Tribological Study of Biolubricant Made from Blend of Waste Cooking Oil and *Calophyllum Inophyllum* Oil

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Abstract: Biodiesel is an alternative and renewable source of fuel that exhibits sustainable properties. It is eco-friendly as its combustion does not lead to the emission of greenhouse gases, which is associated with conventional diesel fuel. This study examined the tribological characteristics of biodiesel made from cooking oil waste and *Calophyllum inophyllum oil.* An untreated cooking oil waste collected from fast-food chains and household were used as feedstock to produce biodiesel via transesterification reaction. The biodiesel was produced at room temperature using the *Calophyllum inophyllum* plant as feedstock, which contained a significant amount of non-edible oil. This led to the formation of biolubricant at different blending ratios with the percentages of biodiesel and biofuel in the range of (5% to 25%) and (95% to 75%), respectively. The tribological investigation was conducted using a 4-ball tribotester following the ASTM D 4172 standard. The result showed that the wear scar and composition of the biolubricant are similar to the actual bearings. Finally, the B75 combinations indicated the best tribological characteristics, therefore, its usage is recommended for the protection of engine parts.

1 INTRODUCTION

The utilization of biodiesel in an internal combustion engine is becoming popular in recent years. This is because they are derived from wastes and renewable sources such as vegetable oils, animal fats, and residual cooking oils. Furthemore biodiesel can be utilized in compression ignition engines to replace fossil fuels for power and energy generation (Zahan and Kano, 2018).

The high cost of biodiesel production is related to the raw materials ranging from 60 to 80% (Zahan and Kano, 2018). Several studies analysed the use of non-edible oils as raw materials derived from different sources, such as vegetables, *Calophyllum inophyllum*, *jatropha,* grease, and cooking wastes, to minimize the use of food crops. These sources of oil have the ability to reduce the production costs by approximately 60-90%. Furthermore, the utilization of these non-edible oil sources serves as waste recycling and management techniques and mitigation to environmental pollution associated with the oil sources.

The major limitation to biodiesel production from non-edible oil is the availability of raw material in large quantities. This limitation can be addressed by blending non-edible with edible oils obtained from esters, which tends to reduce the use of food sources for biodiesel production. The production of biodiesel from blends of edible and non-edible oils, such as *Jatropha curcas, Calophyllum inophyllum,*

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Pongamia pinnata, Ceiba pentandra and waste cooking oils have been investigated (Silitonga et al., 2016). Furthermore, the use of residues from fast food centers and restaurants tends to increase urban biodiesel generation. This also has excellent environmental benefits because it promotes a green atmosphere.

According to Yilmaz et al. (2017) rising demand for petro fuel does not only lead to increase in prices, rather it creates adverse effects on the environment such as biodiversity loss, climate change, and global warming . Therefore, biodiesel is an ideal alternative to reduce the dependency on fossil fuels and it is produced from animal fats, vegetables, and recycled or waste cooking oils. Furthermore, biodiesel is a renewable, non-explosive, biodegradable, non-flammable and non-toxic fuel and has similar combustion characteristics with diesel fuel (Dharma et al., 2017).

Nowadays, non-edible feedstocks are prioritized over edible one due to the shortage of vegetable oil consumed in the nutritional diet as an energy source. Besides, its usage for biodiesel production causes food insecurity across the globe (Cordero-Ravelo and Schallenberg-Rodriguez, 2018).

Furthermore, waste cooking oils not suitable for edible purposes serves as a substitute. This possibly leads to a decrease in biodiesel cost, thereby making it an economically viable option (Cordero-Ravelo and Schallenberg-Rodriguez, 2018). Therefore, waste cooking oil is an economical source of biofuel production used for power generation (Khan et al., 2021). However, a major problem emerged when there was an increase in the market price of these vegetable oils. This led to an increase in biodiesel production costs, thereby making it uneconomical.

Silitonga et al. (2020) stated that the problem associated with biodiesel production can be overcome using less expensive materials such as vegetable and non-edible oils as alternatives. Previous studies carried out in 1990 were centred on the use of raw vegetable oils in pure form or partially blended with appreciable success in engine performance. These studies, showed that clogging and cooking the injectors after prolonged usage equally remained a serious challenge. Therefore, conscious efforts have been made to produce this fuel from various feedstocks compatible with the diesel engine by refining the oil (Adekunle et al., 2020). Ma et al. (2021) stated that the propensity of the resultant biodiesel for carbon deposition, is significantly reduced in engines.

According to Silitonga et al. (2018) the use of biodiesel in diesel engines reduces life cycle emissions of volatile organic compounds (VOCs) except NO_x. In addition, a combination of biodiesel with diesel fuel reduces environmental impact, improves brake power and thermal efficiency, as well as engine torque (Dharma et al., 2017).

Biodiesel is similar to the conventional fossil diesel, produced from an increasingly diverse mix of waste oil. However, the continous use of the biodiesel-diesel blend causes the biofuel to slip into the engine compartment, thereby causing damages to cams, piston rings, cylinder liners, valve stem seal, gaskets and contaminate the lubricating oil (Ge et al., 2018). These damages increases wear in critical components, thereby shortening their life span and engine durability. Therefore, the frequent changing of engine oil is one of the essential maintenance tasks. Besides, regularly scheduled oil changes help keep the engine clean and avoid the enterance of impurities (Arumugam et al., 2014).

Engine oil is an important aspect that supports engine performance and works as a seal to fill the gaps between the piston and cylinder. Hence, it is important because it acts as a lubricant, coolant and silencer in machines to reduce wear due to friction and high temperatures. Engine oil needs to be highly viscous to protect the inner parts of an engine from rust or corrosion, which is extremely relevant to maintain its performance. Furthermore, it ensures all the components of an engine flow at a lower temperature. Several studies have investigated the oil lubricant properties of biodiesel.

For instance, Zulkifli et al. (2013), carried out a research, which investigated the wear prevention characteristics of engines using palm oil-based trimethylolpropane (TMP) and ester blended with lubricating oil. The result showed that 3% of TMP was able to withstand the maximum load (220 kg), while 7% approximately reduced it by 50% . Mosarof et al. (2016) carried out similar research on friction and wear characteristics using *Calophyllum inophyllum* (CIME 10%) and palm biodiesel (POME 20%). The research showed that the addition of diesel fuels increased the frictional coefficient of pure CIME and POME by 28.8% and 23.4%, respectively. In addition, the PB20 compared to other tested fuels, reduced the wear scar, thereby implying better lubrication performance. These perliminary studies clearly indicate that engine oil manufactured from non-edible oil is suitable for lubricating engines.

This study produced biodiesel from biosources, such as waste cooking and commercial engine oil blended with *Calophyllum inophyllum*. The 4 Ball EP test machine was used to investigate the wear test to determine the frictional coefficient and lubrication properties. This test was carried out based on D 4172 to investigate the wear preventative characteristics of lubricity fluids. The effect of these fuels on wear and frictional characteristics of lubricating oil was also compared.

2 MATERIALS AND METHODS

2.1 Materials

The waste cooking and *Calophyllum inophyllum* (CI) oils used to carry out this research were obtained from the local market and supplied by Koperasi Jarak Lestari, Kebumen, Central Java, Indonesia. Furthermore, the chemicals and materials, such as methanol (99.9% purity) and filter paper used were of analytical grade. Furthermore, a chrome alloy steel test ball, with a diameter of 12.7 mm (0.5 in.) and grade 25 EP with Rockwell C hardness relatively 64 to 66, was purchased from SKF Malaysia Sdn Bhd. Castrol Magnatec part synthetic 10W-40 lubricant oil blended with waste cooking biodiesel, produced with a hot plate magnetic stirrer reactor.

2.2 Experimental Set-up

During the experimental set-up, the manufactured biodiesel had high viscosity and density due to the presence of excess fatty acids in its molecular structure. These fatty acids were converted into monoalkyl esters by mixing one mole of triglyceride with ethanol. Furthermore, the lipids obtained was converted into ester when it reacted with methanol in the presence of an acid catalyst. Moreover, the collected waste cooking oil was purified with filter paper to remove the solid impurities. *Calophyllum inophyllum* and waste cooking oils were mixed in the ratio of WC70CI30 (70 (v/v)% and 30 (v/v)%.

In addition, 3 cleaned steel balls were placed in the test-lubricant cup and then locked with a nut at 68 ± ηm using a torque wrench shown in Figure 1. Subsequently, the Schematic diagram of the tribo-tester cup is shown in Figure 2. The experiments were carried out based on the wear preventive characteristics of lubricating fluid (4-ball method) according to ASTM D4172-94 with tests that lasted for 1-hour, at a temperature of 75 ºC, with a spindle speed of 1200 rpm and a 40 kg load.

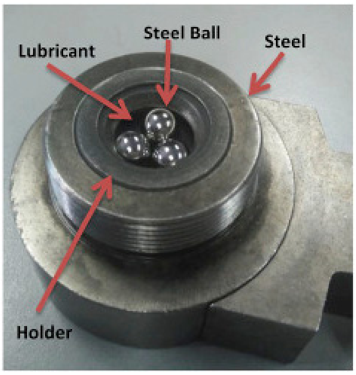

Figure 1: Four ball tribo-tester cup.

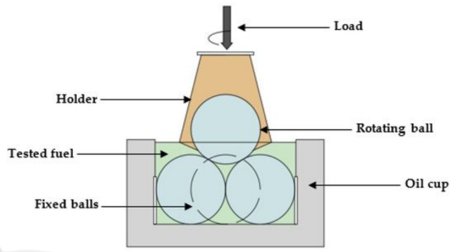

Figure 2: Schematic diagram of the tribo-tester cup.

2.3 Biodiesel Production

The conversion efficiency of biodiesel production depends on the molar ratio of alcohol to oil. The ratio of methanol to 60% oil and 0.5 wt.% of H_2SO_4, was made to react at 60° C for 1 hour. The crude oil is highly acidic, therefore the esterification process using a hot plate magnetic stirrer is required. In addition, a completely dissolved methoxide solution was obtained by dissolving 0.5 wt.% of potassium hydroxide (KOH) in 60% of methanol and the mixture was further added to the preheated waste cooking oil at 65 ºC and a constant speed of 1000 rpm. The entire process lasted for 60 min and after the complete conversion, the extract was left overnight in a separating funnel consisting of 3 layers. The impurities and glycerine were found at the bottom layer of the separating funnel, while the top consisted of pure biodiesel. The middle layer is viscid and needs to be separated. The methyl ester was further dried using a vacuum rotary evaporator at 70 ºC for 45 min to remove the moisture content, while a Whatman filter paper was used to extract any insoluble material. Generally, 5% (L5), 10% (L10), 15% (L15), 20% (L20), and 25% (L25) of biodiesel blended with lubricant were used to test the fluid.

Table 1: Composition of the various blending ratio (v/v)% of lubricants with WC70CI30 biodiesel used in this study.

Biodiesel-Lubricant	Lubricant	WC70CI30 biodiesel
BWCIL95	95	5
BWCIL90	90	10
BWCIL85	85	15
BWCIL80	80	20
BWCIL75	75	25
L100	100	-

2.4 Chemical and Physical Properties with Fuel Characterization

The physicochemical properties of the biodiesel and blended oil produced were determined using the EN/ASTM standards. Furthermore, the automatic density meter was used to measure the Kinematic (Anton Paar) at 15ºC and 40ºC (Mettler Toledo). The acid number was determined through the titration method using a KOH-ethanol solution with the gas chromatograph system equipped with an auto-injector used to quantitatively analyze the samples. The initial oven temperature of 150 ºC for 5 minutes (60 m x 0.25 mm id., 0.25) was later increased to 250ºC at a rate of 2 ºC for 17 minutes. The fatty acid of methyl ester (FAME) yield is calculated using equation (1).

$$Yield\% = \frac{W_a}{W_t} \times 100\% = \frac{W_{ester}}{W_{oil}} \times 100\% \quad (1)$$

Where W_a is the actual mass of FAME, and W_t is the theoretical mass. Meanwhile, Wester and Woil depict the weight of fatty acid methyl ester (g) and oil used (g) produced.

2.5 Test Procedure

2.5.1 Frictional and Wear Evaluation

Frictional and wear evaluation was performed using a high-resolution optical microscope in accordance with the ASTM D4172 standard. The friction of coefficient (CoF) is expressed by Equation (2):

$$T = \frac{\mu \times 3W \times r}{\sqrt{6}} \rightarrow \mu = \frac{T\sqrt{6}}{3W \times r} \quad (2)$$

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. Therefore, the wear scar diameter (WSD) was measured and analyzed using a DuCOM software with an installed image acquisition system.

Table 2: Test parameter for the 4 ball wear tests.

Test parameter	Unit	Value
Fuel Temperature	ºC	75
Test duration	S	3600
Applied load	N	392
Rotation	Rpm	1200

Table 3: Steel ball material.

Steel ball	Unit	Description
Diameter	mm	12.7
Hardness	HRc	62
Surface roughness	um	0.1C.L.A
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

3 RESULT AND DISCUSSION

3.1 Properties of the Biodiesel and Mixed Biodiesel

The thermophysical properties of WC and WC70CI30 biodiesel, such as kinematic viscosity, density, acid value and the flashpoint were determined using the ASTM methods, as shown in Table 4. WC70CI30 biodiesel had a lower kinematic viscosity at 40 °C (4.72 mm^2/s) than the WC (5.01 mm^2/s). According to Blin et al. (2013) the unsaturation factor is effectively applicable due to the lower viscosity in the highly unsaturated oils in glycerides carbon chains, which reduces the compactness of triglycerides units. Furthermore, the decrease in the mean molecular weight of the fatty acids (shorter carbon chain length) was justified by the lower kinematic viscosity (Wardana et al., 2018)

The densities of a moderate WC70CI30 and WC biodiesel samples at 15 °C were 861.8 kg/m^3 and 862, 1 kg/m^3 respectively (Table 4) and is consistent with ASTM D1298. Its value needs to be lower to have proper fuel atomization in the injectors. Further, lower density also reduces the smoke emission when the engine operates at maximum power (Sharma Dugala et al., 2021).

Acid value (AV) is a parameter that indicates the amount of free fatty acid and residual catalyst contained in the samples. Furthermore, the AV within the ASTM D664 was placed in the middle of the feedstock and used for biodiesel production. The residual acid has the potential to produce corrosion effects on the combustion engine as well as cause fuel auto-oxidation. It also reduces the biodiesel shelf-life due to the hydrolytic process, leading to the ester bond's cleavage. An elevated biodiesel acid value is able to increase the rate and level of its lubricant degradation, thereby causing severe corrosion in the engine systems (Haseeb et al., 2011). The acid value of the WC70CI30 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, although higher than that of the WC (0.13 mg KOH/g).

In addition, Table 4 shows that the FAME chemical compositions of the WC and WC70CI30 biodiesels produced are 97.45 (w/w)% and 98.84 (w/w)%., respectively. This indicates that the WC70CI30 biodiesel has higher FAME content than the WC. This fulfils the EN 14103:2011 standard test method requirement, which stipulates that the FAME content needs to be greater than 90 (w/w). The total FAME content is not 100 (w/w)% for all methyl esters due to an unidentifiable peak in the gas chromatograms. This is due to the repeatability and reproducibility limits of the gas chromatograph. *Calophyllum inophyllum* biodiesel relatively had a higher portion of unsaturated FAME compared to the waste cooking oil. Moreover, the high content of unsaturated fatty acid methyl esters was determined at low temperatures. Consequently, a high degree of unsaturation also causes the biodiesel to become more susceptible to oxidation. In addition, the total (0.125 (w/w)%) and free glycerols (0.016 (w/w)%) fulfils the specifications since its values are less than 0.25 (w/w)% and 0.02 (w/w)%, respectively. However, the results of the total (0.205 (w/w)%) and free glycerols (0.017 (w/w)%) are consistent with EN 14214. Meanwhile, two final immiscible products (methyl ester and glycerol) tend to become miscible because of the soapy surfactant effect, reducing the reaction yield. Furthermore, the glycerol was eliminated from the methyl ester during the separation. The WC70CI30 was discovered to have favourable lubrication characteristics, while the biodiesel properties were discussed in the following section.

3.2 Tribology Biodiesel of Lubricant Oil

The biodiesel and lubricant oil physicochemical properties were further examined. These are essential to investigate the compatibility of biodiesel blends with automotive materials. The thermophysical property study of the pure blended biodiesel with lubricant is shown in Table 5**.** On the contrary, the addition of lubricant oil to biodiesel is considered a potential automotive fuel. Besides, the current demand to carry out research related to engine fuel wear and frictional characteristics becomes imperative due to the frequent slippage of biodiesel into the lubricant.

Table 4: Fuel characterization of waste cooking oil, *Calophyllum inophyllum oil*, blended WC70CI30 oil, WC biodiesel and WC70CI30 biodiesel.

Property	Unit	WC raw oil	CI raw oil	WC70CI30 oil	WC biodiesel	WC70CI30 biodiesel
Kinematic viscosity at 40 °C	mm^2/s	49.05	65.48	54.12	5.01	4.72
Density at 15 °C	kg/m^3	904.4	929.2	912.2	862.1	861.8
Acid value	mg KOH/g	2.19	63.25	19.75	0.13	0.46
Flashpoint	^oC	—	—	—	154	160.5
FAME content	(w/w) %	—	—	—	97.45	98.94
Free glycerol	(w/w)%	—	—	—	0.017	0.016
Total glycerol	(w/w)%	—	—	—	0.205	0.125

Table 5: Kinematic viscosity and density of various blending ratios of lubricants with WC70CI30 biodiesel used in this study.

Name	Kinematic viscosity			Density
	40°C	100°C	VI	15°C
BWCIL95	76.34	11.78	148.7	0.8689
BWCIL90	62.26	10.49	158.1	0.8696
BWCIL85	51.33	9.268	164.9	0.8702
BWCIL80	42.82	8.242	171	0.8707
BWCIL75	35.98	7.419	178.8	0.8713
L100	92.08	12.94	138.4	0.8685

A lubricant's viscosity is its most important property and is affected by temperature. The lubricant's formulation and quality tend to impact the extent its viscosity drops with increasing temperature. The change in oil's viscosity due to an increase or decrease in temperature is called viscosity index (VI). This is important to discern whether the lubricant in question meets the asset's requirements based on the operating temperature range. Therefore, adopting a proactive approach to monitor the lubricant's viscosity makes a huge difference in machineries' lives. The viscosity index was calculated using the KV at 40ºC and 100ºC as stated in the ASTM (American Standards for Testing Material) D2270 table, meanwhile the value obtained for the base oil was 138.4.

The addition of lubricant to the waste cooking and WC70CI30 biodiesel increases the viscosity index. Subsequently, high viscosity results in good lubrication properties asides from the hydrodynamic condition. This simply means that they are subjected to slight change in extreme temperatures and are therefore considered to have stable viscosity. The blends are regarded as ideal oils because they maintain constant viscosity in all temperature changes. An increase in the VI value for the different blend proportions was also recorded. This complex mixture of hydrocarbon molecules is relevant for the classifications of products derived from crude oil and biodiesel and is readily available in a great variety of grades. It is a measure of a lubricant's molecular constitution from the hydrocarbon chain size standpoint. Moreover, viscosity is determined by the friction between individual molecules in a liquid, and it is responsible for resistance to flow. Therefore, the higher the intermolecular friction with longer molecular chains, the greater the viscosity.

3.3 Anti-wear Behaviour

The wear tests were carried out using the WC70CI30 biodiesel blend with lubricant at various ratios, as shown in Table . The CoF values were recorded automatically during the tests. The wear preventive tests were carried out with a constant load of 392 N (40 kg) at an operating temperature of 75 °C and 1200 rpm rotational speed for 1 hr. Every experiment was carried out thrice to obtain an average CoF value. Furthermore, the stability and Wear Scar Diameter (WSD) of the specimen was measured. The wear behaviour of all considered oil and biodiesel is shown in Figure 3.

It was observed that the duration of the run-in phase is dependent on the physical, chemical, and geometrical characteristics of the contact surfaces as well as the ability of the lubricant to provide wear protection. The CoF for all blended mixtures was significantly improved. The WCIL75 biodiesel has the least CoF (0.072) compared to other mixtures. Moreover, the WSD obtained for WC70CI30 biodiesel with lubricant L95 is 405.92 µm. It was observed that after the addition of biodiesel content in the various stages from L95 to L75, there was a reduction in the WSD.

Figure 3 shows the CoF and WSD realized from the biodiesel-lubricant blend and lubricant. As depicted in these figures, the CoF and WSD decreased following the increase of biodiesel. The CoF and WSD of BWCIL75 (0.072 and 338.73 µm) was determined to be lower compared to the lubricant (0.113077 and 428.64 µm). The WC70CI30 excellent anti-wear efficiency was explained using these 2 factors. The WC70CI30 has a lower molecular weight, are more active, easily decomposes and reacts with metal surfaces. Therefore, the presence of an unsaturated double bond in the molecule causes it to become more polar and sensitive in terms of adsorbing the newly exposed metal surface to assist in tribochemical reactions. Biodiesel in lubricants tend to improve the lubricity and reduces the WSD additionally, it is used as a wear reducer. Zulkifli et al. (2016) reported that the high concentration of the unique fatty acid methyl ester could be responsible for the lubricity-enhancing properties of biodiesel. Calero et al. (2015) further reported that the monoacylglycerides (MG) was proven to enhance lubricity of biodiesel. The polarity of biodiesel and vegetable oils has a chemical modification, which improves the lubricity of steel balls metal surface.

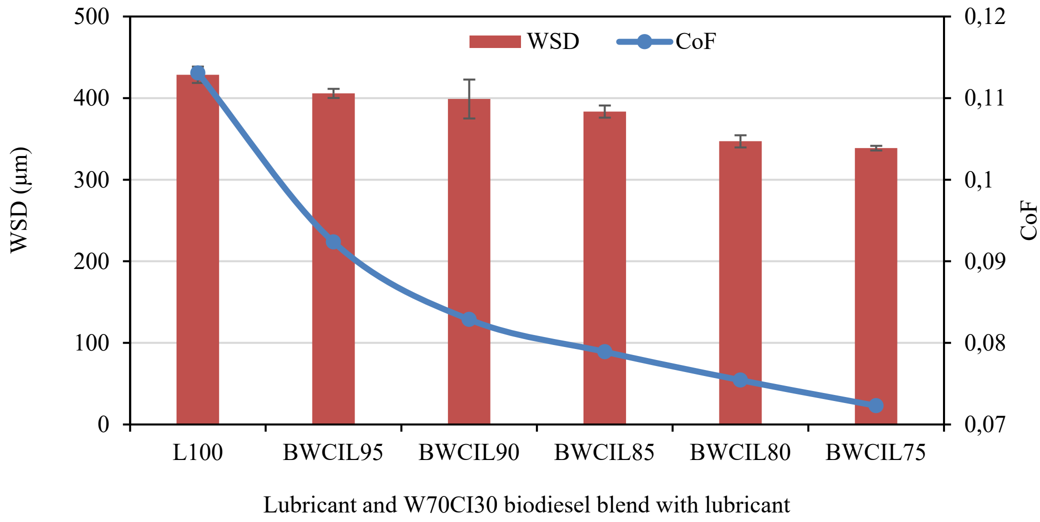

Figure 3: WSD and CoF for WC70CI30 biodiesel blend with lubricant (Conditions: Load: 392 N, Temperature: 75 °C, Duration: 1 hr).

4 CONCLUSIONS

In conclusion, the tribological behavioural characteristics of the lubricant and biodiesel blend with lubricant were evaluated using a 4-ball tribo tester with varying weight percentages. Furthermore, biodiesel is a potential bio-lubricant for automotive applications. It enhances mineral oils such as lubricity, high viscosity, acts as a good anti-wear property, and better frictional coefficient, low emission of metal traces into the atmosphere and rapid biodegradability. Moreover, its addition reduces the lubricating oil's viscosity, thereby investigating frictional and wear performances of the blending oils and biodiesel. This reduces the dependency on mineral oil-based lubricants by a few percentage points. Biodiesel components, such as fatty acid methyl esters, free fatty acids, and monoglycerides, reportedly improve the lubricity of biodiesel, thereby substantially reducing wear tendencies. However, the lubricity of biodiesel at higher temperatures is relatively decreased`. It can be effect the biodiesel-lubricant combination shows a much lower scar diameter than the blends. Furthermore, it reduces WSD and CoF, and provides an opportunity for industries to replace bio-fuel production with lubricants that meet certain quality standards. Furthermore, it reduces WSD and CoF, and provides an opportunity for industries to replace bio-fuel production with lubricants that meet certain quality standards.

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