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Research paper

Tribological study on the biodiesel produced from waste cooking oil, waste cooking oil blend with *Calophyllum inophyllum* and its diesel blends on lubricant oil

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A B S T R A C T

Biodiesel or biodiesel–diesel fuel is the current fuel used to power transportation engines. Contamination on lubricating oil is a common issue due to leakage or extensive use of engines. This study explores the lubricant oil blend's friction and wear with the biodiesel derived from waste cooking oil, waste cooking oil blend with*Calophyllum inophyllum* oil, and biodiesel–diesel blend. The blending of biodiesels and biodiesel–diesel blend with lubricant oil varies from 5% to 25% of biodiesels and biodiesel–diesel with 95% to 75% of lubricating oil based on volume ratio. The test was conducted using a four-ball tribotester according to the ASTM D 4172. The result showed that blending of BWCIL75 with biodiesel–diesel has the lowest friction coefficient (0.072) among tested oil. The wear scar on the ball bearing lubricated with the blending mixture showed an acceptable diameter value. The wear morphology has shown that a worn surface with black spots provides more protection to the tested ball. The result found that fatty acid contained in the biodiesel and the low viscosity of biodiesel significantly reduced the frictional coefficient of the lubricating oil and worked as wear prevention. Mechanical efficiency of machinery component favour low coefficient of friction. This study indicated that biodiesel produced from waste cooking oil blended with *Calophyllum inophyllum* oil shows better lubricity and can be used as an additive to petroleum-based lubricant for better automotive engine performance.

1. Introduction

Biofuel is a solver to the energy crisis and environmental pollution. The continuous use of fossil fuels for power generation and transportation fuels has caused many harmful emissions and hazardous substances exposed to the atmosphere, such as carbon monoxide, carbon dioxide, nitrogen oxides (NOx), and sulphur oxide unburned hydrocarbon (Hosseini et al., 2013; Petroleum, 2014; Safaai et al., 2011). Therefore, biofuel is a high-efficiency biofuel intended to replace the deteriorating fossil fuel source entirely and reduce the detrimental impact of global warming. Moreover, it is verified to support future energy demand (Adenle et al., 2013; Fernandes et al., 2007). Biofuel is produced via renewable feedstock such as edible oil, non-edible oil and lignocellulosic biomass. The second-generation biofuel derived from biomass sources, primarily agricultural residue, forest harvesting residue and wood processing residues and non-edible components from food crops along with cultivation of non-food crops such as waste cooking oil (WCO), *Jatropha curcas*, *Calophyllum inophyllum* (CI), *Millettia pinnata, Pongamia pinnata, Ceiba pentandra* and *Miscanthus giganteus* (Karaj and Müller, 2014; Khayoon et al., 2012; Ajayebi et al., 2013; Andrade-Tacca et al., 2014).

Many developing countries have developed their biodiesel production, and the selection of feedstocks depends on the crop's market value, the sustainability of the crop, and the country's geographical location. For example, the USA produced biodiesel from soybean (Cui and Martin, 2017), while Europe used rapeseed oil and sunflower oil. In Asia, Malaysia and Indonesia use palm oil to produce biodiesel, and the Philippines use coconuts oil,

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Abbreviations

μ	Coefficient of friction
CI	*Calophyllum inophyllum*
CIME	*Calophyllum inophyllum* biodiesel
d	Mean wear scar diameter
EDX	Energy-dispersive X-ray
EG	Ethylene glycol
Fe_2O_3	Iron (III) oxides
Fe_3O_4	Iron (II, III) oxides
FTP	Flash temperature parameter
HFRR	High-frequency reciprocating rig
ISO VG	International Standards Organisation-Viscosity Grade
KOH	Potassium hydroxide
NOx	Nitrogen oxides
PB10	10% palm biodiesel with 90% diesel
PB20	20% palm biodiesel with 80% diesel
POME	Palm biodiesel
r	Distance from the centre of the contact surface on the lower balls to the axis of rotation
SEM	Scanning Electron Microscope
T	Frictional torque
TMP	Trimethylolpropane
VOCs	Volatile organic compounds
W	Applied load
WC biodiesel	Waste cooking biodiesel
WCO	Waste cooking oil
WSD	Wear scar diameter

while Bangladesh found that rubber seed oil is their potential feedstock (Morshed et al., 2011). However, the oil mentioned above were edible oil and currently, many researchers swift their interest in non-edible oil (Milano et al., 2018b; Liu et al., 2019), waste oils (Topare and Patil, 2021; Binhayeeding et al., 2020; Goh et al., 2020b), grease (Bashir et al., 2020; Tran et al., 2018), microalgae (Gozmen Sanli et al., 2020; Kale et al., 2021; Milano et al., 2016), and insect oil (Kalu-Uka et al., 2021; Manzano-Agugliaro et al., 2012; Su et al., 2019) to look for other sustainable feedstock which does not compete with edible oil in terms of global food supply, land used and agriculture prospect.

Diesel fuel used in transportation is one source of world pollution that leads to global warming and other environmental issues. Therefore, biodiesel is an alternative to diesel fuel that can reduce the dependency on fossil fuels. Biodiesels have similar fuel properties to diesel, and it is renewable, biodegradable, environmentally friendly and sustainable compared with diesel fuel (Dharma et al., 2017). However, established biodiesel using vegetable oil solitary as an alternative fuel directly competes with the food supply, which will cause an increase in the price of the vegetable oil, which will burden poor consumers in their daily consumption. Therefore, an alternative to vegetable oil, such as non-edible WCO, is suitable for biodiesel production. Annually, WCOs are produced by processing plants such as fast-food restaurants, post-processing factories, and others, ready for disposal (Cordero-Ravelo and Schallenberg-Rodriguez, 2018; Goh et al., 2020a). The WCO and gutter oil has been collected, recycled, refined, and reprocessed again as a cooking oil that received many negative comments claiming it would cause serious health hazards (Cordero-Ravelo and Schallenberg-Rodriguez, 2018; Zhang et al., 2014). Thus, devising implementation strategies to safeguard WCO management becomes of utmost importance in food safety. Therefore, WCO has become an economical source of biofuel production and can be used for power production (van Eijck et al., 2014; Baldini et al., 2014). However, WCO has low physicochemical properties that need to be improved as fuel. Therefore, the blending of WCO with enhanced biofuel from tropical biodiversity will alter the WCO properties and produce a high-performance biofuel in the transportation sector (Milano et al., 2018a) and power generation sector. However, biodiesel is not advisable to use directly on diesel engines, as biodiesel viscosity is much higher than diesel fuel (Yilmaz et al., 2017). Hence, fuel quality was the primary concern of biodiesel's usability, affecting fuel combustion performance, emission characteristics, and durability (Rao et al., 2020). Moreover, biodiesel's blending with diesel is a standard action adopted by many countries' transportation fuels and worked as a substitute energy resource for direct-inject compression ignition engines (Ma et al., 2021).

Engines that are powered by using blended biodiesel–diesel are beneficial for reducing greenhouse gas emission (Silitonga et al., 2018) and volatile organic compounds (VOCs) (Ge et al., 2018) except NOx and smoke emission (Raman et al., 2019). In addition, blended biodiesel–diesel fuels improved fuel efficiency, thermal brake efficiency, engine torque, and brake power (Dharma et al., 2017). However, continuous using the biodiesel–diesel blend might cause blended biofuel to slip into the engine compartment due to damage in bearing, cams, piston rings, cylinder liners, valve stem seal or gaskets and lastly, contaminate the lubricating oil. Commonly, lubricating oil contamination may cause increased wear in the critical components, shorten or extend the oil life span, influence the frequency of oil changes, and reduce engine compartment durability. Therefore, it is essential to understand the effect of biofuel contaminations on lubricating oil. The function of lubricating oil is to decrease the frictional force formed by moving surfaces or parts in the engine, subsequently reducing wear and power loss and eventually improving engine performance and reducing fuel consumption. Besides, engine oil works like a detergent to remove sludge or impurities and deposit and transfer them to an oil filter to prevent metal deposit adherence at engine parts.

Moreover, engine oil protected the engine against corrosion and worked as a seal to fill the gaps between piston and cylinder. Hence, engine oil properties are crucial to deciding the engine's performance. Viscosity is one of the main properties that decide the lubricating oil quality as it must maintain the lubricating film thickness. Moreover, the lubricating oil must ensure engine oil flow in the engine components at a lower temperature. Therefore, many researchers have investigated biodiesel's lubricity properties. For example, Zulkifli et al. (2013) have conducted wear prevention characteristics on palm oil-based trimethylolpropane (TMP) ester blended with lubricating oil. The wear characteristic of palm oil-based TMP ester blend with lubricating oil (1%, 3%, 5%, 7%, and 10% palm oil-based TMP ester) was tested using a four-ball machine. The result showed that 3% of TMP could carry the highest load (220 kg), while 7% of TMP could reduce up to 50% of friction (Zulkifli et al., 2013). Mosarof et al. (2016) investigate the friction and wear characteristics of *Calophyllum inophyllum* (CIME) and palm biodiesel (POME). This study blended CIME and POME with diesel fuels and tested using a four-ball machine, where they did not blend it with lubricating oil. The investigated blending ratios were 10% and 20% of CIME and POME with diesel fuel. Their study claimed that the addition of diesel fuels increased the friction coefficient of pure CIME and POME by 28.8% and 23.4%, respectively. PB10 and PB20 exhibits lower formation of oxides and lower metal composition. PB20 reduced the wear scar diameter compared with other tested fuels, implying better lubrication performance with the high lubricating film (Mosarof et al., 2016). Xiao et al. (2019) has conducted a study to observe the wear behaviour of soybean biodiesel with petrodiesel at different volume ratios 20%, 40%, 60%, and 80% of soybean biodiesel using a ball-on-disk high-frequency reciprocating rig (HFRR). They claimed that the addition of biodiesel reduces the wear scar diameter on steel–steel contact by 25% (Xiao et al., 2019). WC biodiesel with ethylene glycol (EG) to form dioleoyl ethylene glycol ester (biolubricant) and meet the specification of ISO VG68 viscosity grade (Hussein et al., 2021). Biolubricant can also derive from sesame seed oil (Ocholi et al., 2018), safflower oil (Nogales-Delgado et al., 2021), soyabean oil (Parente et al., 2021; Aguieiras et al., 2020), castor bean oil (Aguieiras et al., 2020; Diaz et al., 2017), chicken fat (Hernández-Cruz et al., 2017), *litsea cubeba* kernel (Cai et al., 2021), cottonseed oil (Gul et al., 2020), jojoba oil (Gupta et al., 2020) and others. Most studies investigated the lubricity properties and wear characteristic of biodiesel.

In contrast, relatively little information is reported regarding lubricating oil contamination with dual blend biodiesel and a ternary blend of biodiesel–diesel. Therefore, this investigation will determine the tribological characteristic of biodiesel produced from WCO, WCO-CI (dual blend), and WCO-CI-diesel (ternary blend) with commercial engine oil. The fuel characterisation (detailed physicochemical properties) is also presented in this study. The four-ball machine performed a wear test to determine the coefficient of friction and lubrication properties. The test will be done based on D 4172 to investigate the wear preventative characteristics of lubricity fluids. The wear behaviour of lubricant with various percentages of blended biofuel is then compared. Finally, the images of rubbed or worn surfaces will be investigated using Scanning Electron Microscope (SEM) and Energy-dispersive X-ray (EDX) to identify the scar morphology's element composition.

2. Materials and methods

2.1. Materials

WCO was collected from various restaurants in the food and beverage industry. The non-edible feedstocks named *Calophyllum inophyllum* (CI) oil was obtained from Kebumen, Central Java, Indonesia. The reagents used in the biodiesel production are methanol ACS Reagent (99.9%), sulphuric acid (>98.9%), orthophosphoric acid (85%), anhydrous sodium sulphate (99%), sodium hydrogen carbonate and potassium hydroxide pellets (purity: 99%), FAME MIX C_8-C_{24}, 100 mg, Methyl Nanodecanoate, C_{19}, min 99.5% purity, C_{19}, Phenolphthalein solution (1% in ethanol) was used in this study. Cleaning solvent (Acetone), rinse solvent (n-Heptane), and chrome alloy steel test ball, with a diameter of 12.7 mm (0.5 in.), Grade 25 EP (Extra Polish) with Rockwell C hardness shall be 64 to 66. SAE-40 API CF/SF lubricant oil with anti-corrosion, acid neutraliser, anti-wear, anti-foaming, soot reducer, and antioxidants properties was purchased from a petrol company in Kuala Lumpur, Malaysia. The engine oil was used as the base oil and blended with waste cooking biodiesel, W70CI30 biodiesel and W70CI30 biodiesel–diesel blend produced in this study.

2.2. Experimental set-up

2.2.1. Oil selection

The collected WCO was refined with filter paper to remove the solid impurities. The CI oil was mixed with WCO in a ratio of W70CI30 (70 (v/v)% WCO with 30 (v/v)% CI oil). The observation noted that the density, acid value, and FFA are almost linear, with a 10% increment of *Calophyllum inophyllum* oil shown in Table 1. However, the oxidation stability of the oil mixture in W90CI10 and W80CI20 are relatively low. Therefore, these two oils mixture were not recommended for further exploration. However, there is a remarkable increase in oxidation stability for both W70CI30 and W60CI40 of oil mixtures favourable for this study. From observation, it is noted that the acid value for the W70CI30 and W60CI40 is 19.75 mg KOH/g and 26.72 mg KOH/g respectively, the acid value for these both oil mixtures were high. An acid catalyst esterification can solve it. However, the W60CI40 with 26.72 mg KOH/g required a second round of esterification (pre-treatment process), increasing the cost of biodiesel production. Therefore, the W70CI30 oil mixture was chosen in this study and the mixture was esterified using a double jacketed reactor then transesterified using a monowave reactor.

Table 1
Raw oil mixture and properties of a specific ratio.

Raw oil mixture Unit	Raw oil properties Density 40 °C kg/m^3	Acid value mg KOH/g	FFA %	Oxidation h
W100	902.7	2.19	1.10	1.26
W90CI10	904.9	8.15	4.10	1.35
W80CI20	906.3	14.1	7.09	1.95
W70CI30	910.5	19.75	9.92	3.25
W60CI40	912.2	26.72	13.43	4.02
CI100	927.5	63.25	31.78	10.3

2.2.2. Experimental set-up of friction and wear testing

The new steel balls, test-lubricant cup, and chuck assemblies were cleaned thoroughly with acetone and then rinsed with toluene to remove impurities and stained lubricant oil. The equipment was then wiped with tissue to ensure they were moisture-free. Three cleaned steel balls were placed in the test-lubricant cup and then locked with the nut at 68 ± ηm with a torque wrench, as shown in Fig. 1. Another steel ball was placed into the ball chuck and mounted chuck into the chuck holder. The test fuel was poured into the cup until fully covered all three balls (approximately 10 mL). After that, the test-lubricant cup was assembled on the test apparatus to contact the fourth ball situated on the chuck holder. The experiments were conducted based on the Wear preventive characteristic of lubricating fluid (Four-ball method) according to ASTM D4172-94 (Reapproved 1999) with tests of 3600 s duration, at a temperature of 75 °C with a spindle speed of 1200 rpm and 40 kg for the load. After the test, the wear scar diameters of the three stationary balls were measured. The collected stationary balls were prepared for SEM and EDX analysis. For each set of test runs, four new steel balls were used (see Fig. 2).

2.3. Biodiesel production

An alkaline-catalysed transesterification process was used to produce WC biodiesel. A fully dissolved methoxide solution 0.4 (w/w)% of potassium hydroxide (KOH) in 50 (v/v) % of methanol was prepared. Then a mixture of WCO with methoxide solution and a magnetic stirrer was placed in a Monowave 400 reactor and ran for 8 min, with a constant stirring speed of 1500 rpm. Upon completion, the mixture was poured into a separating funnel for at least 6 h to separate the glycerol, impurities, excess methanol and methyl ester. The methyl ester, which is low density, will be at the top layer and the bottom layer with higher density was glycero–methanol solution and impurities. The by-product and glycerol can be removed entirely by gently washing with warm distilled water several times until the water is clear and separated. The washed methyl ester was further dried to remove

Fig. 1. Four ball tribo-tester cup.

moisture content using a vacuum rotary evaporator at 70 ° C for 45 min. The anhydrous sodium sulphate was added to cooled methyl ester to absorb any moisture molecule content. Finally, the filter paper was used to remove any insoluble material.

The blended W70CI30 oil mixture was degummed with 5% of diluted orthophosphoric acid (20%) in a double jacket reactor (60 °C) with a constant stirring speed of 1500 rpm for 30 min. Then the degummed oil was esterified with 70 (v/v)% of methanol and 1.5 (v/v)% of sulphuric acid in a double jacket reactor equipped with a condenser set at 15 °C to prevent losses of methanol from evaporation with a constant stirring speed of 1000 rpm, and the process took 120 min. The product from the esterification process was called esterified W70CI30 oil. For the alkaline-catalysed transesterification process, a fully dissolved methoxide solution 0.774 (w/w)% of KOH in 59.6 (v/v) % of methanol was prepared. The esterified W70CI30 was mixed with the dissolved methoxide solution then placed in a Monowave 400 reactor, where the esterified W70CI30 oil was converted into biodiesel. The details of the conversion process and parameters acquired were presented in the other paper (Milano et al., 2018a). The purification process is similar to the aforementioned method.

2.4. Lubricant and contamination sample preparation

In order to investigate the physicochemical and tribological properties of contaminated lubricant oil with WC biodiesel, W70CI30 biodiesel and W70CI30-diesel blends, 5%–25% various blends of WC biodiesel, W70CI30 biodiesel and W70CI30-diesel blend were prepared with SAE-40 API CF/SF commercial lubricant oil. SAE-40 API CF/SF lubricant oil was selected as a reference lubricant for comparison purposes. A magnetic stirrer was used to prepare the contaminated samples at 1000 rpm at room temperature for 5 h to obtain a homogeneous mixture. The samples were prepared and blended based on Table 2.

2.5. Chemical and physical properties with fuel characterisation

The physicochemical properties of waste cooking biodiesel and blended oil were obtained. The physicochemical properties such as kinematic viscosity, density, acid value, calorific value, oxidation stability, flash point, pour point, cloud point, cold filter plugging point, copper strip corrosion and Conradson carbon residue were determined according to the ASTM D6751, and EN 14214 standard then assessed with the physicochemical properties of diesel fuel. Gas chromatography (GC) was used to determine the FAME, total glycerol and glycerides, and methanol content in the biodiesel.

2.6. Test procedure

2.6.1. Frictional and wear evaluation

The coefficient of friction was calculated by the multiplication of the mean friction torque and spring constant. Thus, the friction torque on the lower balls can be expressed as:

$$T = \frac{\mu \times 3W \times r}{\sqrt{6}} \rightarrow \mu = \frac{T\sqrt{6}}{3W \times r}$$

where, μ = coefficient of friction, r = distance from the centre of the contact surface on the lower balls to the axis of rotation, which is 3.67 mm, T = frictional torque in kg-mm; W = applied load in kg. Then the wear scar diameter (WSD) was measured and analysed by DuCOM software with an installed image acquisition system.

2.6.2. Flash temperature parameter (FTP)

The flash temperature parameter is developed in terms of load, rotational speed and operating time using the response surface and design experiments. The FTP indicates the potential for lubricant film to break down. A high value of FTP indicates the high performance of the lubricant. For conditions existing in the four-ball test, the following formula is used:

$$FTP = \frac{W}{d^{1.4}}$$

where, W is load in kg and d is mean wear scar diameter in mm (see Tables 3 and 4 for the test parameters and chemical composition of the steel ball)

3. Result and discussion

3.1. Properties of the WCO, CI oil, W70CI30 blended oil, diesel, WC biodiesel, and W70CI30 biodiesel

The physicochemical properties of diesel, WC diesel, and W70CI30 biodiesel obtained in this study were presented in Table 5. The W70CI30 biodiesel has a lower kinematic viscosity at 40 °C (4.72 mm^2/s) and density at 15 °C (861.8 kg/m^3) compared with WC biodiesel (5.01 mm^2/s and 862.1 kg/m^3). Therefore, the W70CI30 has favourable lubrication characteristics. The acid value of the W70CI30 biodiesel is 0.46 mg KOH/g, which is less than the permissible limit (0.5 mg KOH/g) specified in the ASTM D6751 and EN 14214 standards but higher than WC diesel (0.13 mg KOH/g). The calorific value of the WC biodiesel and W70CI30 biodiesel is 38.77 MJ/kg and 41.35 MJ/kg, which both is slightly lower than that for diesel (45.361 MJ/kg), indicating that the energy content of the methyl ester is comparable to that of diesel. The flash point of the WC biodiesel and W70CI30 biodiesel is 154 °C and 160.5 °C, respectively, which is significantly higher than that for diesel (75.5 °C). The flash point of the methyl ester fulfils the requirements of the ASTM D6751 and EN 14214 standards, which specify that the flash point of the fuel should be within a range of 100–170 °C and more than 101 °C, respectively. The high flash point of the optimised W70CI30 methyl ester will reduce the risk of fire hazards when the fuel is exposed to an ignition source such as flame or spark.

The cold flow properties are also significantly improved for the W70CI30 biodiesel compared with WC biodiesel where the W70CI30 pour point, cloud point, and cold filter plugging point are 2, 2, and 1 °C, respectively, indicating that the methyl ester is

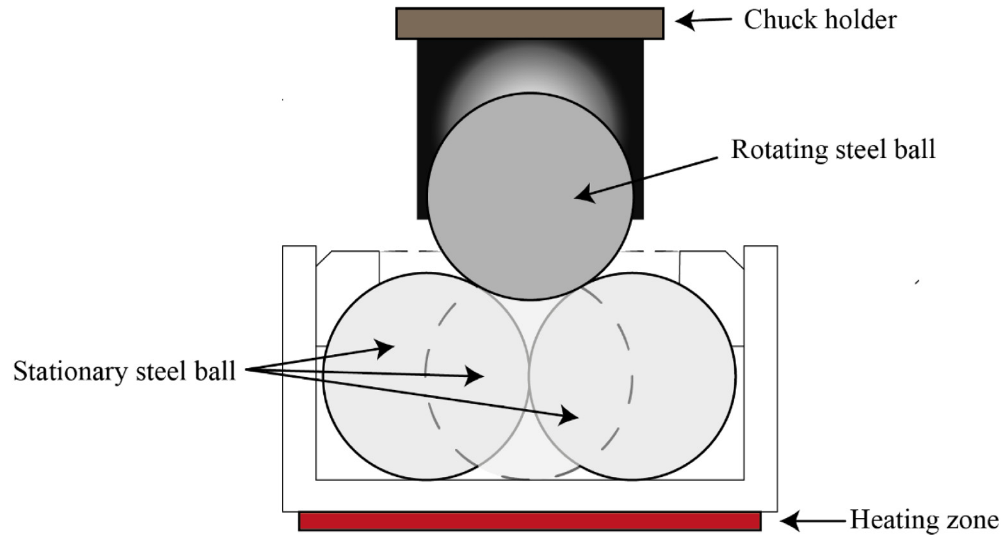

Fig. 2. Schematic diagram of the tribo-tester cup.

Table 2
Composition of the various blending ratio (v/v)% of lubricants with WC biodiesel and W70CI30 biodiesel used in this study.

No	Named	Lubricant	WC biodiesel	W70CI30 biodiesel	B25 W70CI30 blend
1	L100	100	–	–	–
2	BWCL95	95	5	–	–
3	BWCL90	90	10	–	–
4	BWCL85	85	15	–	–
5	BWCL80	80	20	–	–
6	BWCL75	75	25	–	–
7	BWCIL95	95	–	5	–
8	BWCIL90	90	–	10	–
9	BWCIL85	85	–	15	–
10	BWCIL80	80	–	20	–
11	BWCIL75	75	–	25	–
12	BWCID25	0	–	–	100
13	BWCID25L95	95	–	–	5
14	BWCID25L90	90	–	–	10
15	BWCID25L85	85	–	–	15
16	BWCID25L80	80	–	–	20
17	BWCID25L75	75	–	–	25

Table 3
Test parameter for four ball wear tests.

Test parameter	Unit	Value
Applied load	N	392
Rotation	rpm	1200
Fuel temperature	° C	75
Test duration	s	3600

Table 4
Steel ball material.

Steel ball	Unit	Description
Materials		Carbon-chromium steel (SKF)
Composition	%	10.2% C; 0.45% Si; 0.12% P; 0.07% S; 1.46% Cr; 0.42% Mn; 0.06% Ni; 2.15% Zn and 85.06% Fe
Diameter	mm	12.7
Hardness	HRc	62
Surface roughness	µm	0.1C.L.A

suitable for use in cold-climate countries. The oxidation stability at 110 °C of the WC biodiesel is 4.61 h, which is higher than the minimum value specified in the ASTM D6751 (3 h) while the oxidation stability for the WC70CI30 biodiesel is 18.03 h. However, the oxidation stability of the WC biodiesel does not fulfil the requirement of the EN 14214, which specifies a minimum oxidation stability of 6 h. The addition of *Calophyllum inophyllum* oil with WCO and have them converted to biodiesel have shown great improvement in the oxidation stability. This indicates that the oxidation stability is quadrupled by blending the two types of feedstocks, and the oxidation stabilities of the W70CI30 biodiesel are superior to that for diesel (15.2 h). Oxidation stability is an important property since it indicates the rate of fuel degradation, which plays a vital role for storage, handling, and transportation of the fuel. Fuel oxidation is highly undesirable because it degrades the physicochemical properties of the fuel where the acid value is increased, which will lead to corrosiveness of engine components, and the kinematic viscosity is increased, which will clog the fuel injectors. Based on the results, it is observed that blending waste cooking vegetable oil with *Calophyllum inophyllum* oil significantly enhances the oxidation stability of the methyl ester.

In addition, the FAME content of the WC biodiesel and W70CI30 biodiesel produced is 97.45 (w/w)% and 98.84 (w/w)%., respectively. This indicated that W70CI30 biodiesel have higher FAME content than WC biodiesel; both fulfil the requirement given in the EN 14103:2011 standard test method, where the FAME content must be greater than 90 (w/w)%. The linolenic methyl ester content should be within the range of 1–15 (w/w)%. The linolenic methyl ester content for biodiesel prepared in this study is less than the range specified in the EN 14103:2011

Table 5
Fuel characterisation of WCO, CI oil, blended WC70CI30 oil, diesel, WC biodiesel and WC70CI30 biodiesel.

Property	Unit	WCO	CI raw oil	WC70CI30 oil	Diesel	WC biodiesel	WC70CI30 biodiesel
Kinematic viscosity at 40 °C	mm^2/s	49.05	65.48	54.12	2.96	5.01	4.72
Dynamic viscosity at 40 °C	mPa s	44.27	60.73	49.27	–	–	–
Density at 40 °C	kg/m^3	902.7	927.5	910.5	–	–	–
Density at 15 °C	kg/m^3	904.4	929.2	912.2	846.1	862.1	861.8
Acid value	mg KOH/g	2.19	63.25	19.75	0.017	0.13	0.46
FFA content	(w/w)%	1.1	31.78	9.92		–	–
Heating value	MJ/kg	38.59	37.16	37.29	45.36	38.77	41.35
Oxidation stability at 110 °C	hr	1.26	10.3	3.25	15.2	4.61	18.03
Flash point	°C	–	–	–	75.5	154	160.5
Pour point	°C	–	–	–	3	12	2
Cloud point	°C	–	–	–	2	2	2
Cold filter plugging point	°C	–	–	–	0	7	1
Copper strip corrosion	–	–	–	–	1a	1a	1a
Sulphur content	ppm	–	–	–	449.65	0.89	3.32
Conradson carbon residue	(w/w) %	–	–	–	0.187	0.04	0.022
Saturated FAME	(w/w) %	–	–	–	–	39.2	39.04
Unsaturated FAME	(w/w) %	–	–	–	–	58.25	59.90
FAME content	(w/w) %	–	–	–	–	97.45	98.94
Linolenic ME content	(w/w) %	–	–	–	–	0.47	0.56
Methanol content	(w/w) %	–	–	–	–	0.05	0.03
Monoglycerides	(w/w) %	–	–	–	–	0.435	0.333
Diglycerides	(w/w) %	–	–	–	–	0.205	0.064
Triglycerides	(w/w) %	–	–	–	–	0.459	0.142
Free glycerol	(w/w) %	–	–	–	–	0.017	0.016
Total glycerol	(w/w) %	–	–	–	–	0.205	0.125

standard. The total FAME content is not 100 (w/w)% for all methyl esters because of an unidentifiable peak in the gas chromatograms due to the repeatability and reproducibility limits of the gas chromatograph. The presence of higher polyunsaturated fatty acid methyl esters is known to significantly improve the cold flow properties of the biodiesel (i.e. lower pour point, cloud point, and cold filter plugging point) of the biodiesel, and hence, it is expected that the W70CI30 biodiesel will have better cold flow properties. However, it is also known that the presence of higher polyunsaturated fatty acid methyl esters will also reduce the oxidation stability of the biodiesel. Still, it will be proved that this is not the case in the following section.

The total monoglyceride content (0.333 (w/w)%), total diglyceride content (0.064 (w/w)%), and total triglyceride content (0.142 (w/w)%) of the W70CI30 biodiesel fulfils the requirements of the EN 14214 standard. In addition, the total glycerol (0.125 (w/w)%) and free glycerol (0.016 (w/w)%) fulfils the specifications since the values are less than 0.25 (w/w)% and 0.02 (w/w)%, respectively. For WC biodiesel, the total monoglyceride content (0.435 (w/w)%), total diglyceride content (0.205 (w/w)%), and total triglyceride content (0.0.459 (w/w)%) fulfils the requirements of the EN 14214 standard. In addition, the total glycerol (0.205 (w/w)%) and free glycerol (0.017 (w/w)%) fulfils the specifications. The low values of monoglycerides, diglycerides, and triglycerides indicate that most glycerides have been successfully converted into methyl ester. Most of the glycerol has been eliminated from the methyl ester during the separation and purification processes. The properties of biodiesel blend with diesel were discussed in the following section.

3.2. Biodiesel-diesel fuel blend of W70CI30

The addition of the W70CI30 biodiesel into diesel fuel at a 25 (v/v)% ratio altered the physicochemical properties and was named BWCID25. The kinematic viscosity and dynamic viscosity at 40 °C of BWCID25 are 3.51 mm^2/s and 2.95 mPas. The density of 40 °C and 15 °C are 839.1 and 855 kg/m^3, while the acid value and heating value of BWCID25 are 0.36 mg KOH/g and 43.58 MJ/kg, respectively. Blended biodiesel with diesel shows a linear change in viscosity and density. The properties above show that BWCID25 biodiesel–diesel fuel blends to B25 are still within the limit as per ASTM D6751 and EN 14214 specifications. Therefore, the BWCID25 biodiesel–diesel fuel blends are suitable for diesel engines without modification.

3.3. Tribology study of waste cooking biodiesel, W70CI30 biodiesel, W70CI30 biodiesel–diesel (BWCID25) with the lubricant oil

The physicochemical properties study of the blending of pure biodiesel with lubricant and blended biodiesel with diesel then mixed with lubricant (base oil) was tabulated in Table 6. Table 7 shows the ISO viscosity specification for four grades of lubricant oil: ISO VG 32, ISO VG 46, ISO VG 68 and ISO VG 100. The physicochemical properties are essential to study the compatibility of biodiesel and biodiesel–diesel blends towards automotive materials. Simultaneously, the tribological study aimed to examine the wear and friction of the biodiesel and biodiesel–diesel blends. The purpose of this study is to investigate the unforeseen circumstances of biodiesel and biodiesel–diesel blend with lubricant. Then the effect of adding biodiesel and biodiesel–diesel with lubricant oil was also investigated. Besides, wear, and friction analysis were made automotive materials when biodiesel slips into the lubricant.

Viscosity index is calculated based on the difference between the viscosity at 40 °C and 100 °C. A low viscosity index means a significant difference in viscosity at the temperature range. Higher viscosity index is preferable because this indicates that viscosity does not change much at the temperature difference and is more suitable for automotive lubricants. The pure lubricant oil's viscosity index is 138.4. By adding the waste cooking biodiesel, W70CI30 biodiesel, and W70CI30 biodiesel with 25% (BWCID25) of diesel with lubricant will increase the viscosity index. High viscosity index will help promote rapid starting and provide good lubricant circulation. Blended 25% of diesel with lubricant will slightly reduce the blended mixture's viscosity index compared with biodiesel blended with lubricant. All blended fuel met the viscosity requirement of ISO VG32 for both 40 °C and 100 °C. However, BWCL75, BWCIL75, and BWCID25L75 do not meet the minimum requirement of 41.4 mm^2/s stated by ISO VG46 (40 °C). In contrast, only B5 and B10 of the biodiesel and biodiesel–diesel blend with lubricating oil can meet the standard stipulated in ISO VG68 (40 °C).

Table 6
Kinematic viscosity, dynamic viscosity, and density of various blending ratios of lubricants with waste cooking biodiesel, W70CI30 biodiesel, and BWCID25 biodiesel–diesel blend used in this study.

Name	Kinematic viscosity (mm²/s)			Dynamic viscosity (mPa s)		Density (kg/m³)
	40 °C	100 °C	VI	40 °C	100 °C	15 °C
L100	92.083	12.937	138.4	78.506	10.536	868.5
BWCL95	75.778	11.878	152	64.65	9.6766	869.2
BWCL90	62.147	10.403	156.5	53.034	8.4754	869.5
BWCL85	51.213	9.2685	165.4	43.73	7.5512	870.2
BWCL80	42.443	8.2229	172.3	36.258	6.6998	870.7
BWCL75	34.093	7.2259	183.5	29.134	5.8862	871.2
BWCIL95	76.338	11.780	148.7	65.103	9.5931	868.9
BWCIL90	62.256	10.485	158.1	53.133	8.5414	869.6
BWCIL85	51.327	9.2679	164.9	43.826	7.5499	870.2
BWCIL80	42.820	8.2421	171.0	36.579	6.7147	870.7
BWCIL75	35.983	7.4190	178.8	30.753	6.0454	871.3
BWCID25L0	3.3384	1.3387	167.4	2.7893	1.0607	853.5
BWCID25L95	76.154	11.753	148.6	64.864	9.5622	867.6
BWCID25L90	62.279	10.400	156	53.004	8.449	867.2
BWCID25L85	50.657	9.1381	163.9	43.068	7.4135	866.4
BWCID25L80	42.238	8.0852	168.2	35.878	6.5505	865.8
BWCID25L75	33.985	7.0463	175.7	28.833	5.6995	864.9

Table 7
ISO specification for four different viscosity grades (Hussein et al., 2021).

ISO Standard for lubricating oils	Kinematic viscosity (mm²/s)		
	At 40	At 100	VI
ISO VG32	>28.8	>4.1	>90
ISO VG46	>41.4	>4.1	>90
ISO VG68	>61.4	>4.1	>198
ISO VG100	>90	>4.1	>216

Generally, the density of lubricant which derived from hydrocarbons varies from 860.0 to 980.0 kg/m^3. The minimum and maximum density of blended biodiesel-lubricant and biodiesel–diesel-lubricant were 864.9 and 871.3, respectively. Addition of biodiesel have cause increment in density due to the structure and composition of biodiesel. It can be observed that addition of diesel will slightly reduce density of the blended fuel. To conclude, adding diesel in the lubricant will not be favourable in viscosity and density, but further investigation, such as the wear preventing test, will show a more comprehensive overview of these effects.

3.4. Anti-wear condition

The wear preventive tests have been conducted using the WC biodiesel, W70CI30 biodiesel, and B25W70CI30 biodiesel blend with lubricant at various ratios, according to Table 6. The wear preventive tests were conducted at a constant load of 392 N (40 kg) at an operating temperature of 75 °C with 1200 rpm rotational speed for 1 h. Every experiment was conducted in thrice to obtain an average value of coefficient of friction (CoF) for its stability, and the WSD of the specimen is measured. The WSD and CoF of the WC biodiesel, W70CI30 biodiesel, and B25W70CI30 biodiesel blend with a lubricant are plotted and shown in Fig. 3.

It can be observed that there is a significant improvement in CoF for all the blended mixtures. With all blended biodiesel or diesel and lubricants at 5:95, the WC biodiesel has the lowest CoF (0.082) among the three mixtures. As the lubricant concentration decreased from L95 to L75, it can be observed that there was a significant improvement in CoF of all mixtures. At the blending of L75, the CoF of the W70CI30 biodiesel is lowest (0.072), followed by WC biodiesel (0.075) then lastly, W70CI30 biodiesel–diesel blend (0.080). The WSD obtained for WC biodiesel, W70CI30 biodiesel, and B25W70CI30 biodiesel blend with lubricant at L95 is 480.44 μm, 405.92 μm, and 362.19 μm, respectively. It can be observed that after the addition of biodiesel and biodiesel–diesel content by stages from L95 to L75, the WSD reduces.

The observation shows that adding biodiesel or biodiesel–diesel blend in lubricant have improved the lubricity and reduced the WSD; this indicated that biodiesel or biodiesel–diesel blend could be used as a wear reducer. The results showed that WC biodiesel is an excellent lubricity enhancer, approximately 22% and 40% more than W70CI30 biodiesel and B25W70CI30 biodiesel blend with lubricant ratio L80 and L75. However, WC biodiesel is not favourable when the blending ratio is relatively little such as L95 till L85, as it causes more wear than other blended lubricants. Biodiesel possesses better lubricity properties than other blends at a lower blend ratio. Issariyakul et al. found that methyl ester has the best lubricity characteristics among other ester groups such as ethyl, propyl and butyl ester due to $COOCH_3$ (Issariyakul et al., 2011). Sarin et al. claimed that the free fatty acids and glycerol contents also enhance the lubricity properties (Sarin et al., 2007). With the biodiesel's nature, these properties, which is polarity, helps to provide lubricity to the metal surface of the steel balls.

3.5. Worn surface characteristics

Fig. 4 shows the SEM micrographs of the wear scar of the stationary steel ball under 40 kg load at 75 °C for 1 h of WC biodiesel, WC70CI30 biodiesel, and B25WC70CI30 biodiesel–diesel blend with L95 lubricant which are BWCL95, BWCIL95, and BWCID25L95, respectively. Fig. 5 shows the SEM micrographs of the stationary ball's wear scar at the same working conditions as mentioned earlier for WC biodiesel, WC70CI30 biodiesel and B25WC70CI30 biodiesel–diesel blend with L75 lubricant, which are BWCL75, BWCIL75, and BWCID25L75, respectively.

As seen in Fig. 4, the scar diameter is the largest (480 μm), and the wear is significant for WC biodiesel blend with lubricant (BWCL95). The W70CI30 biodiesel blend with lubricant (BWCIL95) shows a scar diameter of (406 μm), but the scratch is minimal. Fig. 4 (c) shows a minor scar diameter (362 μm) for BWCID25L95 with deeper wear compared to Fig. 4 (b). As the lubricant concentration is reduced from L95 to L75, the scar diameter decreases. The average scar diameter for BWCL75, BWCIL75 and BWCID25L75 is 274 μm, 339 μm and 301 μm, respectively. The SEM micrograph shown in Fig. 5 shows that the adhesive wear is uniform on the WSD, as shown in Fig. 5 (a) and (c). Fig. 5(b) shows a slightly lower spot of adhesive wear. More black spots have been spotted on the 75% lubricant mixed with biodiesel and diesel–biodiesel blend than the 95% lubricant and

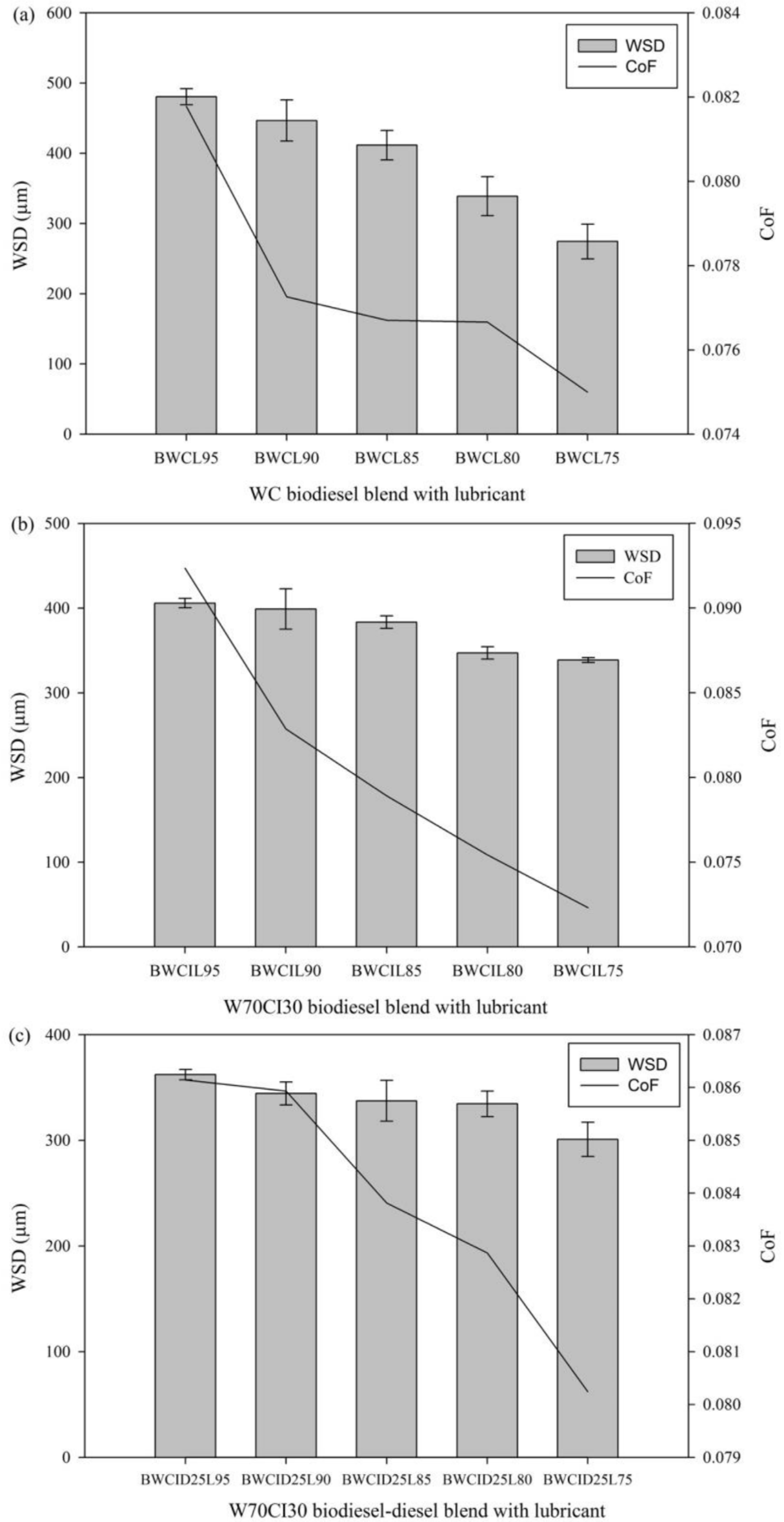

Fig. 3. WSD and CoF for (a) WC biodiesel, (b) W70CI30 biodiesel, (c) B25W70CI30 biodiesel blend with lubricant (Conditions: Load: 392 N, Temperature: 75 °C, Duration: 1 h).

5% other biodiesel–diesel blends. These black spots are formed during the sliding process when the tested balls' elements are oxidised to create a protective film on the surface of the tested balls. Black spots with groove lines on the ball showed smaller wear scar diameter than balls with less black spots. The flash temperature parameter (FTP) is inverse to WSD; the lowest scar diameter will have the highest FTP, BWCL75, BWCID25L75, and BWCIL75 in between 180 to 250. Higher FTP indicated higher stability of the lubricating oil in higher temperatures and are favourable for the lubricity characteristic (Thangarasu et al., 2019).

The wear scar diameter found in this study is the smallest compared to the scar diameter found in S. Syahrullail et al. study (Syahrullail et al., 2013). This study showed the effect of biodiesel and biodiesel–diesel on the lubricating oil, indicating that adding biodiesel or biodiesel–diesel blend reduces the scar diameter, which means it will not cause immense damage to the moving component in the engine. The biodiesel's lubricity further enhances the character of the lubricating oil. Moreover, fatty acid's polar nature provides higher strength lubrication film that will interact strongly with a metallic surface. This film offers a protective layer to the metallic surface by reducing friction and wear (Nik et al., 2007). S. Arumugam et al. performed an engine performance test with B20 rapeseed biodiesel mixed with lubricating oil as engine oil. They found lesser frictional losses at high loads and a high compression ratio. Besides, adding biodiesel into lubricating oil did not cause a further increase in CO emission (Arumugam et al., 2014).

Hence, slipping of partial fuel made from biodiesel or biodiesel–diesel blend will not cause severe damage to the engine compartment such as piston, cylinder liners, gears, crankshaft, which is similar to our tested conditions. Besides, Dhar and Agarwal investigated the effect of *Karanja* biodiesel on lubricating oil's tribological properties. They found a trace of metals in the lubricating oil after 100 h of endurance test due to the oxidation of lubricating oil with biodiesel blend (Dhar and Agarwal, 2014). However, the quality of the lubricating oils in the engine compartment is not only dependent on the possibility of slight fuel entering the other moving compartment of the engine, but also depend on the oxidation and combustion of the lubricating oil that resulted due to the formation of insoluble deposits on the metal surfaces. These insoluble deposits are causing insufficient lubrication and increasing wear.

3.6. SEM/EDX analysis (Elemental analysis)

Details of the six EDX spectra of measured elements in atomic and weight percentages are listed in Table 8. EDX was performed in the corresponding area shown in Figs. 4 and 5 to study the targeted area's elemental analysis or chemical characterisation. The tested SKF balls are a combination of transition metals such as iron, chromium, and nickel. The wear scar diameters for the 5% blended fuels with 95% lubricant in volume percent is much larger than 25% blended fuels with 75% lubricants. However, based on the worn surface morphology for the lower biodiesel blend, the wear shown is minimum with a light groove on the surfaces. This is due to the reaction film that acts as a mechanical protective barrier during the sliding process (Hutchings, 1992). This protective film will act as a cushion to prevent the contact between metals or metal oxides that reduce the contact and stress caused by metal substrate (Spikes, 2004). While the wear diameter caused by higher biodiesel and diesel-biodiesel concentration is smaller, it can be seen from Fig. 5 that there is a darker region with grooves being found on the surface of the tested ball. These darker regions can be referred to as oxidation scars. EDX results show that oxidation film has been generated to provide a protective film on the sliding surface. During the continuous sliding process between tested balls, the wear forms where metal particles are exposed to the lubricant. Wear that happens to the surface will encourage anodic corrosion that forms a protective oxidised passive surface film (Revie, 2008).

Generalisation, the oxidation film that formed during the sliding process has resulted from the elements of the four-ball and surrounding elements from the tested fuels, which it produces iron (III) oxides (Fe_2O_3) and iron (II, III) oxides (Fe_3O_4) (Zulkifli et al., 2013). These two oxides are mainly produced due to a large amount of iron present in the tested balls. The heat increase evolved the thermal motion of surrounding iron molecules has caused spontaneous oxidation of iron to iron (III) oxides, and the equation is shown in Eq. (1). Iron (II) oxides tend to be disproportionate to iron and iron (II,III) oxides, and the equation is shown in Eq. (2). Iron (II,III) oxide is visually black in colour and named hematite. Besides, other oxides produced and worked as anti-wear are Zn, P, S and Ca that bind with methyl ester and form other oxides that will protect the surface of the four-ball. Oxides formed during the sliding process have generated a protective film on the ball's surfaces and work as anti-wear (Lu et al., 2005). Generally, the lower concentration of biodiesel and diesel-biodiesel blends in the lubricant will cause larger wear than a higher concentration of biodiesel and diesel-biodiesel blends. In addition, biodiesel and diesel-biodiesel blends have reduced wear caused by commercial lubricant, which is favourable for a lubricant and these blends can function as wear reducer.

BWCIL75 showed a higher amount of carbon element in terms of atomic and weight percentages with 22.99% and 28.04%, respectively than other blended fuels used in this study. Then follow by BWCID25L75 with 19.18% and 5.63% for atomic and weight percentages. While the highest amount of oxygen element can be found in BW95CL (atomic = 36.83% and weight = 16.23%) and follow by BWCIL75 (atomic = 35.83% and weight = 14.36%). Both tested balls show a very comparative amount of oxygen element. A high amount of oxygen element means more oxides form in the specific position on tested balls. Considerable details have been explained in the above paragraph. The study shows that the more concentration of WCO-CI in fuel will promote more oxides being formed on the ball's surface. As evidenced by the wear size, it has positive potential in becoming wear preventive material.

Spontaneous oxidation of iron to iron (III) oxides

$$4Fe\ (s) + 3O_2\ (g) \rightarrow 2Fe_2O_3\ \ (s) \tag{1}$$

Iron (II) oxides is not stable and tends to disproportionate to iron and iron (II,III) oxides:

$$4FeO\ (s) \rightarrow Fe(s) + Fe_3O_4\ \ (s) \tag{2}$$

4. Conclusion

Based on the lubricating oil blend's tribological investigations, biodiesel and biodiesel–diesel. The possibility of the fuel being slipped into the engine compartment after extended usage will not cause enormous damage to the engine compartment due to biodiesel's lubricity, resulting in lower wear of the vital moving components. The addition of biodiesel and the biodiesel–diesel blend has reduced the lubricating oil's viscosity. The lubricating oil's viscosity reduces due to fuel dilution found in the biodiesel–diesel mixture. The scar wear diameter has shown that as biodiesel's ratio increases in the blend, the wear scar diameter decreases gradually. The biodiesel–diesel–lubricant combination shows a much lower scar diameter than the biodiesel-lubricant blends at a higher lubricant blend. The addition of WC biodiesel, W70CI30 biodiesel, and W70CI30 biodiesel–diesel blend with lubricant has significantly improved CoF and reduction in WSD. WC biodiesel with lubricant at 25:75 ratio showed a significant decrease in WSD, followed by W70CI30 biodiesel–diesel blend then W70CI30 biodiesel. Adding biodiesel and diesel in lubricant has shown a reduction in WSD and CoF, which are favourable in the lubricant industry and provide insight into the consumer's confidence in using biodiesel or biodiesel–diesel blend fuels to replace diesel.

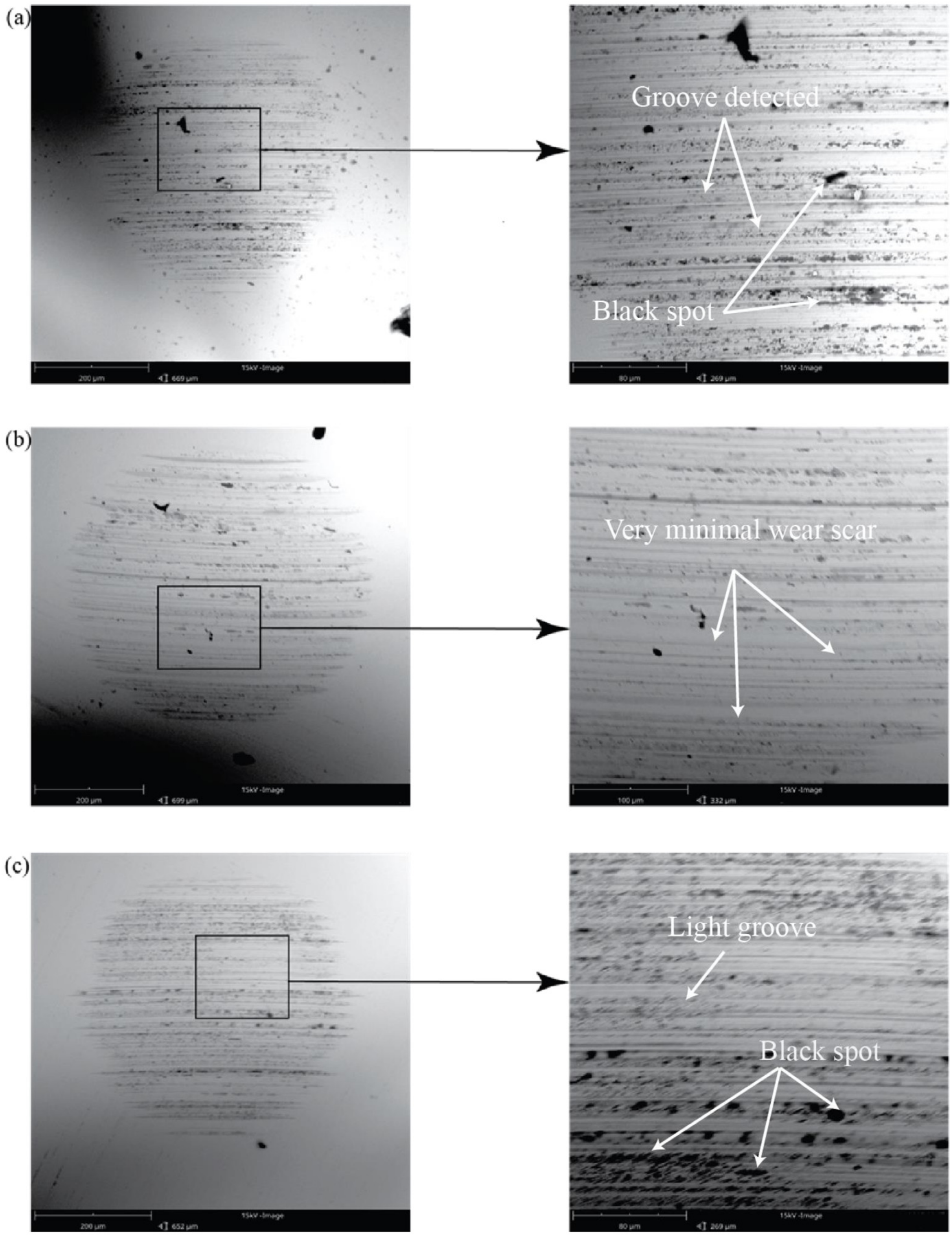

Fig. 4. SEM micrographs of the worn surfaces of the stationary ball (Conditions: Load: 392 N, Temperature: 75 °C, Duration: 1 h) (a) BWCL95 (b) BWCIL95 (c) BWCID25L95.

Table 8
EDX atomic and weight percent of elements on the four-ball using the spectrum focus on the distinct area.

Fuel	Iron (FE)		Oxygen (O)		Carbon (C)		Chromium (Cr)	
	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)
BW95CL	40.29	61.97	36.83	16.23	8.84	2.92	14.04	18.88
BWCIL95	64.01	81.31	19.64	7.15	13.07	3.57	3.28	7.97
BWCID25L95	78.27	89.83	16.46	5.41	4.47	3.9	0.8	0.86
BW75CL	77.71	90.4	17.61	5.87	3.13	5.18	1.55	1.68
BWCIL75	41.18	57.60	35.83	14.36	22.99	28.04	0.00	0.00
BWCID25L75	63.3	86.42	16.28	6.37	19.18	5.63	1.24	1.58

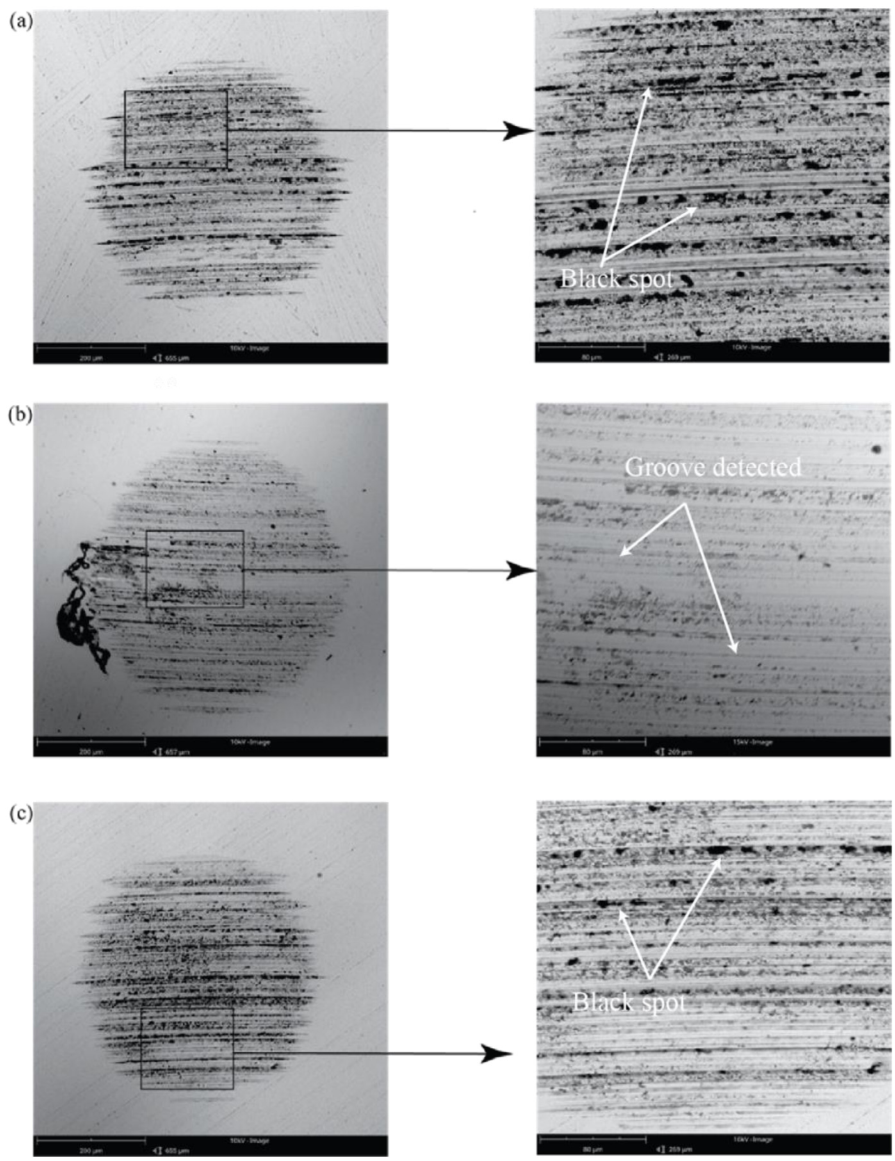

Fig. 5. SEM micrographs of the worn surfaces of the stationary ball (Conditions: Load: 392 N, Temperature: 75 °C, Duration: 1 h) (a) BWCL75 (b) BWCIL75 (c) BWCID25L75.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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