting a suitable base stock for lubricant formulation, a variety of final product properties and performance can be evaluated and forecasted. Being aware of the base fluid features, particularly all possible limitations, is of great necessity for the effective formulation of lubricants. The American Petroleum Institute (API) categorized lubricant base oil quality, as indicated in Table 1 [30]. The latest base oil categories are described in API 1509 (API 2007). Table 1 includes some necessary information on different groups of base oils [31].

Mineral oils

As the most commonly used base stock, mineral oils are manufactured from fractional distillation and the refinement of crude oil. This is done to eliminate high molecular weight paraphenes (to prevent wax deposition while lubricating), aromatic hydrocarbons (to slow down the decrease in oil viscosity), and compounds with sulfur and nitrogen (to prevent corrosion of wearing surfaces).

Based on the source of crude oil and the refining method being used, mineral oils can widely vary from one another. Chemical structure, sulfur content, and viscosity are some of the factors that results in the main differences observed in mineral oils. In the context of chemical forms, three basic forms, known as paraffinic, naphthenic, and aromatic is dependent upon the source of the crude oil. Regarding the sulfur content in oil, a small amount of sulfur is required to induce oxidation and lubrication properties. However, if this amount exceeds 1%, the seals will undergo a rapid process of corrosion. The viscosity of common mineral oils falls within 5 [cS] to 700 [cS] at room temperature.

Synthetic oils

Over the last century, the application of synthetic lubricants has increased steadily, particularly for certain applications that could not tolerate mineral oils. Although mineral oils possess a number of positive characteristics, including availability and low cost, they simultaneously exhibit some serious weaknesses, such as solidification at low temperatures, viscosity loss and oxidation, and the combustion or explosion in the presence of oxidizing elements.

Table 1 – American Petroleum Institute (API) categories for base oils [31].

Group	Properties
Group I	Saturates are <90%, and sulphur is >0.03%, and VI is >80 and <120
Group II	Saturates are >90%, and sulphur is <0.03%, and VI is >80 and <120
Group III	Saturates are >90%, and sulphur is <0.03%, and VI is >120
Group IV	Polyalphaolefins (PAO)
Group V	All other base stocks not included in Group I, II, III, or IV

These types of synthetic lubricant are commonly used for:

- Synthetic hydrocarbon lubricants

There are a huge number of hydrocarbon fluids that could be used as a lubricating agent. However, production economics rigorously restricts their respective applicable range. Oils currently being synthesized by oil refineries is low-cost to produce, and consequently cost-effective for applications requiring bulky volumes such as engine oils. Synthetic hydrocarbons could be classified as following:a) polyalphaolefins [32,33], b) esters [34–36], c) cycloaliphatics [15] and d) polyglycols [15,37].

- Silicon analogues of hydrocarbons

This group consist of multiple synthetic lubricants that are proved to be chemically stable. Two main categories of compounds are of significance: silicones [38,39] and silahydrocarbons [15]. Although the compounds in both classes contain silicon, their molecular structure differ quite significantly.

- Organohalogens

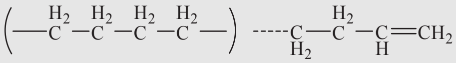
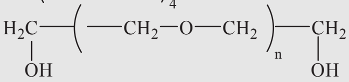
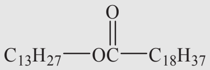
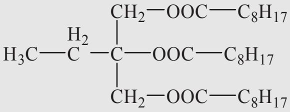
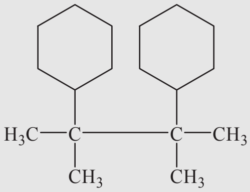
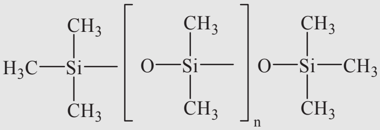
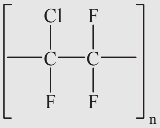
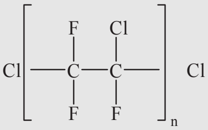
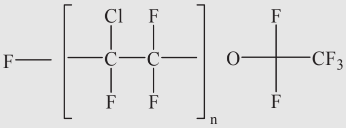
Organohalogens and their respective permutations with halogenated hydrocarbons are known for being anti-oxidation. Previously, machines, for example air or oxygen compressors, are dependent on pure sulfuric acid. Due to the fact that it could be utilized as a lubricant for steel, preventing acids from mixing with moisture and contamination from compressed gas remains a problem. Corrosion is another problem associated with the usage of sulfuric acid. Fluorine and chlorine-thanks to their excellent oxidation and thermal stability-are used to develop compounds with the desired properties. The most common synthetic lubricants that falls in this group are perfluoropolyethers [40–42], chlorofluorocarbons, chlorotrifluoroethylenes [43], and perfluoropolyalkylethers [44–46]. Typical chemical structures and properties of all aforementioned groups are summarized in Table 2.

Work on the development of high-performance lubricants for extreme conditions of vacuum and temperature (cryogenic and high) or for applications in microelectromechanical and nanoelectromechanical systems (MEMS and NEMS) is currently ongoing. Recently, two new groups of promising advanced lubricants have emerged: ionic liquid lubricants and mesogenic lubricants [14,47–53].

Additives

Base fluids play a crucial role in lubricating and separating moving surfaces while eliminating heat, wear, and

Table 2 – Chemical structures of the most common synthetic lubricants [15].

HYDROCARBON SYNTHETIC LUBRICANTS	Polyalphaolefins e.g	 $(-\overset{H_2}{C}-\overset{H_2}{C}-\overset{H_2}{C}-\overset{H_2}{C}-)\text{-----}\underset{H_2}{C}-\overset{H_2}{C}-\underset{H}{C}=CH_2$
	ESTERS E.G	
	Diesters e.g	$C_8H_{17}-O-CO-C_8H_{16}-CO-O-C_8H_{17}$
	Phosphate esters e.g	$(H_3C-C_6H_4-O)_3\ P\equiv O$
	Silicate esters e.g	$Si(HO-C_8H_{17})_4$
	Polyglycol esters e.g	 $\underset{OH}{H_2C}-(-CH_2-O-CH_2)_n-\underset{OH}{CH_2}$
	Fluoro esters e.g	$F(CF_2)_4\ CH_2OOC\ (CF_2)_4\ F$
	Fatty acid esters e.g	 $C_{13}H_{27}-O\overset{\overset{O}{\Vert}}{C}-C_{18}H_{37}$
	Neopentyl polyol esters e.g	 $H_3C-\overset{H_2}{C}-\overset{\overset{CH_2-OOC-C_8H_{17}}{\vert}}{\underset{\underset{CH_2-OOC-C_8H_{17}}{\vert}}{C}}-OOC-C_8H_{17}$
	Cycloaliphatic e.g	
	Polyglycols e.g	$HO-CH_3-CH_3-O-CH_3-CH_3-O\text{----}CH_3-CH_3-OH$
SILICON ANALOGUES OF HYDROCARBONS	Silicones e.g	 $H_3C-\overset{CH_3}{\underset{CH_3}{Si}}-\left[O-\overset{CH_3}{\underset{CH_3}{Si}}-\right]_n O-\overset{CH_3}{\underset{CH_3}{Si}}-CH_3$
	Silahydrocarbons e.g	$C_{12}H_{25}\ Si\ (C_6H_{13})_3$
ORGANOHALOGENS	Perfluoropolyethers e.g	$F_3C-CF_2-O-CF_2-CF_3$
	Chlorofluorocarbons e.g	 $\left[-\overset{Cl}{\underset{F}{C}}-\overset{F}{\underset{F}{C}}-\right]_n$
	Chlorotrifluoroethylenes e.g	 $Cl\left[-\overset{F}{\underset{F}{C}}-\overset{Cl}{\underset{F}{C}}-\right]_n Cl$
	Perfluoropolyalkylethers e.g	 $F-\left[-\overset{Cl}{\underset{F}{C}}-\overset{F}{\underset{F}{C}}-\right]_n O-\overset{F}{\underset{F}{+}}-CF_3$

contamination from the system. However, in any lubricated oil blend, suitable additives needs to be included to enhance certain properties, such as oxidation stability, anti-friction and wear, anti-corrosion, and stability against biological degradation. Another issue is that the base fluid -as a carrying agent for additives-should be able to keep the additive in the solutions in all operating conditions [13]. Almost 10 weight percent of the final lubricant product are made up of additive packages. However, this may vary significantly, based on applications [54]. Based on Rudnick [55], the additives could be categorized in the manner shown in Table 3.

Nano-additives in lubricant oil

To control systemic wear and friction, studies on lubricants containing nanoparticles has significantly increased over the past few years. Wide range of work has been done on both organic and inorganic nanoparticles for applications as extreme pressure (EP) and anti-wear agents. Friction researchers discussed multiple views pertaining to adsorption, penetration, and tribo–chemical reaction for the friction reducing characteristic and anti-wear mechanisms of nanoparticles. Studies have shown that nanoparticle additives

Table 3 – The common lubricant additives being used in industry.

	Additive	Effect	Common chemicals
Deposit control additives	Anti-oxidants	By preventing lubricant form oxidation, these products prevent the formation of corrosive components. Anti-oxidants act by two different behaviors: Peroxide inhibition and radical scaventing [54]	• Sulfur compounds • Sulfur-nitrogen compounds • Phosphorous compounds • Sulfur-phosphorous compounds • Aromatic amine compounds • Hindered phenolic (HP) compounds • Organo-copper compounds
	Zinc dithiophosphates (ZDDP)	ZDDP is commonly used as anti-oxidant and wear additive. ZDDP under high temperature and pressure create a glassy phosphate layer on Fe-based surfaces and reduce wear and friction [56,57].	ZDDP
	Ashless phosphorus–containing lubricating oil additives	This group of additives, in contrast to ZDDP, create a smoother and thinner films than can protect surface against wear. They proved to have lower friction than ZDDP [58–60].	• Phosphate esters: a) Neutal phosphates b) Acid phosphates (Non-ethoxylated, Ethoxyalkyloxy) • Phosphites and phosphonates • Alkyl or aryl phosphites/ phosphonates
	Detergents	Detergents by containing base components neutralize acids that can attack metal surfaces. Other function of detergents is to suspend polar-oxygenated components in oil [61,62].	The metal salts of following acids are being used as detergents: • arylsulfonic acids • alkylphenols • carboxylic acids • petroleum oxidates
	Dispersants	Combined with detergents, dispersants are designed to suspend the insoluble particles and contaminants in oil and keep the surfaces clean. Having polar groups, dispersants can keep nonpolar molecules suspended in lubricant [63,64].	

(continued on next page)

Table 3 – (*continued*)

	Additive	Effect	Common chemicals
Film-forming additives	Solid lubricants as friction modifiers	Being used as additive in oil, solid lubricants considerably reduce friction between surfaces where liquid lubricants do not perform desirably. Five main properties that these group should have are: Yield strength, Adhesion to substrate, Cohesion, Orientation, Plastic flow	• Graphite • Molybdenum disulphide • Boron nitride • Polytetrafluoroethylene
	Organic friction modifiers	In order to adjust friction characteristics and improve the lubricity and energy efficiency, friction modifiers are the added in boundary and/or mixed lubrication conditions. Friction is known to be responsible for about 20–25% of fuel energy consumption [65].	Organic friction modifiers can be found in the following categories: • Carboxylic acids or their derivatives, for example, stearic acid and partial esters • Amides, imides, amines, and their derivatives, for example, oleylamide • Phosphoric or phosphonic acid derivatives • Organic polymers, for example, methacrylates
Anti-wear additives and extreme-pressure additives	Ashless anti-wear and extreme-pressure additives		
	Sulfur carriers	This term is applied to group of extreme pressure and anti-wear additives that contain sulphur in their oxidation and they only contain one kind of heteroatom which is oxygen [66].	• Sulfurized Isobutene • Active-Type Sulfurized Olefins • Inactive Sulfurized α-Olefins • Sulfurized Synthetic Esters (Light Color) • Sulfurized Fatty Oil (Black Color) • Sulfurized Fatty Oil/Olefin Mixture (Light Color)
Viscosity control additives	Olefin copolymer viscosity modifiers (OCP)	OCPs by having relatively low cost and high thickening efficiency they are one commonly utilized viscosity modifier for engine oils [67,68].	
	Polymethacrylate viscosity modifiers (PMA) and pour point depressants (PPD)	PMAs by thickening the oil film improve the viscosity index (VI) of lubricant. PPDs control wax crystallization and deposition in mineral lubricant. One application of PMAs is being utilized as PPD by interacting with waxes chain length with their alkyl side chains.	Various methacrylate monomers are used for PMA construction.
	Pour point depressants (PPD)	PPDs modify crystal size of wax and control their shape during growth. The additives do not fully stop wax crystal growth, but rather reduce the temperature at which a rigid structure is formed [69–71]	• Acrylates • Alkylated • Styrenes • Alpha olefins • Ethylene/vinyl acetates • Methacrylates • Olefin/maleic anhydrides • Styrene/acrylates • Styrene/maleic anhydrides • Vinyl acetate/fumarates.

have superior tribological properties than traditional solid lubricant additives [21,72–75].

Some mechanisms are believed to be the main reason that nano-additives can reduce friction and wear, such as size effect, colloidal effect, protective film, and third body effects [12,76,77]. In this review, studies on nanoparticle additives are categorized into five main groups, which are:

Metals

Metal nanoparticles are widely used in many different applications, such as semiconductors, magnetics, catalysts, and photonic fields. Recently, tribological properties of metal nanoparticles have been of interest to researchers. For example, in addition to tribological effectiveness, Cu nanoparticles showed excellent self-repairing properties, while also being environmentally friendly [12,20,76,78–81]. However, due to high surface activity, these nanoparticles are only weakly compatible with base oils. However, this problem could be mitigated by surface modification techniques. In Padgurskas et al. [82], the effect of different metal nanoparticles on mineral oil was analyzed. The nanoparticles, including Fe, Cu, and Co, and their respective mixtures were tested. Some surface investigations were performed to determine the wear effect of the aforementioned particles. Fig. 1 shows how Fe, Cu, and Co exhibit different behaviors when added to SAE 10 oil. From Scanning Electron Microscope (SEM) images, it could be concluded that Cu nanoparticles possess the most effective wear resistance capabilities. Moreover, it was confirmed that a mixture of nanoparticles are more effective than using them on their own.

In Zhang et al. [28], the effect of Cu nanoparticles on the tribological behavior of diesel oil with serpentine powder as its nanoparticle was investigated. When the concentration of Cu nanoparticles was 7.5 wt%, maximum friction and wear reduction property was observed.

Zhang et al. [83] investigated the effect of Sn and Fe nanoparticles, which they were added to multialkylated cyclopentanes (MACs). MACs have been used as lubricants in the space industry. These nanoparticles reduced friction, wear, and heat in MACs oil. Due to the better solubility of Fe in steel bass surfaces, its nanoparticles offer better anti-wear ability, as is obvious in Fig. 2.

Metal oxide

Metal oxides are commonly added to lubricant base fluids as additives, and the resulting combination used for anti-friction and anti-wear applications:

Nano-TiO_2

Nano-TiO_2 is considered as an effective heterogeneous catalyst for ring-opening of epoxides. It was proven that even after several cycle of reactions, the catalyst remained unweakened. Nano-TiO_2, as additives in API-SF engine oil and mineral oil, demonstrated acceptable friction reduction and anti-wear behaviors. Wu et al. [29] analyzed TiO_2 and also CuO behaviors as nano-additives to lubricant oil. It was shown that the addition of two different nanoparticles to oil decreases its friction (CuO performed better than TiO_2). In addition, both exhibited uniform dispersion and distribution in base oil [29]. Previously, the effects of various nanoparticles as anti-wear additives for exclusively mineral and synthetic-based lubricants were comprehensively described. Nevertheless, in 2013, Arumugam and Sriram studied the influence of nano and microscale particles on the tribological behavior of chemically-modified vegetable oil. They chemically modified raw rapeseed oil by epoxidation, hydroxylation, and esterification to improve oxidation stability and cold flow behavior. They reported that the addition of TiO_2 nanoparticles improve the lubricating properties (15.2% reduction in friction coefficient) of rapeseed oil better than metal oxide micro particles (6.9% reduction in friction coefficient). This is attributed to the fact that spherical TiO_2 particles have a lower aspect ratio. In addition, the 0.65 mm diameter wear scar for the chemically-modified rapeseed oil was reduced by 11 and 6.1% for nano and micro scale TiO_2, respectively. Moreover, the solubility of TiO_2 nanoparticle in rapeseed oil seemed to be adequate, and it was observed that the particles did not settle even after 80 h [84].

Only very few studies distinguishes the rutile and anatase phases' tribological behavior of TiO_2. Recently, Ingole et al. conducted fundamental friction and wear study of the mixture of rutile and anatase phases of the P25 and anatase phase of TiO_2. Their investigation showed that 0.25 wt% addition of TiO_2 nanoparticles can reduce and stabilize the coefficient of friction. This effect could be attributed to the uniform formation of TiO_2 films on sliding surfaces. P25 slightly increased the friction coefficient [85].

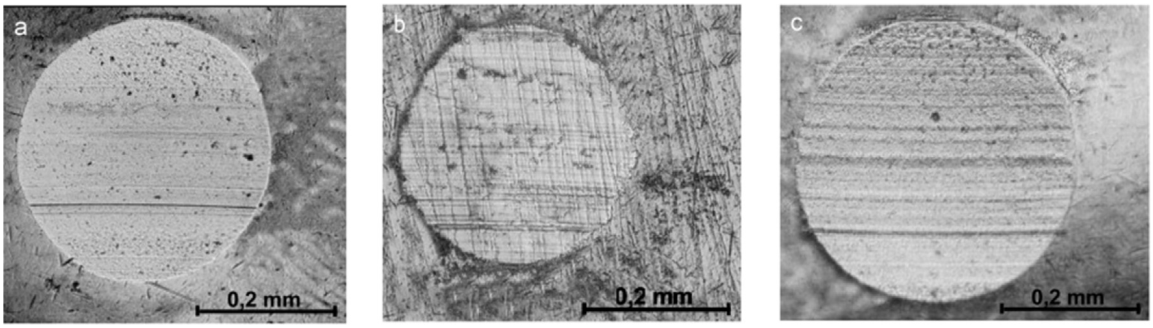


Fig. 1 – SEM images of scar resistance behavior of a) Fe nanoparticles, b) Cu nanoparticles, c) Co nanoparticles [82].

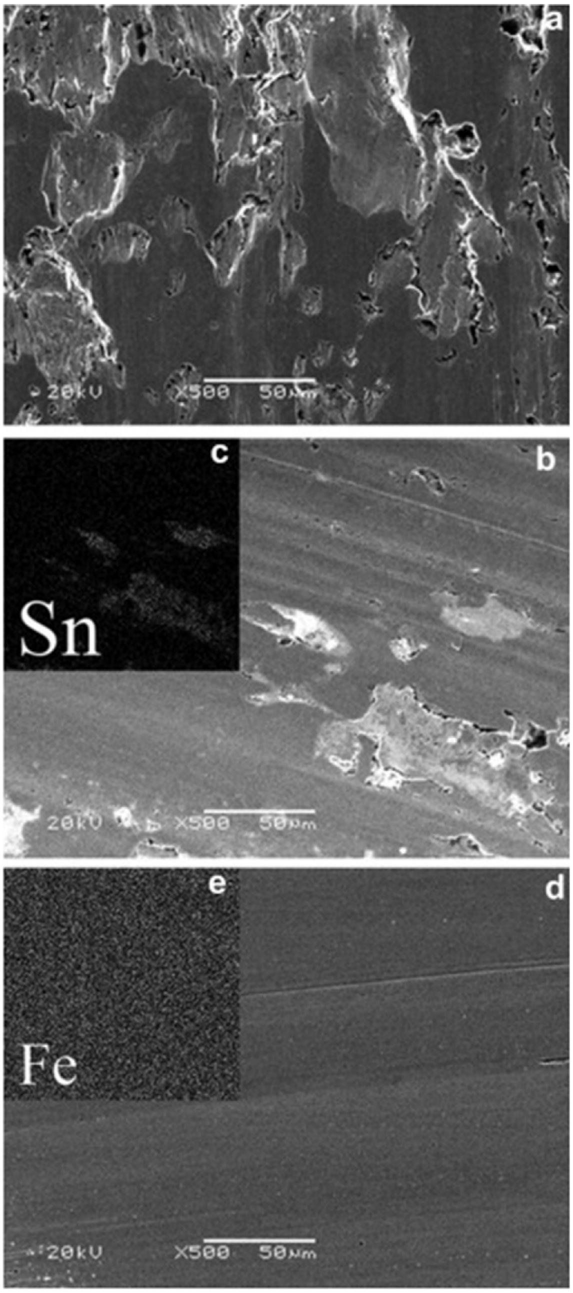


Fig. 2 – SEM micrographs of surface for a) MACs oil, b) MACs +1% Sn, c) Sn elements mapping of b, d) MACs +1% Fe, e) Fe elements mapping of d [83].

Compared to amorphous TiO_2, anatase TiO_2 has superior thermal stability, and therefore demonstrates outstanding tribological behaviors. However, more investigations into synthesizing highly pure monodispersed anatase nanoparticle is necessary. Therefore, in a study by Zhabg et al. [86], anatase nanoparticles were modified with steric acid, and the product's tribological behavior when used as an additive in liquid paraffin was investigated.

In another work, Sabareesh et al. [87] investigated the effect of the addition of low volume fractions of titanium oxide nanoparticles in mineral oil lubricant. Their main objective was to measure the coefficient of performance (COP) -which is the ratio of the heat transfer rate at the evaporator to the power input to the compressor- of a vapor compression refrigeration system. With the addition of small amounts of nanoparticles into the lubricant, it was noted that COP improved significantly. The viscosity was measured using a pin-on-disc tool. As the content of nanoparticles was increased, the coefficient of friction was decreased. However, this trend was only evident up till 0.01% nanoparticle in oil. The addition of these particles beyond 0.01% increases the coefficient of friction.

Some other studies pertaining to the tribological behavior of titanium oxide nanoparticles are also available in Refs. [88–91].

Nano-ZnO

Nano-ZnO has been grabbing considerable attention thanks to its characteristics of large surface area, high surface energy, strong adsorption, high diffusion, easy sintering, and a low melting point [92]. In addition, it is easy to prepare nano-ZnO, therefore, it is a common metal oxide, and its addition to base oil will not only improve the tribological behavior of lubricant, but also considerably reduce costs. Nevertheless, due to the low solubility of ZnO in oil, their dispersion in the base oil could prove to be a challenge [93]. In 2011, Jianhua et al. [94] prepared ZnO by homogeneous precipitation method using lauryl sodium sulfate (SDS) as the surfactant, and studied the oil solubility, anti-corrosion, and tribological properties of prepared nano-ZnO being used as a lubricant additive. They results from SEM showed that the average size of ZnO particles reached 125 nm. Taking into account the solubility, the addition of 1.0%, 2.0%, 3.0%, and 4.0% mass fraction of ZnO resulted in oil samples being clear and unstratified after being left for 10 days. ZnO nano-scale particles could decrease the wear of direct contact area of friction by being depositing onto the sliding surfaces and forming a lubricating layer on moving surfaces. The friction reduction and anti-wear properties of base oil were significantly improved by the addition of surface-modified nano-sized ZnO particles.

Using the ball-on-disk tests, Gara and Zou [95] investigated the friction and wear properties of ZnO and Al_2O_3 as water-based nanofluids. The combination of these nanoparticles reduces friction for smooth surfaces. Wear analyses for a polished surface show that the nanoparticles acted in a manner similar to abrasive wear particles, resulting in wear tracks on the surfaces, with harder particles resulting in more visible wear tracks.

Nano-$ZnAl_2O_4$

$ZnAl_2O_4$ powders have been broadly utilized in electroconductive materials and aero-space machineries as ceramic, catalyst, anti-thermal coatings. Recently, $ZnAl_2O_4$ nanoparticles are being intensely investigated due to their excellent properties that are compatible for anti-friction agent, which are high thermal stability, high mechanical resistance, hydrophobicity, and low surface acidity. Therefore, it becomes prudent that $ZnAl_2O_4$ nanoparticles additive be analyzed for compatibility with lubricant oils. Previously, only a few studies focused on the dispersion tendency of nanoparticles in water or alcohol. Similar to other nanoparticles, $ZnAl_2O_4$'s poor stability in organic solvents and oil highly restricts their applications as additives. As a result of this, the synthesis of monodispersed $ZnAl_2O_4$ nanoparticles with good dispersion stability in organic solvents is of great importance. Song et al. [96] synthesized and modified the surface of $ZnAl_2O_4$ nanoparticles by the solvothermal method with oleic acid in one step. The narrow size distribution and acceptable dispersion in solvent oil was observed. When nanoparticles are modified, they will be nearly monodispersed in lubricant. The nanoparticles of $ZnAl_2O_4$ significantly increase anti-wear and anti-friction property of oil, with the optimum concentration of 0.1 wt. %. In addition, compared to Al_2O_3 and ZnO, $ZnAl_2O_4$ exhibited superior performance.

Metal oxide composite nanoparticles

Furthermore, recent studies have shown the importance of using composite nanoparticles, which demonstrated better tribological properties than single-component nanoparticles [97]. Among the aforementioned nanoparticles, studies have shown that Al_2O_3 and TiO_2 nanoparticles, as additives, are perfect for anti-wear and anti-friction applications, respectively. Therefore, Al_2O_3/TiO_2 nanocomposite is expected to be excellent for good anti-wear and anti-friction tribological applications.

In Wei et al. [98], the tribology of Al_2O_3–TiO_2 nanocomposites were analyzed via friction and wear tests. The nanocomposites were ~75 nm, and exhibited acceptable stability in base oil. The addition of only 0.1 wt% of nanocomposites improved the antiwear performance and reduced the coefficient of friction. More interestingly, Al_2O_3/TiO_2 nanoparticles exhibited better anti-wear and anti-friction properties than pure Al_2O_3 or TiO_2.

In Le [99], composite nanoparticles of ZrO_2 and SiO_2 were modified using aluminum zirconium and used as additives in lubricating oil. Then, the performance of the final product was examined under various applied load and concentration fraction. It has been proven that the lubricating properties were significantly improved by the addition of these modified nanoparticles. The friction coefficient reduction was around 16.24% when an optimal concentration of 0.1 wt% was added to the base oil.

Table 4 summarizes some other earlier studies conducted on metal oxide nanoparticles and their composites.

Nano-carbon materials

Using carbon materials as nano-additives for lubricant oil is an almost recent innovation. Some tribological behavior of these materials, such as graphite, diamond, and fullerene has been studied in Refs. [104–108]. For example, Fig. 3 shows how various nanocarbon materials can affect the lubricative behavior of the lubricant.

Table 4 – Earlier studies on metal oxide nanoparticles as lubricant additives.

Author	Nanoparticle
Battez et al. [21]	CuO, ZnO, ZrO_2
Wu et al. [29]	CuO, TiO_2
Battez et al. [100]	CuO
Shi et al. [101]	Al_2O_3
Song et al. [96]	$ZnAl_2O_3$
Mangam et al. [102]	Cu, CeO_2
Jia et al. [103]	Al2O3, SiO_2

Nanocarbon materials can be divided to four main allotropes which are: zero-dimensional (0D), one-dimensional (1D), two-dimensional (2D) and three-dimensional (3D).

Zero-dimensional carbon nanomaterials

Lee et al., in 2007 [109] investigated the effect of various volume concentration of fullerene nanoparticle in mineral oil. A disk-on disk testing facility was used in the experiments to obtain the friction surface temperature and friction coefficient. These two parameters were estimated for raw mineral oils and oils, including nanoparticles, by changing the variables such as the volume fraction of additive fullerene and normal forces. The frictional surfaces wear and the coefficient of the fraction is controlled via the volume concentration of the fullerene nanoparticles in oil. Furthermore, the reduction of contact surfaces of moving parts is achievable via the addition of nanoparticles to the lubricants [109].

Ku et al. [110] evaluated the extreme pressure and anti-wear properties of fullerene with disk-on-disk tester for multiple viscosities. The presence of fullerene nanoparticles reduced friction, mostly via the reduction of contacts between metal surfaces. Moreover, it was illustrated that fullerene is more effective when added to oils with lower viscosities.

In a more recent study, Ettefaghi et al. [111] investigated the effectiveness of fullerene nanoparticles when added to 20W50 oil, and compared its performance to other carbon nanomaterials. For dispersing nanoparticles in oil, the planetary ball mill method was used. The stability of fullerene nanoparticles in oil was reported to be acceptable, as after 720 h, there were no precipitation observed. It was also pointed out that fullerene resulted in the lowest thermal conductivity increase amongst all other carbon nanomaterials.

One-dimension carbon nanomaterials

Nanotubes, nanowires, and nanorods, which are regarded as one-dimensional nanomaterials, have a wide range of applications [112,113]. Carbon nanotubes (CNTs) have been of interest to various applications over the past decade. However, due to the chemical inertness of CNTs, their dispersion in solvents remains quite difficult. Therefore, in the context of lubricative additives, the issue that should be addressed first is its stability in the base oil. In 2005, Chen et al. [114] started studying multi-walled carbon nanotubes (MWNTs) as an oil additive. They modified MWNTs by performing treatments using sulfuric and nitric acids, and refluxing with stearic acid (SA) to enhance the tribological properties. They proved that the ability of nano-lubricant for wear and friction reduction depended not only on the tribological behavior of the nanoparticles, but also on the dispersion pattern of particles in oil. Fig. 4 shows how the size and density of aggregates were modified after treatment with SA. They pointed out that modified MWNTs are stable in oil for more than six months, while for unmodified particles, it is only two months.

In 2013, Ettefaghi et al. [115] investigated the effects of MWCNTs on viscosity, flash point, pour point, and thermal

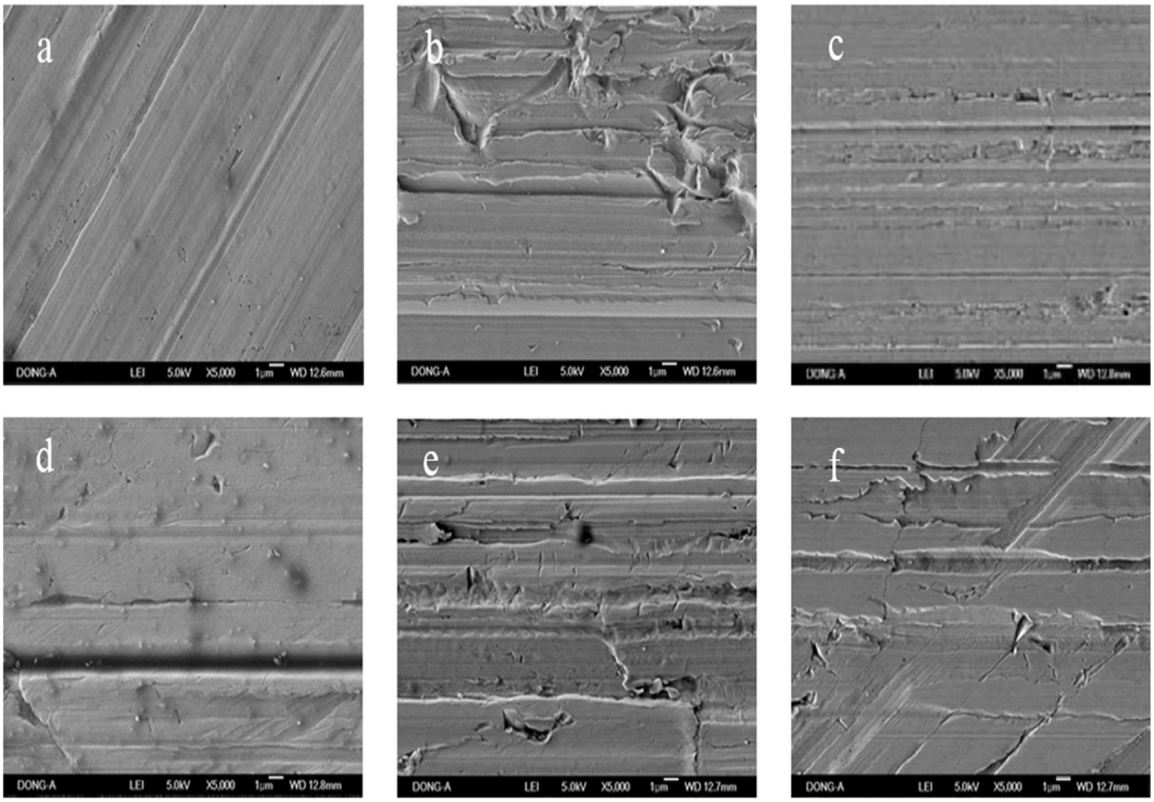


Fig. 3 – SEM images of surface showing; a) Initial condition of surface, b) After testing with raw lubricant, c) After testing with raw lubricant + graphite, d) After testing with raw lubricant + carbon black, e) After testing with raw lubricant + graphite nanofibers, f) After testing with raw lubricant + carbon nanotubes [75].

conductivity. In order to prevent particles from agglomerating and precipitating in oil, they utilized the planetary ball mill. They continued this by increasing the concentration of nanotubes by 0.2 wt%, resulting in an increase of 13% and the 3.3% to the flash point and pour point of oil, respectively. However, this increasing trend increases the content of nanotubes. The lubricating properties of oil may decrease by increasing the amount of nanotubes, which could be the result of agglomeration. Taking into account all these countercurrent effects, they reported that 0.1 wt% of MWCNTs in oil could be regarded as an optimal amount.

Two-dimensional carbon nanomaterials

Graphene is a two-dimensional material arranged in honeycomb lattice, offering significant anti-wear and friction properties. It also has suitable thermal, electrical, mechanical, and optical properties, and is a good candidate for lubricating machineries [116,117]. However, until now, only a few numbers are devoted to the evaluation of the tribological properties of graphene.

In most nano-scale computational and experimental studies on graphene, the tribological behavior was shown to rely on stacking and other structural features, as well as the

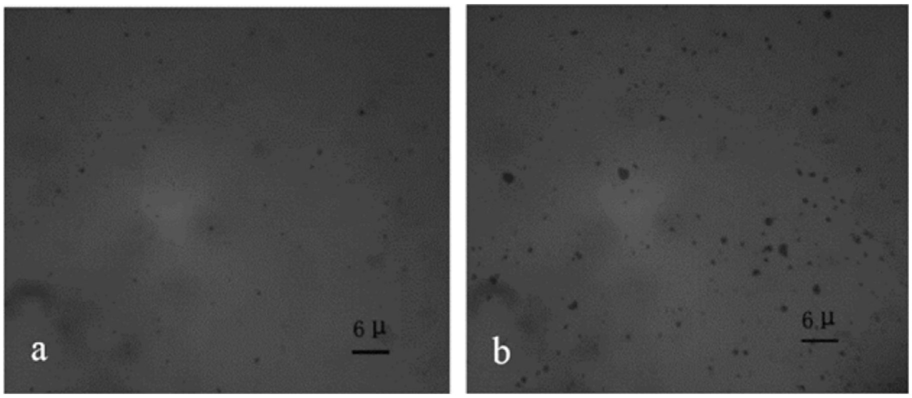


Fig. 4 – Micrograph of CNTs agglomerates in lubricant oil. a) Unmodified MWNTs, b) Modified MWNTs with SA.

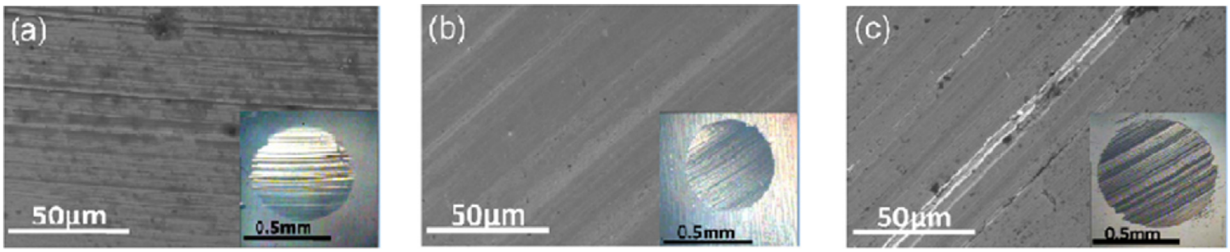


Fig. 5 – SEM images of wear scars a) Pure base oil, b) 0.06 wt% of graphene, c) 5 wt% graphene.

nature of the sliding surfaces. This observation is assumed to be due to two systems: (a) the friction of a nano-scale atomic force microscopy (AFM) tip sliding on a surface of graphene layers (which simulates AFM lateral force measurements), and (b) the friction between the graphene layers themselves. In (a), it was shown that the friction decreases with the increase of the number of layers, while for (b), the friction was found to behave in an opposite manner, achieving higher friction or stick-slip behavior once the number of layers exceeded three.

Using focused solar electromagnetic radiation to exfoliate graphite oxide, Eswariah et al. [118] synthesized high deoxygenated grapheme, which are less defective, and super hyphobic grapheme, which could easily disperse in engine oil. The wear scar diameter (WSD) and frictional characteristics (FC) decreased by 33 and 80% respectively, when 0.025 mg/mL of graphene is added to engine oil. Therefore, without surface modification, graphene is capable of reducing the frictional coefficient. Due to coalesce and separation of nanoparticles, FC and WSD grow when the graphene concentration is increased.

In Zhang et al. [119], graphene was modified using oleic acid to enhance its solubility in base oil. They posited that an optimum amount of graphene can significantly improve the wear behavior of the lubricant. Fig. 5 show the clear effect of three different concentrations of graphene particles in the SEM images.

In order to make graphene platelets stable in the base oil, Lin et al. [120] tested a number of modifiers, such as sodium dodecyl benzene sulfonate, stearic acid, dodecyl trimethyl ammonium chloride, oleic acid, sorbitan monooleate, as well as polysorbate and others. They concluded that among all of those chemicals, stearic and oleic acid are the most suitable modifiers. Using 0.075 wt % optimal concentration of modified graphene platelets (MGP) as an additive, the wear and load-carrying of oil is enhanced. Raw base oil or base oil with modified natural flake graphite (MNFG) show higher friction coefficient than oil with MGP.

Three-dimensional carbon nanomaterials

Although the micro scale particles of diamond are being widely used in the industry as a polishing material, it has been proven that nano-sized particles of diamond could act as ball bearings between sliding parts of a machine [121,122]. Table 5 summarizes some of these studies.

Graphite has also been used both as a powder lubricant and additive. Being used as solid powder lubricant, graphite has friction coefficients of 0.5–0.6 and 0.1–0.2 in dry and humid conditions, respectively. Graphite has been widely used as micro-scale powder lubricant in the industry [127–129], however, a detailed review of their use is beyond the scope of this communication.

Lee et al. [130] studied graphite as a lubricant additive for industrial gear oil, with a kinematic viscosity of 220 *cSt* (at 20 °C). Fig. 6 shows the SEM images of a) initial condition of surface and b) lubricant + nanoparticle (0.5 vol%). In this work, it was concluded that graphite nanoparticles decreases the metal contact between sliding surfaces by acting as ball-bearing spacers.

Martorana et al., in 2010 conducted a new study on the lubricative performance of ethanol containing graphite for gear pump [131]. Graphite proved to be significantly resilience under operating conditions without undergoing scission and degradation. The efficient concentration of nanoparticle is reported to be 400 and 1600 ppm for this case, while power consumption was independent of this concentration. In this study, it will be shown that carbon nanomaterials increase the volumetric efficiency without significantly increasing the viscosity of the working fluid.

As previously mentioned, in Hwang et al. [75], the effect of graphite as an additive was investigated. The disc-on-disc teribotester was utilized to study the nanoparticle's size and shape effect in mineral oil tribology behaviors. The addition of graphite helped maintain sliding surfaces with lesser wears and scars (Fig. 3), mostly due to the presence of spherical nanoparticles.

Table 5 – Summary of studies conducted on lubricating properties of diamond nanoparticles.

Author	Nanoparticle	Effect
Xu et al. [123].	Diamond nanoparticle	Anti-wear, Anti-friction, excellent load-carrying capacity. (caused by surface polishing and increase in rubbing surface hardness effects of diamond nanoparticles)
Shen et al. [124].	Diamond nanoparticle	Viscosity increasing effect, Friction coefficient decrease
Chou and Lee [125]	Diamond nanoparticle	Increasing the friction and wear
Chu et al. [126]	Diamond nanoparticle	Improvement of anti-scuffing performance Significant reduction in friction

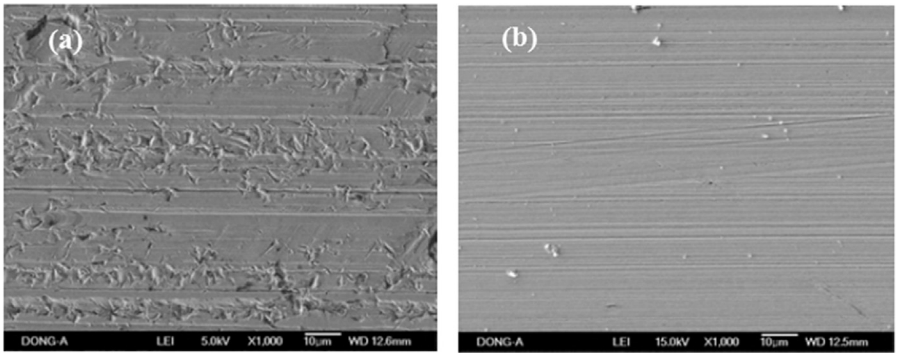


Fig. 6 – SEM images of the metal surfaces a) Pure lubricant b) Lubricant +0.5 vol % graphite nanoparticle [130].

Boron-based nanoparticles

Recently, boron-based nanoparticles have been under investigation due to its load carrying and anti-wear behaviors. It is also environmental friendly and thermally stable, making them a reliable candidate for use as a lubricant oil additive. Table 6 summarizes studies on the properties of boron-based nanoparticles as oil additives.

In 2014 [143], Zhao et al. explored nano-calcium borate (NCB) for anti-wear and load-carrying capabilities for use as lithium grease additive. They also investigate the nano-additive performance in greases. The ethanol supercritical fluid drying method was utilized for nanoscale synthesis of calcium borate. The tribological performance of calcium borate as grease additive for lubricating steel-steel contacts was assessed using an oscillating reciprocating friction and wear tester (SRV). Using an SEM, the microstructure of calcium borate particles was obtained, while the sizes of the nanoparticles were characterized using laser dynamic light scattering (LDLS). It was concluded that the coefficient of friction is dependent on the concentration of nanoparticles'. Adding NCB to lithium grease at a concentration range of 1.5–6% (with optimal concentration of 6%) reduced the friction coefficient and wear, which makes them viable for application of nanoparticles as friction-reducing agent (friction modifier). XPS analysis proved that NCB could be deposited onto surfaces to form a boundary layer of deposited nano-calcium borate, iron oxides, CaO, and B_2O_3 protect the sliding pairs from wear.

In the same year and in another work by Zhao et al. [144], the performance of zinc borate was analyzed as an additive to sunflower oil additive. Utilizing pin-on-disc and four-ball tribotesters, the anti-wear and friction reduction behavior of sunflower oil, including zinc borate as additive, were studied. By a pin-on-disc tester, the zinc borate ultrafine powder (ZBUP) was assessed for its friction reduction characteristic in sunflower oil (Teer Coatings Ltd.), with a friction force measurement sensitivity of 0.02N. The tests were conducted in a boundary lubrication regime at 22C for 30 min, at a 50 mm/s sliding speed under a 50 N load. With particle sizes of 500–800 nm, ZBUP was proven to be outstanding in terms of friction reduction and anti-wear properties. Major tribological enhancement was realized using 0.5% of ZBUP, leading to a reduction in the friction coefficient of more than 14%, and in WSDs, about 10%. This could be due to tribochemical reactions taking place on worn surfaces that hardens the substrate. These Fe, O, C, Zn, and B elements contain tribofilms with lower hardnesses than the substrate material. The employment of different lubricant samples resulted in variations in the tribofilms size and profile. The results from this study have confirmed the advantages of using ZBUP in bio-

Table 6 – Summary of publications about boron-based nanoparticles being used as lubricant oil additive.

Author	Nanoparticle	Effect
Adams et al. [132]	Potassium borate	Extreme pressure additive (for gear lubrication)
Norman et al. [133]	Calcium borate-overbased salicylate	Anti-wear
Kong et al. [134]	Nano-cerium borate	Friction modifier
Hu and Dong [24]	Titanium borate	Anti-wear
Hu et al. [135]	Ferrous borate, Magnesium borate	Anti-wear
Dong and Hu [136]	Zinc borate	Anti-wear
Hutchinson and Reid [137]	Hexagonal boron nitride	Boron nitride can withstand extremely high temperatures and high loads, it is also non-reactive, thermally conductive, electrically insulating, and is white in appearance.
Rahim & Walker [138]	Hexagonal boron nitride	
Mosleh et al. [139]	Hexagonal boron nitride	Friction modifier, Anti-wear
Lovell et al. [140]	Boric acid	Friction modifier, Anti-wear
Aravind et al. [141]	Boric acid	Friction modifier, Anti-wear
Abdullah et al. [142]	Hexagonal boron nitride	Reducing coefficient of friction and scar diameter

based lubricant in order to produce an environmentally-friendly lubricant.

Reeves et al. [145], in one recent study, investigated how the size of particulate additives influences their friction and wear performance in bio-based lubricant. Nano-sized particles were shown to offer the best tribological performance in canola oil when compared to micron-and submicron-sized particles in canola oil. Nano-sized particles in canola oil coalesce in the asperities valleys, creating a superior protective transfer film between the contacting surfaces that eschew friction and wear. The nano-sized particles' ability to improve the tribological performance was present in mixtures containing submicron-and micron-sized boron nitride particles.

Zinc dialkyl dithiophosphate (ZDDP) is an anti-wear additive for steel surfaces that are currently used in engine oils. Its anti-wear performance (against abrasion) is from the tribochemical reaction of zinc polyphosphate with abrasive metal oxides nanoparticles under the combined effect of pressure and shear. However, phosphorus and sulfur are nocuous for environmental issues. Borates are possible replacement candidates for phosphates in engine oils. Friction reduction with borates is found to be better than ZDDP, but its anti-wear efficiency is lower. In 2013, Martin et al. [146] show how chemical hardness models and computer simulation can explain these behaviors. In experimental work, the combination of two additives at similar concentrations (P:B is unity) results in an intermediate performance in the context of anti-wear effect. However, its friction coefficient is lower compared to that of ZDDP on its own. In an MD simulator, we utilized different P:B ratios, and the results are that the reaction starts when the P:B ratio exceeds unity and is almost complete, with P:B equal to (75:25). Also, we found that the friction of the mixture is intermediate between the two extremes. Overall, in spite of very simplified situations and different tribological conditions (high sliding speeds and short experimental times), the use of computer methods (particularly MD and hybrid methods with quantum chemistry) is extremely powerful in dealing with tribochemical reactions in the case phosphate and borate-containing additives on steel surfaces. In this case, the classical MD simulation shows that phosphorous can be replaced advantageously by boron if its corresponding ratio is optimized.

As previously mentioned, in order to prevent the solution from aggregation and create a stable aqueous dispersion of carbon-based nano-structures, one possible approach is to force the attachment of carbon-based nanoparticles to other polymers or molecules. Having an ultra-flat surface, the hexagonal boron nitride (h-BN) with less than 2% lattice mismatch with graphite could be a reliable candidate as an additive [147]. Its low friction is not only limited to layered structures, but it has also been proven that h-BN could display low friction behavior without sliding at the contact interface between layers even when single-layered [148]. Therefore, boron-based lubricants have been used as colloidal additives, such as h-BN, hydrogen borate, borate esters, and alkali-borates. In Cho et al. [149], h-BN nano-sheet aqueous dispersions were synthesized without using any chemical stabilizers at concentrations of 1, 0.05 and 0.01 wt%. It was finally concluded that h-BN repeated the exfoliation and were deposited on moving surfaces. Therefore, friction and wear were significantly reduced due to this deposited film. As a result of this, h-BN nano-sheets is regarded as a promising green lubricating oil additive in water.

Hexagonal boron nitride (hBN) possesses unique characteristics, making it an attractive performance-enhancing alternative to inorganic solid lubricants, such as graphite and molybdenum disulfide. The lubricating performance of hexagonal boron nitride is from the easy shearing along the basal plane of its crystalline structure. Celik et al. [150] synthesized nano hexagonal boron nitride particles by reacting boron oxide with ammonia and grinding. Different amounts of nano hBN particles were added to the engine oil. The tribological properties of AISI 4140 steel with nano hexagonal boron nitride, used as an additive in engine oil, were investigated using a ball-on-disc tribometer. The addition of the nano hexagonal boron nitride particles did not change the viscosity of the lubricants, while the addition of nano hBN to engine oil changed the coefficients of friction. The presence of sufficient nano hBN additives in oil prevents direct contact and results in decreased friction and wear.

In open air and under dry-sliding condition, boric acid (H_3BO_3) is an effective lubricant, resulting in a friction coefficient as low as 0.02. Micro size particles of boric acid were proven to effectively reduce surface roughness and the temperature of tools [151]. Moreover, the frictional behavior of oil was better when a combination of micro and sub-micro size particles of boric acid were utilized as additives. Therefore, scientists have focused their studies on the behavior of boric acid powder when the particle sizes are reduced from 500 μm to nano size regions [152]. Nano crystalline boric acid was used as a lubricant in the machining of AISI 1040 steel [153,154] for analysis purposes. Cutting forces, tool temperatures, and surface roughness were measured and used as evaluation parameters. It was observed that as the particle size of boric acid was reduced to nano levels (from 538 nm to 50 nm), the corresponding cutting forces increased. Tool temperatures also increased as the particle size decreased to nano levels. The surface roughness of the machined surface increased with reduced solid lubricant particle size. Previous studies improved the efficiency of lubricant mixtures; therefore, boric acid was mixed with canola oil, and based on the pin-on-disc experiments, the performance of this mixture proved to be quite desirable.

Similar to the case of GPFL (Green Particulate Fluid lubricant i.e. Boric acid with canola oil) assisted machining, PPFL (Petroleum based Particulate Fluid Lubricant, i.e. boric acid with SAE 40 oil) assisted machining exhibits an inverse relationship between the particle size of boric acid and the coefficient of friction. All the measured machining parameters increase with decreasing particle size of boric acid at the nano level. The effect of Canola and SAE 40 oils as carrying mediums for nano boric acid particles was then compared. The feed and main cutting force components exhibited better performance with Canola oil. Thrust force measurements were in favor of the SAE 40 oil; however, it was prominent nonetheless. Tool temperature and surface roughness measurements were low with SAE 40 oil carrying medium for all studied cases [154–162].

Result and conclusion

For the past few years, there has been a rapid change to both the content and scope of tribology. Tribology is a science that closely follows developments in physics. This field has entered the micro-and nano-scales, totally changing the scope of friction, wear, and sliding motions. In spite of several decades of study on nanoparticles and their corresponding characteristics, our grasp on their tribological behavior is far from complete. Despite the many advantages of nanoparticles as oil additives being analyzed and summarized in this communication, there are also some challenges hardwired to their applications, which could potentially form future research topics. The first and maybe the most important challenge is to prepare and maintain homogenous mixtures of nanostructure particles and oils. Strong van der Waals force between the particles causes them to aggregate in solutions. Therefore, various modification techniques should be investigated for the stabilization of nanoparticles in all groups of base oils to produce lubricants that are both physically and chemically stable. Increased viscosity of working fluids, due to the addition of high concentrations of particles, is another issue that needs to be accounted for. This phenomenon causes a high pressure drop in the system, which in turn lead to increased power consumption by the machines. Another challenge associated with nanoparticle applications is their high production cost, due to the necessity of using high-tech devices for their production. Therefore, attention should be on improving production techniques of nanoparticles, in order to make their applications more economically feasible.

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