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Effect of palm-sesame biodiesel fuels with alcoholic and nanoparticle additives on tribological characteristics of lubricating oil by four ball tribo-tester

M.A. Mujtaba [a,b,*], M.A. Kalam [a,*], H.H. Masjuki [a,c], Manzoore Elahi M. Soudagar [d], Haris Mehmood Khan [e], H. Fayaz [f], M. Farooq [b], M. Gul [a,g], Waqar Ahmed [h], Mushtaq Ahmad [i], Mamoona Munir [i], Haseeb Yaqoob [j], Olusegun D. Samuel [k], Luqman Razzaq [b]

[a] *Center for Energy Science, Department of Mechanical Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia*
[b] *Department of Mechanical Engineering, University of Engineering and Technology, New Campus, Lahore, Pakistan*
[c] *Department of Mechanical Engineering, Faculty of Engineering, IIUM, 50728 Kuala Lumpur, Malaysia*
[d] *Department of Mechanical Engineering, Glocal University, Delhi-Yamunotri Marg, SH-57, Mirzapur Pole, Saharanpur District, Uttar Pradesh 247121, India*
[e] *Department of Chemical, Polymer and Composite Materials Engineering, University of Engineering & Technology, Lahore (New Campus), Pakistan*
[f] *Modeling Evolutionary Algorithms Simulation and Artificial Intelligence, Faculty of Electrical & Electronics Engineering, Ton Duc Thang University, Ho Chi Minh City, Viet Nam*
[g] *Department of Mechanical Engineering, Faculty of Engineering and Technology, Bahauddin Zakariya University, 60000 Multan, Pakistan*
[h] *Advanced CFD lab, Department of Mechanical Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia*
[i] *Biofuel lab, Department of Plant Sciences, Quaid I Azam university Islamabad Pakistan*
[j] *Khwaja Fareed University of Engineering and Information Technology, Rahim Yar Khan, Pakistan*
[k] *Department of Mechanical Engineering Federal University of Petroleum Resources Effurun P. M. B. 1221, Delta State, Nigeria*

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Abbreviations: ASTM, American society for testing and materials; CV, Calorific value; COF, coefficient of friction; CNT, Carbon nano tubes; CPO, Crude palm oil; CO, Carbon mono oxide; CSO, Crude sesame oil; DMC, dimethyl carbonate; GC, Gas chromatography; EN, European; GO, Graphene oxide; FAME, Fatty acid methyl ester; KV, Kinematic viscosity; KOH, Potassium hydroxide; SEM, Scanning electron microscopy; TiO_2, titanium oxide; WSD, wear scar diameter; T, Temperature

* Corresponding authors at: Center for Energy Science, Department of Mechanical Engineering, University of Malaya, Kuala Lumpur 50603, Malaysia.
E-mail addresses: m.mujtaba@uet.edu.pk (M.A. Mujtaba), kalam@um.edu.my (M.A. Kalam).

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KEYWORDS

Palm-sesame biodiesel;
Tribology;
Lubricant degradation;
Alcoholic additives;
Nanoparticles;
Four ball

Abstract Dilution of engine oil with unburned fuels alters its lubricity and tribological properties. In this research paper, SAE-40 lubricating oil samples were contaminated with known percentages (5%) of fuels (diesel, palm-sesame biodiesel blend (B30), B30 + ethanol, B30 + dimethyl carbonate, B30 + carbon nanotubes and, B30 + titanium oxide). The effect of all these fuels on wear and frictional characteristics of lubricating oil was determined by using a 4-ball tribo tester and wear types on worn surfaces were analyzed by using SEM. Lubricating oil diluted with B10 (commercial diesel) showed highest COF (42.95%) with severe abrasive and adhesive wear than mineral lubricant among other fuels. Lubricating oil diluted with palm-sesame biodiesel (B30 blend) with alcoholic additives showed comparatively less COF, less wear scar diameter and polishing wear due to presence of ester molecules. Lub + B30 + Eth exhibited increment in COF value (35.81%) compared to SAE-40 mineral lubricant. While lubricating oil contaminated with B30 with nanoparticles showed least frictional characteristics with abrasive wear. Lub + B30 + TiO_2 showed least increment in COF value (13.78%) among all other contaminated fuels compared to SAE-40 mineral lubricant. It is concluded that nanoparticles in biodiesel blends (B30) helps in reducing degradation of lubricants than alcoholic fuel additives and commercial diesel.

1. Introduction

Due to the rapid decline in mineral reservoirs, global warming, and environmental concerns, more importance is being given to alternative renewable, green and sustainable biofuels (biodiesel & biolubricants) [1]. Nowadays up to 30% biodiesel with diesel fuel is used in different regions of the world without any modification in the diesel engine [2]. Recently Indonesia and Malaysia are using B20 and B10 palm-biodiesel blends successfully, while B30 implemented in Indonesia by 2020 [3,4]. Malaysia is expected to adopt B30 (30% palm biodiesel in diesel) by 2025 [5]. Biodiesel-diesel blend as a fuel improved the engine emissions except for NOx but at the same time decreased the engine performance. So various researchers used different fuel additives (like alcohols and nanoparticles) in the biodiesel-diesel blend to enhance the engine characteristics [6,7].

In metal additives, TiO_2 showed better engine performance with fewer smoke emissions [8]. While CNT non-metal additives exhibited more reduction in engine exhaust emissions and specific fuel consumption [9,10]. CNT's nanoparticles with biodiesel-diesel fuel also improved the cetane number, the calorific value of fuel and combustion efficiency [11,12,13,14,15,16].

While some researchers mixed alcohol as additives in biodiesel-diesel blends to improved diesel engine performance with a reduction in emissions [17,18,19]. Ethanol and dimethyl carbonate appeared as significant alcoholic additives to reduce engine exhaust gas emissions [20,21,22].

Some studies also evaluated the degradation of lubricating oils of CI engines fueled by biodiesel and diesel fuels. Wear and friction contribute approximately 25 percent of the world's total energy loss, Oakridge National Laboratory (USA) states [23]. A variety of investigations have been conducted into applications where nanoparticles have reinforced conventional fluids and lubricants [24,1,25]. Agarwal et al. [26] reported that lubricating oil with 20% biodiesel-fueled system contains less possible contaminants (like soot, wear debris, oxidation products, resins, moisture) and low wear than the diesel-fueled system. This was because of the self-lubricity characteristic of biodiesel. Arumugam et al. [27] examined the effect of 10% B20 rapeseed oil biodiesel on the bio lubricant (synthesized from rapeseed oil) lubricity using HFRR equipment. Results showed less coefficient of friction and wear compared to synthetic lubricant blended with 10% petroleum diesel.

Also, Atul Dhar et al. [28], diesel engine was run for 200 h on petroleum diesel and B20 Karanja biodiesel diesel to investigate the tribological characteristics of lubricating oil. Results revealed that lubricating oil contained more ash content, wear trace metals, soot and resinous during biodiesel run compared to petroleum diesel run.

As the chemical composition of biodiesel fuels (as synthesized from different sources of vegetable oils) and mineral diesel are different so their effects on lubricating oil life and its degradation will also be different as investigated by Agarwal et al. [29]. During engine operation, all the debris resulted from corrosive and metallic wear were added in lubricating oil. Hence, it is necessary to examine the effect of combustible fuels on tribology of lubricant to check their suitability for CI engines.

Few researchers conducted a study on lubricating oil degradation by biodiesel fuels like Singh et al. [30] observed that 5–8% dilution of B100 moringa methyl ester in synthetic lubricant enhances the lubricity of the lubricant but further increment in dilution resulted in high wear rate. Similar improvement in tribological characteristics of synthetic lubricant with dilution of 5% biodiesel reported by other researchers [31,32].

A 5% of contamination of lubricant with fuel occurred because of crankcase dilution during engine operation [33]. But no-one conducted research on lubricating oil degradation with diesel, diesel–biodiesel and diesel–biodiesel-fuel additive fuels to study the tribological behavior.

Now-a-days everyone is using different fuel additives (oxygenated alcohols and NPs) to enhance the CI engine characteristics [34,35]. Nanoparticles shape and structure play an important role in providing the lubrication between metallic surfaces. The shape of the nanoparticle corresponds to the pressure experienced by the nanoparticles during loading. Spherical shaped nanoparticles have a high load carrying ability due to their ball bearing effect [36]. The reason for this behavior is the contact between the nanoparticle and the lubricated surface. TiO_2 nanoparticles have sphere like shape [37] and Carbon nano tubes have shape like dahlia [38]. The spherical shape of nanoparticles results in a point of contact with the counter surface. Line contact is associated with nanosheets, while plane contact is a feature of nanoplatelets. TiO_2 nanoparticles have point contact and CNT nanoparticles have line contact according to their morphology. Spherical shaped nanoparticles are preferable to use as a fuel additive to improve tribological characteristics.

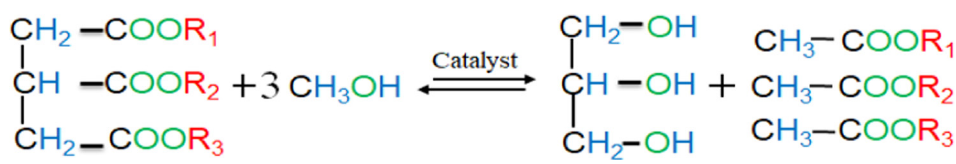
However, no-one is concerned about lubricating oil degradation during crankcase dilution due to the mixing of combustible fuel with lubricant. This study should be conducted as the engine life will be shortened due to contamination of the lubricant oil with diesel fuel (blended with biodiesel and fuel additive). In this current investigation, palm-sesame biodiesel fuels with alcoholic or nano-particle additives diluted up to 5% (by volume) of commercial lubricant to evaluate their influence on contaminated lubricant tribology. According to our best knowledge, no one conducted research to evaluate the influence of different diesel-methyl ester blends with fuel additives on the tribology of contaminated lubricant. Six different types of fuels (including commercial diesel (B10), palm-sesame biodiesel blend (B30), B30 with alcoholic (Ethanol and DMC) and nanoparticle (TiO_2 and CNT additives were selected to investigate their effects on tribological characteristics of lubricating oil by four-ball tribo-tester.

2. Materials and methods

Crude sesame oil (CSO) and crude palm oil were procured from local industry of Pakistan and Malaysia, respectively. Methanol with purity 99.9%, dehydrated Ethanol (purity: > 99.5%) and KOH pellets of Friendemann Schmidt AR grading system. Dimethyl Carbonate Extra Dry (purity: 99 + %) from Acros Organics and Nanoparticles (Carbon Nano Tubes and Titanium oxide) were obtained from Sigma-Aldrich. 12.7 mm diameter steel balls were sourced from a local market of Malaysia.

2.1. Biodiesel production

The methyl ester of palm (P50S50) was synthesized from 50% palm oil (P) 50% sesame oil (S) blend by using transesterification process on ultrasound sonication setup as shown in Fig. 1 with following operating parameters as 60 V/V % CH_3OH to P-S oil ratio, 0.70 wt% KOH catalyst, 59.52% duty cycle, 60 °C T and at a reaction time period of 38.96 min [39]. A chemical reaction between oil and methanol during the ultrasound-transesterification process is shown in **Equation 1**.



Triglyceride of P50S50 oil Methanol Glycerol P50S50 Biodiesel

The FA composition of P50S50 methyl ester was determined by GC: Agilent 7890, USA using European standard (EN 14103:2011) operating conditions as illustrated in Table 1 and the obtained results are presented in Table 2.

2.2. Characterization of nanoparticles

In this section, the morphology, particle size and other parameters of carbon nanotubes and titanium oxide nanoparticles summarized. Table 3 exhibited the specifications and properties of CNT and TiO_2 nanoparticles. Fig. 2 showed the SEM analysis of TiO_2 and CNT nanoparticles derived from the preceding literature [40].

2.3. Lubricant preparation

Commercial Malaysian diesel (B10) was sourced from local fuel pump station. B30 fuel sample was prepared by mixing 70% commercial diesel and 30% P50S50 biodiesel, B30 fuel + 20% by volume dimethyl carbonate (DMC) and B30 fuel + 10% by volume ethanol fuel samples were prepared at 900 rpm stirring speed for 20 min.

Similarly, B30 + 100 ppm carbon nanotubes and B30 + 100 ppm TiO_2 nanoparticle fuel samples were prepared at 800 rpm for 20 min on a magnetic stirrer and then sonicated for 30 min with 30% amplitude at pulse rate of 3 s on and 2 s off.

As 5% contamination of lubricant with fuel occurred because of crankcase dilution [33]. Thus, 5% of each above-mentioned fuel was blended with the SAE-40 (a commercial lubricant) utilizing the magnetic stirrer at a speed of 800 rpm for a duration of 20 min. A viscometer (Anton Paar: SVM 3000) was used to measure the physio-chemical properties of SAE-40 and all other contaminated lubricant samples as shown in Table 4. Contamination of the fuel in the lubricating oil caused its dilution which decreased its viscosity and lubricity.

2.4. Tribology of lubricant samples by four-ball tribo-tester

All the lubricant samples were tested on an automatic 4-ball tribo tester (FBT-3: DUCOM brand) to evaluate the tribological characteristics of lubricant with dilution of different fuels.

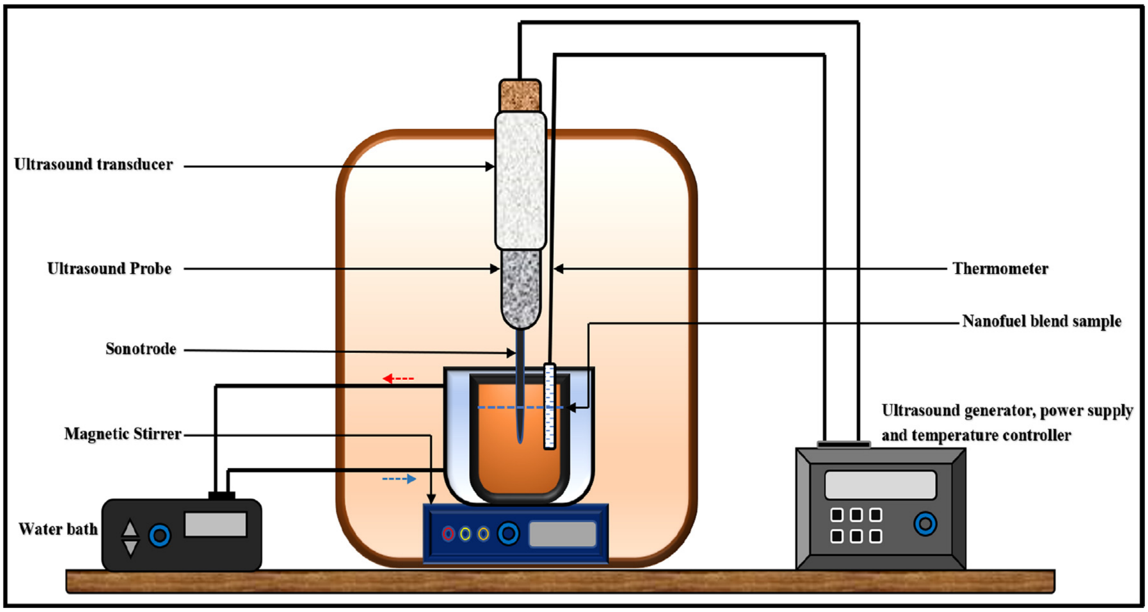


Fig. 1 Ultrasonication test rig for transesterification reaction of P50S50 blended oil [40].

Table 1 Gas chromatography operating conditions.

Operating parameters	Specifications
Column	HP-Innowax (30 m × 0.320 mm × 0.25 mm)
Carrier gas	He
Flow rate	1 ml/min
Split flow	100 ml/min
Injector T	250 °C
Flame Ionization detector	250 °C
Column T	60 °C for 2 min 10 °C/min to 200 °C 5 °C/min to 240 °C Hold 240 °C for 7 min

Table 2 FAME of P50S50 biodiesel.

FAME	Carbon structure	Palm-sesame blend biodiesel (P50S50) (w/w) %
Myristic	C14:0	0.48
Palmitic	C16:0	24.59
Stearic	C18:0	4.45
Oleic	C18:1	42.48
Linoleic	C18:2	26.92
Linolenic	C18:3	0.49
Arachidic	C20:0	0.57
Total saturated		30.09
Total unsaturated		69.89

The cup holder with three stationary steal balls containing 10 ml of lubricant samples was attached to a temperature-sensor. While the fourth steel ball was fixed in the spindle shaft that rotates on three fixed balls after applying load as shown in Fig. 3. New separate 4 steel balls have been used for each experiment. Commercial lubricant SAE-40 has also been evaluated for comparison of the results as a reference lubricant.

Table 3 Specifications and properties of TiO_2 and CNT nanoparticles.

Description	Specification	
Chemical Name	Carbon nanotubes	Titanium dioxide
Appearance	Black	White
Linear Formula	CNT	TiO_2
Nanoparticle avg. size	20–30 nm	30.5 nm
Molecular wt.	146.23	79.87

All tests were conducted using the standard ASTM D4172; its working conditions are given below in Table 5.

3. Results and discussion

3.1. The tribological characteristics of lubricant samples: COF and WSD

COF of all lubricant samples during running-in state and steady-state are presented in Fig. 4. There is a sharp spike in the COF value of all measured samples at the beginning of the run, known as the run-in period which can be visualized from Fig. 4. COF is very high because of the lack of lubricating film between these metallic contact during this run-in-period.

After a few minutes, the friction trend stabilizes because of the protective lubricating film formation among rubbing surfaces, known as steady-state condition [41]. Lubricant with biodiesel fuel dilution showed less frictional force and almost 7.7% lower COF than lubricant contaminated by commercial diesel. Biodiesel fuel dilution in lubricating oil causes less degradation as described by earlier research [27,42].

Also, the addition of NPs in methyl esters enhanced the lubricity of lubricant as compared to diesel contaminated

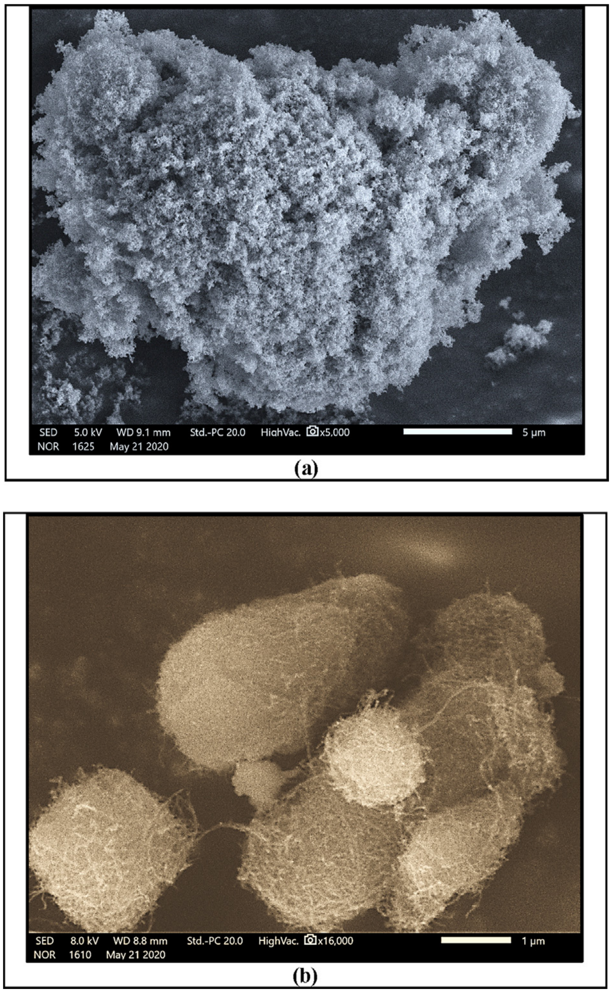


Fig. 2 SEM analysis of (a) TiO_2 and (b) CNT [40].

lubricant and showed the least COF. B30 + TiO_2 and B30 + DMC fuels caused the least degradation of lubricant among all other lubricant samples. The reason for least degradation of lubricant is presence of oxygenated moieties and excess amount of oxygen in chemical composition of DMC and TiO_2 led to an additional improvement in the overall lubricity of B30 + fuel additives-contaminated lubrication oil [27,43].

Fig. 5 showed the average COF between stationary balls and rotating ball and WSD of tested steel balls. 100% pure reference lubricant (SAE-40) showed minimum COF. But dilution of lubricant by fuels caused an increase in COF because of the decrease in viscosity. Lubricant contaminated with commercial diesel (B10) showed the highest (0.1053) COF than all other lubricant samples. B30 + DMC, B30 + nanoparticles contaminated lubricant showed considerably low COF than diesel, biodiesel, and biodiesel + ethanol fuel contaminated lubricants. On average, all contaminated lubricant fuel samples Lub + B10, Lub + B30 + Ethanol, B30, Lub with (B30 + CNT), Lub with (B30 + DMC) and Lub with (B30 + TiO_2) exhibited higher COF 42.295%, 35.81%, 31.355%, 27.56%, 27.02% and 13.78% respectively than 100% lubricant sample. Lub with (B30 + TiO_2) sample showed the least friction coefficient among all tested fuels under similar operating conditions. Lub + B30 + TiO_2 lubricant sample showed 13.4% lower COF than biodiesel (B30) contaminated lubricant and 20.04% lower COF than diesel contaminated lubricant. B30 + TiO_2 acts as a friction-reducing agent in the lubricant sample as it produces a protective coating between the metallic contact because of ester molecules in biodiesel and the spherical shape of TiO_2 NPs acting as a ball bearing between rubbing surfaces [44]. Similar results were obtained by various researchers [45,46]. Lub + B10 showed a high average COF value and lowered lubricity among all analyzed samples because of a high percentage of Sulphur content in B10 fuel. B30 + Eth fuel sample contains a large quantity of oxygen content which resulted in high wear and COF due to oxidation of the metallic surface. Many researchers have reported a similar result [47,48]. Lubricating film stability between rubbing surfaces mainly affected by speed, applied load, fluid viscosity, dispersion of nanoparticles, the fatty acid composition of fuel and temperature [31].

The WSD of reference commercial lubricant (SAE-40) was significantly high while dilution of lubricant with biodiesel fuels decreased WSD because of polarity-imparting oxygen atoms present in the methyl esters group of biodiesel fuel that improves the lubricity. The same reasons were described by other researchers [49,50]. B30 with addition of fuel additives exhibited less WSD values because of ester molecule as well as nanoparticles that act as a tiny ball bearing which roll between rubbing surfaces and resulting in lower wear compare to pure 100% lubricant and Lub + B10 [43,51]. Lub + B30 + Eth showed maximum WSD value among fuel additive blends due to oxidation products which will remove the oxides layers between metallic contact surface like piston and cylinder, which resulted in high wear and coefficient of friction. Similar behavior was reported by other researchers [52,53].

Table 4 Lubricant samples physicochemical characteristics with addition of 5% fuel blends.

Tested samples	Lubricant samples (physicochemical characteristics)			
	Viscosity @ 40 °C (mm^2/s)	Viscosity @ 100 °C (mm^2/s)	Viscosity Index	Density (Kg/m^3)
SAE 40 Lubricant	87.022	16.237	201.3	873.7
5% B10 + Lubricant	71.072	13.985	205.3	872.0
5% B30 (70% Diesel and 30% Biodiesel) + Lubricant	76.652	15.851	221.6	873.3
5% (B30 + 20% (V/V) Dimethyl carbonate) + Lubricant	64.509	–	–	872.0
5% (B30 + 10% (V/V) Ethanol) + Lubricant	69.316	–	–	871.0
5% (B30 + 100 ppm Carbon Nano Tubes) + Lubricant	69.606	15.126	230.6	872.7
5% (B30 + 100 ppm TiO_2) + Lubricant	69.311	13.705	205.4	872.1

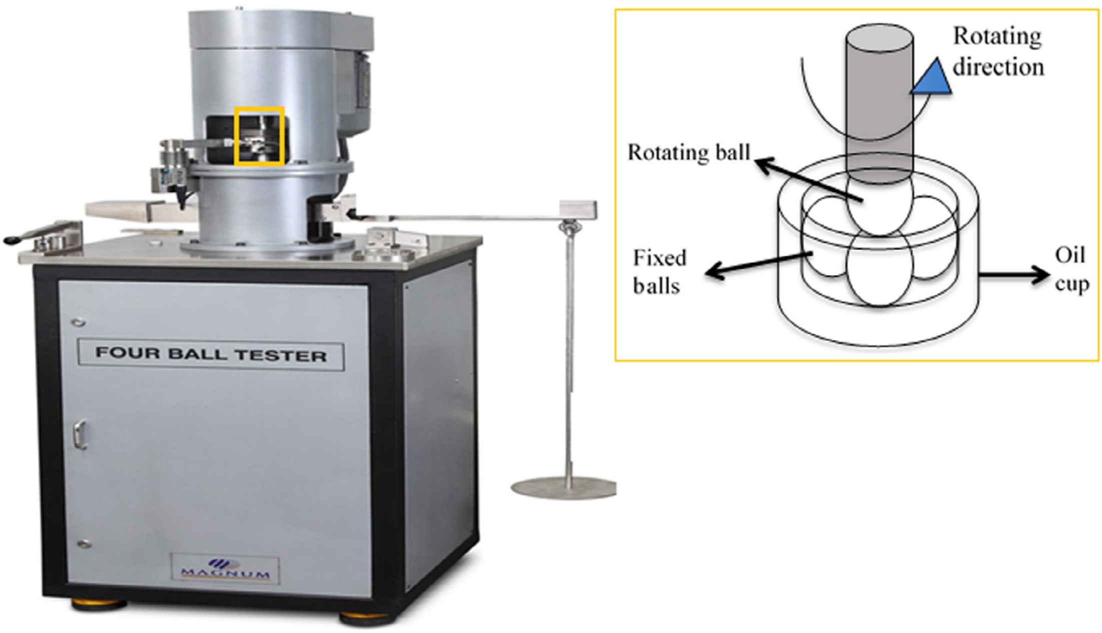


Fig. 3 4-ball tribo-tester for testing lubricant samples.

Table 5 Four-ball Tribological test operating conditions and specifications of tested ball.

Test Parameters	Operating values
Spindle rotational speed	1200 rpm
Load	40 kg
Sample temperature	75 °C
Require time	60 min
Specification of tested ball	
Hardness	62
Surface roughness	0.04 µm
Diameter of ball (size)	12.7 mm
Material	Carbon- chromium steel (SKF)

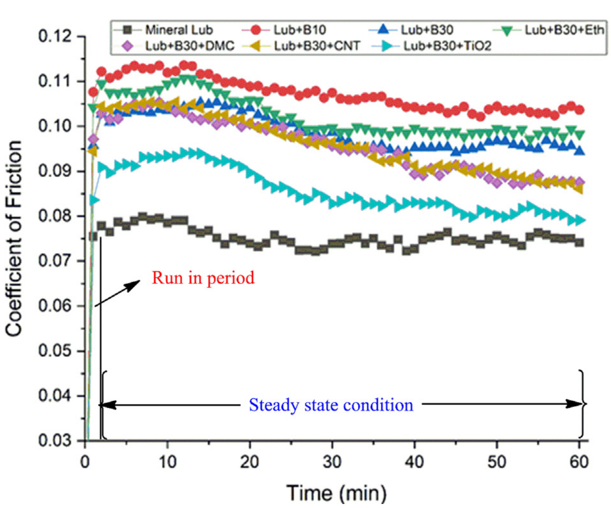


Fig. 4 Coefficient of friction trend for all tested samples with respect to time.

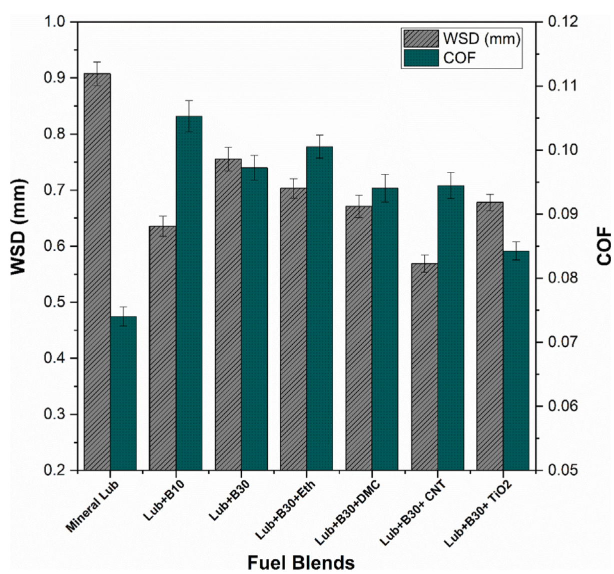


Fig. 5 Avg. COF and Avg. WSD of lubricant samples.

All contaminated lubricant fuels exhibited a decreasing trend in WSD values because of the ester molecule in a B30 biodiesel sample. Lub + B30 + Eth tested sample showed maximum COF and WSD among all contaminated lubricant with different fuel additives. Lub + B30 + Eth sample's poor lubricity is due to its lower kinematic viscosity that creates difficulty in the forming of a protective layer between metallic surfaces. Formation of anti-adhesive layer among metallic contact in case of Lub + B30 + DMC sample exhibited better lubricity compared to ethanol diluted lubricant sample. Lubricant diluted with B30 + nanoparticles exhibited less coefficient friction and wear compared to other samples.

Lub + B30 + CNT showed the lowest value of WSD among all fuel additive contaminated lubricant samples. The

COF value of Lub + B30 + CNT is high compared to Lub + B30 + TiO_2 due to the clustering of NPs in fuel samples because of poor dispersion stability of CNT nanoparticles. Few studies reported that the dispersion of CNT in the fuel sample is very difficult due to chemical inert behavior of CNT nanoparticles [54]. However, the use of surfactant in the current study stably blends the CNT in the fuel samples, which avoids the agglomeration, settling and clustering of nanoparticles in the fuel blends. Also, to stably the mix the CNT nanoparticles in the fuel blends, initially it is mixed using the magnetic stirrer, followed by bath sonication for a period of 60 mins and probe sonicated at 25 Hz ultrasonic waves at 3 s ON/OFF, which enables the mixture to stably blend. Lubricant contaminated with B30 fuel showed the least WSD value due to the long carbon chain fatty acids and high percentage of unsaturated fatty acids in bioiesel. On average, there is reduction in WSD values 31.7%, 29.8%, 26%, 25.2%, 22.4% and 16.7% compared to pure lubricant for B30 + CNT + Lub, B30 + Lub, B30 + DMC + Lub, B30 + TiO_2 + Lub, B30 + Eth + Lub and B10 + Lub respectively. The reduction in the WSD values is due to the presence of ester molecules in biodiesel and the presence of oxygen content contributing to better wear properties.

3.2. Worn ball surface analysis by SEM

Morphological images of worn steel ball used in testing the lubricant samples are shown in Fig. 6. Contamination of commercial diesel (B10) in lubricant increased its wear as abrasive and adhesive wear is observed. Material detached from the worn steel ball cavity of lubricant contaminated with B10 is bigger than 20 μm, which is named as adhesive wear. The par-

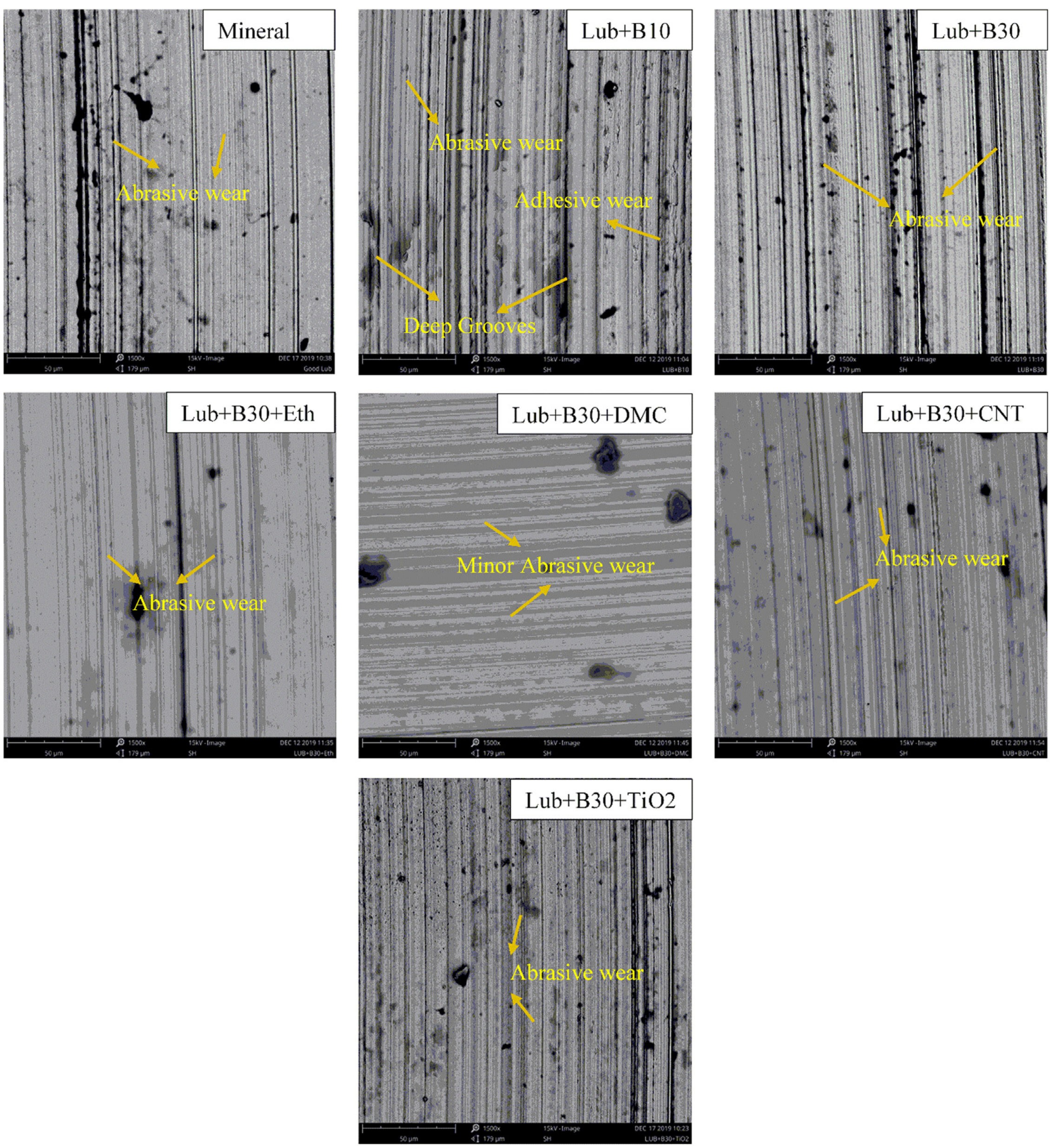


Fig. 6 Morphological images of worn steel ball by SEM.

ticles in all tested samples removed from the worn steel ball cavities are smaller than 20 μm, which is known as abrasive wear [55]. Abrasive wear in the form of scratches can be seen. Scratches and deep grooves can be observed in the case of commercial diesel (B10) contaminated lubricant. This result attributed to the unsteady friction coefficient behavior and absence of lubricating thin film between mating metallic surfaces. B30 biodiesel fuel in lubricating oil caused less wear than diesel fuel and can be seen as light and deep grooves (abrasive wear) on the worn surface. Biodiesel blend B30 showed less wear than commercial diesel (B10) because P50S50 biodiesel has a high concentration of linoleic and oleic acids that acts as lubricity enhancer [56].

Alcoholic based B30 fuel in lubricant showed the polishing wear as a result of chemical reaction of fuel on the steel ball surface to protect rubbing surfaces. It means ethanol and dimethyl carbonate are chemically active additives in the fuel that gave comparatively smooth surfaces. Dimethyl carbonate gave less wear debris in lubricating oil due to chemically active film formation between rubbing surfaces. Lubricant samples contaminated with B30 + DMC fuel showed the least degradation of lubricant as presented in Fig. 6.

Similarly, nanoparticle based B30 fuel in lubricant showed less wear than the commercial diesel contaminated lubricant. B30 + CNT showed less abrasive wear than B30 + TiO_2 fuel.

The wear and overall coefficient of friction of lubricating oil decreases with addition of nanoparticles in biodiesel blend (B30) because of lubricating film generation between rubbing surface and nanoparticles serves as a ball bearing between these surfaces [43]. Tribofilm formation is caused by the reaction between the treated material and the additives in the given atmosphere. Tribofilm is also named protective film on the tested surfaces [57,58]. In the previous study, various researchers reported filling deep groves with nanoparticles known as the mending process, which enhances wear-protection capability [51]. Nanoparticles stability and dispersion are major parameters that lead to better tribological characteristics compared to structure, size, and shape of nanoparticles. Lubricant sample contaminated with B30 + TiO2 exhibited better tribological behavior (less wear and COF) compared to B30 + CNT due to better stability and dispersion of TiO_2 in the fuel sample. Ball-bearing effect can be another reason for better tribological characteristics in the case of B30 + TiO_2 due to the spherical shape of TiO_2 nanoparticles. The concept of active lubrication mechanisms remains a subject of debate for several research studies on nanoparticle-based lubrication systems. Several mechanisms have been proposed by various researchers including ball bearing effect, protective film formation, mending effect and polishing effect [59]. These mechanisms can be divided into two major categories. The first is the immediate effect of nanoparticles, including the effect of ball bearings and the creation of protective/tribofilm. The second is the secondary effect which contributes to the improvement of the surface by the remediation/repairing effect and the polishing/smoothing effect. In current investigation, Lubricant with TiO_2 nanoparticles exhibited better tribological characteristics compared to other fuel additives. Spherical shaped TiO_2 nanoparticles act as a ball bearing between metallic contact. In previous research articles, different lubrication mechanisms of various nanoparticles reported like, ball bearing effect, mending effect and tribo film formation using XPS rubbing surface characterization technique [60,61].

4. Conclusion

In the current research work, the effect of 5% palm-sesame biodiesel fuels, with alcoholic and nano-particle additives, on tribological characteristics of lubricating oil (SAE-40) was evaluated by using four-ball tribo-tester according to the ASTM D4172 standard. The considerable reduction in the parameters, COF and WSD was observed for lubricant contaminated with palm-sesame biodiesel fuel with nanoparticles among other contaminated lubricant samples. P50S50 biodiesel fuel dilution in lubricating oil causes its less degradation.

Among alcoholic additives dimethyl carbonate (DMC) based biodiesel fuel caused less degradation of CI engine's lubricating oil. Lub + B30 + Eth showed very high COF due to a high rate of evaporation that resulting in the poor lubricating film between rubbing surfaces. While nanoparticle-based biodiesel blend (B30) caused less degradation of lubricating oil SAE-40 compared to alcoholic additives because nanoparticles act as a sacrificial layer between metallic contact. It is concluded that nanoparticles (TiO_2) additives in biodiesel appeared as competitive additives because it showed the least degradation of the lubricant.

Declaration of Competing Interest

The authors declared that there is no conflict of interest.

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