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Tribological performances of palm oil derived-oleic acid biolubricant incorporated with antioxidant and viscosity improver

Amanda M. Norazman [1], Zulhanafi Paiman [1,2], Syahrullail Samion [1,2]

[1] Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, MALAYSIA.
[2] Institute for Sustainable Transport (IST), Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, MALAYSIA.
*Corresponding author: amandamaisarah@graduate.utm.my

KEYWORDS

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Antioxidants
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Fourball test
Coefficient of friction

ABSTRACT

A tribological study on oleic acid (OA) as a base stock, with added antioxidants and a viscosity improver, was conducted to determine the coefficient of friction (COF) and wear scar diameter (WSD) values. This study aims to identify the most effective antioxidant for achieving high-performance lubrication. Antioxidants such as tert-butylhydroquinone (TBHQ), butylated hydroxytoluene (BHT), and 3-tert-butyl-4-hydroxyanisole (BHA) were used in the four-ball test, performed under varying operating conditions in accordance with ASTM D4172 standards, with a load of 392 N, and speeds ranging from 1200 to 2400rpm at 75°C. The antioxidant concentration was maintained at 0.5wt.% while the viscosity improver, ethylene vinyl acetate (EVA), was used at 1.2 wt.%. The combination of TBHQ and EVA was observed to result in a maximum COF reduction of approximately 22% compared to BHA and BHT. Scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) analyses were conducted to examine the morphology and thermal behavior of the biolubricants, respectively. TBHQ produced the best results for COF and WSD, followed by BHT, and then BHA.

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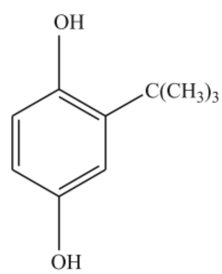
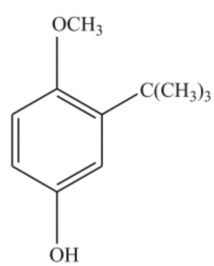
1.0 INTRODUCTION

The 21st century has seen a significant decline in ecological management practices. While efforts to restore ecosystems are widespread, effective implementation strategies are essential to prevent irreversible consequences for future generations. Friction is a pervasive force that adversely impacts systems, reducing their efficiency, whereas lubrication is vital for maintaining the efficiency of machinery and systems. Conventional lubricants, such as synthetic and mineral oils, pose environmental hazards due to their toxicity and harmful effects on waterways. Although these lubricants are widely used, substantial efforts have been made to develop biodegradable alternatives derived from plant oils. However, plant oil-based lubricants often exhibit low thermo-oxidative stability and poor viscosity stability. The incorporation of additives can improve their oxidative stability, while viscosity improvers enhance viscosity stability. Commonly used antioxidants include TBHQ, with EVA serving as a typical viscosity improver. Although other antioxidants are available, they are less frequently utilized. This study aims to investigate the tribological effects of an oleic acid-based lubricant formulated with the antioxidants TBHQ (Figure 1), BHA (Figure 2), and BHT (Figure 3), in combination with the viscosity improver, EVA.



TBH$_2$Q

Figure 1: Chemical structure of TBHQ (Tang et al., 2018).



BHA

Figure 2: Chemical structure of BHA (Tang et al., 2018).

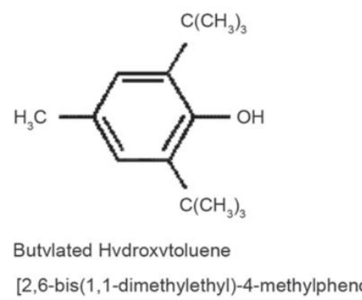


Figure 3: Chemical structure for BHT (Veghela et al., 2011).

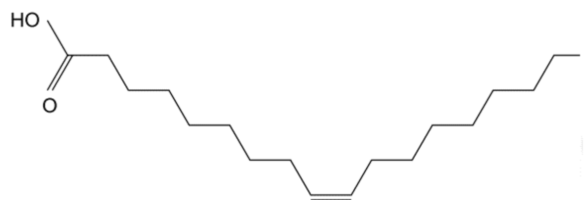


Figure 4: Chemical structure of oleic acid (Lopez et al., 2010).

Oleic acid (cis-9-octadecenoic acid) is an omega-nine fatty acid and has the formula $C_{18}H_{34}O_2$ ($CH_3(CH_2)_7CHCH$ $(CH_2)_7COOH$) (Figure 4). As a monounsaturated fatty acid with a double bond between the 9th and 10th carbons, it provides an active site for oxidation (Mishra et al., 2021). Oleic acid is commonly derived from plant oils, with olive oil being the primary source, followed by canola, sunflower, palm, and various other oils (Choi et al., 2010). However, in this study, oleic acid derived from palm oil, a plant native to Malaysia, is utilized.

1.1 Oleic Acid in Biolubricants

Palm oil has been used for centuries. According to the United States Department of Agriculture (2023), Malaysia is one of the world's leading producers of palm oil. Like most vegetable oils, palm oil consists primarily of triglycerides, which are composed of glycerol and three fatty acids. Oleic acid can be extracted through various processes, including hydrolysis (Nitbani et al., 2020) and enzymatic methods (Mohammed et al., 2003), among others. As an unsaturated fatty acid with a double bond, oleic acid exhibits notable resistance to oxidation. In the context of human nutrition, unsaturated fats are believed to help reduce cardiovascular diseases, including coronary risks and cholesterol levels.

In biolubricants, oleic acid is commonly used as a chemically modified base stock (Paiman et al., 2024; Chen et al., 2020; Zulhanafi & Syahrullail, 2019). They synthesized a biolubricant from oleic acid that demonstrated characteristics comparable to ExxonMobil PAO6 in terms of viscosity, pour point, thermal and oxidative stability. Another study by Salimon et al., (2011) used a chemical modification method in which oleic acid was epoxidized and and subsequently converted into various derivatives through a ring-opening reaction, resulting in enhanced low-temperature properties beneficial for cold climates. Additionally, Sarker et al., (2023) reported that various oleic acid triesters, produced through esterification, exhibited excellent low-temperature performance.

A study by Encinar et al., (2022) used chemical modification through transesterification of high oleic safflower oil, establishing optimal production conditions: a reaction temperature of 140°C, catalyst concentration (sodium methoxide) of 1% w/w, vacuum pressure of 400 mmHg, stirring rate of 350 rpm, and a FAME/alcohol ratio of 1:1. This process achieved a high yield (exceeding 92%), with the final product displaying strong oxidative and thermal stability. Among the various modification methods available—such as chemical modification, blending, additivization, and enzymatic methods—chemical modification is the most commonly used method for biolubricant production. However, a techno-economic analysis by Khan et al., (2022), suggests that the additive method is more cost-effective.

Oleic acid is also utilized as an additive, as demonstrated by Vilas Bôas et al., (2021), who combined fuel oil with oleic acid and added a lipase enzyme, resulting in ester properties superior to those of commercial ester. Additionally, Ramos et al., (2022) employed a chemical modification method by esterifying oleic acid to TMP ester for use as a biolubricant, identifying an optimal oleic acid to TMP ester ratio that offers favorable economic margins. While these studies examine the economic aspects and chemical properties of oleic acid-based lubricants, they lack tribological studies for practical application. Given Malaysia's abundance of palm oil supply, it could serve as a potential source of oleic acid for lubricants, although this may compete with the food supply.

1.2 Additive-Based Route

As Almasi et al., (2021) stated, additives encompass a range of substances that enhance biolubricant quality, including detergents, dispersants, viscosity modifiers, corrosion inhibitors, pour point depressants, extreme pressure additives, anti-wear agents, and nanoparticles. Prathiba et al., (2022) further demonstrated that incorporating titania nanoparticles reduced the coefficient of friction (COF) by 25% compared to a biolubricant made solely from chicken fat waste.

Tert-butylhydroquinone (TBHQ), butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) are classified as synthetic antioxidants. TBHQ is widely used in food, pharmaceuticals and cosmetic products, with a regulatory limit of 0.7mg/kg, as excessive exposure through ingestion or topical application can lead to various biological disorders (Khezerlou et al., 2022). However, safety data sheets provided by manufacturers do not specify a maximum allowable concentration for its use in lubricants. Nevertheless, a basic guideline suggests that excessive amounts, which are toxic in food, could also pose risks to aquatic organisms (Wang et al., 2021). Additionally, these antioxidants (TBHQ, BHT, and BHA) are used in packaging to enhance the tensile strength of plastic films (Fasihnia et al., 2020).

Synthetic phenolic antioxidants can prevent food oxidation, enhance food stability, and extend shelf life; however, excessive use or improper application may harm human health, and combining different antioxidants can amplify their toxic effects and cause side effects (Kim et al., 2016 as cited in Wang et al., 2021). In tribological studies, adding 0.3 wt.% TBHQ to palm oil produced COF values 14% lower than those of Shell T46 oil on a reciprocating linear machine (Ojaomo et al., 2023). Additionally, the study showed that TBHQ increased the oxidation onset temperature of palm oil by 22% compared to palm oil without TBHQ. In a separate study, Thbayh and Fiser (2022) reported that, among BHT, TBHQ, BHA, and curcumin, BHT is the most effective radical scavenger in terms of hydrogen atom transfer (HAT) mechanisms. Radical scavengers, or antioxidants, mitigate the harmful effects of free radicals by neutralizing them, primarily through HAT mechanisms, where a scavenger donates a hydrogen to a free radical, forming a stable radical. Encinar et al., (2022) also noted that TBHQ concentrations as low as 0.05 wt.% can enhance oxidative stability for up to 10 hours.

Ali et al., (2023) tested EVA and TBHQ in fig oil, obtaining results comparable to commercial SAE-5W-30 when using EVA at 0.9 mass% and TBHQ at 0.3mass%. In contrast, Hamid et al., (2021) found that a higher concentration of 4.0% EVA was required to achieve optimal performance of Musa Aluminata Balbisiana (MBS) oil extract under the lowest COF condition. Additionally, Ibrahim Ali et al., (2023) reported that adding 0.5% EVA to base fig oil reduced COF by 30% compared to base fig oil alone, as tested with a 120kg mass on a four-ball tester.

Most of these studies focus primarily on TBHQ as the preferred additive, with limited research on BHA and BHT. Furthermore, they often lack tribological applications, particularly the use of oleic acid as a base stock. This study aims to investigate the tribological effects of antioxidants BHA, BHT and TBHQ, along with viscosity improver EVA, in an oleic acid base stock.

1.3 Antioxidants in Biolubricants

Antioxidants are one of several options for modifying the composition of biolubricants. A techno-economic analysis by Khan et al., (2022) found that chemical modification is 30-160% more expensive than using additives in biolubricants. Chemical modification methods are prevalent in research; a simple search on Web of Science shows that over 90% of studies on oleic acid biolubricants focus on chemical modifications rather than solely additives.

The benefits of using additives have been previously discussed, with one key aspect being the improvement of tribological properties—specifically those related to friction and wear. Joshi et al., (2023) demonstrated that waste cooking oil, when converted into biolubricant, exhibited a lower coefficient of friction compared to commercial lubricating oil.

Despite the additive route being more cost-effective, few researchers choose to apply this method. Research on antioxidants such as BHA, BHT, and TBHQ remains limited, even though these compounds have broad applications across various fields. This is especially apparent in studies where more emphasis is placed on viscosity improvers than on TBHQ, as shown in Ali et al., (2023) using fig oil as a base stock. Mishra et al., (2021), in a review of antioxidants in edible oils, found that only five authors since 2003 have used BHA, BHT and TBHQ as antioxidants. Additionally, antioxidant concentrations in oils other than oleic acid typically range from 100-200ppm. This trend is particularly evident for oleic acid, which is more commonly studied in chemical modification methods than as an additive, as seen in the work of several authors (Salimon et al., 2011b; Chen et al., 2020; Sarker et al., 2023b; Thakur et al., 2023).

Previous studies indicate that oleic acid, particularly as a base stock, is not widely used in the field of tribology, including the application of BHA, BHT, and TBHQ as antioxidants and EVA as a viscosity modifier.

2.0 EXPERIMENTAL PROCEDURE

This study employed a four-ball tester, as shown in Figure 6, following ASTM D4172 (D4172, 1999). The four-ball tribometer is a testing device used to evaluate the anti-wear and friction properties of lubricants under controlled conditions. It operates by rotating one ball against three stationary balls, all coated with the lubricant under test, under a specified load, speed, and temperature. The device measures the coefficient of friction (COF) and wear scar diameter (WSD) on the balls, providing insights into the lubricant's effectiveness in reducing wear and friction between metal surfaces. A close-up view of the mechanism is presented in Figure 7.

The four-ball tribometer collects data through sensors and measurement systems integrated into the device. During testing, the rotating ball generates friction against the three stationary balls, with sensors measuring torque and rotational speed to calculate the COF. After the test, the WSD on the stationary balls is measured using optical or digital microscopy. These measurements yield quantitative data on the lubricant's effectiveness in reducing friction and wear.

2.1 Sample Preparation

Since the EVA has a melting point of 120°C (Table 5), it is incorporated into oleic acid using a hot plate and a magnetic stirrer. All antioxidants are maintained at 0.5wt.%, while EVA is added at 1.2wt.%. For 100g of oleic acid, the mass of each antioxidant is calculated by multiplying 0.5% by the mass of oleic acid, with the same approach applied for EVA. After incorporating EVA, the biolubricant undegoes homogenization at 13000rpm for 45 minutes to fully dissolve all the antioxidant particles. The result is shown in Figure 5. The properties of oleic acid are listed in Table 2.

2.2 Experimental Set-Up

Before opearting the fourball tester (Figure 6), 10mL of biolubricant is added to the the ball pot, ensuring it covers the stationary balls (Figure 7). The speed and temperature are set using the controller, while the desired load is applied using load plates on the load holder. Once the specified temperature is reached, rotation begins, and the test runs for one hour according to ASTM D4172. During the test, frictional torque, normal load and COF are recorded.

Figure 5: Oleic acid biolubricant.

Figure 6: Fourball test rig.

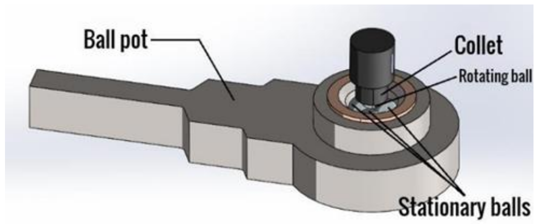


Figure 7: Ball pot assembly (Suresha et al., 2022).

2.3 Experimental Conditions

The experimental condition for the fourball tester experiment is tabulated in Table 1.

Table 1: Experimental condition for fourball tester.

Speed (RPM)	Load (N)	Temperature (℃)
1200-2400	392	75

2.4 Physicochemical Properties of Bio-Based Lubricants

The physicochemical properties of oleic acid based from supplier's safety data sheet.

Table 2: Oleic acid fatty acid properties (Evyap Oleo, 2019).

Parameter	Unit	Specification
Appearance at 30°C	-	Pale yellow liquid
Acid value	mg KOH/g	195-205
Saponification value	mg KOH/g	196-206
Iodine value	g I2 / 100g	88.0 – 95.0
Cloud point	°C	7.0 Max
Fatty Acid Composition	**Unit**	**Specification**
Up to C 18 (Saturated)	% by GC	12.0 Max
C 18:1	% by GC	75.0 Min
C 18:2	% by GC	13.0 Max
Others	% by GC	1.0 Max

Oleic acid is selected for its favorable melting point, thermo-oxidative stability and viscosity (McNutt and He, 2016). To further enhance the oxidative stability and viscosity of the oleic acid, TBHQ, BHA, and BHT are added at 0.5wt.% while viscosity improver, EVA is maintained at 1.2wt.%. The text matrix is provided in Table 3.

Table 3: Test matrix for the fourball test.

Test no	Load (N)	Speed (RPM)	Temperature (℃)	Composition
1	392	1200	75	0.5 wt.% TBHQ + 1.2 wt.% EVA
2	392	1200	75	0.5 wt.% BHA + 1.2 wt.% EVA
3	392	1200	75	0.5 wt.% BHT + 1.2 wt.% EVA
4	392	1600	75	0.5 wt.% TBHQ + 1.2 wt.% EVA
5	392	1600	75	0.5 wt.% BHA + 1.2 wt.% EVA
6	392	1600	75	0.5 wt.% BHT + 1.2 wt.% EVA
7	392	2000	75	0.5 wt.% TBHQ + 1.2 wt.% EVA
8	392	2000	75	0.5 wt.% BHA + 1.2 wt.% EVA
9	392	2000	75	0.5 wt.% BHT + 1.2 wt.% EVA
10	392	2400	75	0.5 wt.% TBHQ + 1.2 wt.% EVA
11	392	2400	75	0.5 wt.% BHA + 1.2 wt.% EVA
12	392	2400	75	0.5 wt.% BHT + 1.2 wt.% EVA

2.5 Additives and Data Collection and Analysis

Data collection and analysis using a four-ball tester involve measuring the coefficient of friction (COF) and wear scar diameter (WSD). During the test, sensors record the torque and rotational speed to calculate the COF, indicating the lubricant's friction-reducing capability. After the test, the wear scar on the stationary balls is measured using optical or digital microscopy to determine the WSD, which reflects the lubricant's effectiveness in minimizing wear. These values provide crucial insights into the lubricant's performance under specific conditions. Technical specification for TBHQ, EVA, BHT and BHA were shown in Table 4, 5, 6 and 7 respectively.

Table 3: Technical specification of TBHQ (Spectrum Chemical Mfg. Corp, 2012).

Item	Explanation
Physical state	Solid
Odor	Slight. Aromatic.
Appearance	Crystalline
Color	White
Molecular/formula weight (g/mole)	166.22
Formula	C10H14O2
Melting point/range (°C)	127
Boiling point/range (°C)	295
Specific gravity	1.05
Flashpoint (°C)	171

Table 4: Technical specification of EVA (Sahara International Petrochemical Company, 2022).

Type	Detail
Physical state	Solid in the form of pellets
Colour	Clear, opaque, off-white
Odour	Faint odor
Melting point	80-120 ℃
Boiling point	228 ℃

Table 5: Technical specification of BHT (Sigma-Aldrich, 2024).

Item	Detail
Physical state	Crystalline powder
Color	Colorless
Odor	Odorless
Melting point/freezing point	63 – 73 °C
Initial boiling point and boiling range	265 °C

Table 6: Technical specification of BHA (Sigma-Aldrich, 2006).

Item	Detail
Product name	Butylated hydroxyanisole
Purity	98.5%
Formula	C11H16O2
Formula weight	180.24 g/mol
Appearance (color)	White to light yellow
Appearance (form)	Powder
Solubility (color)	Colorless to faint yellow

3.0 RESULTS AND DISCUSSION

Antioxidants at a concentration of 0.5wt.% (TBHQ, BHT, and BHA) were added into oleic acid base stock to improve its oxidation stability. A viscosity improver, EVA, was added at a concentration of 1.2wt.%, determined to be the optimum concentration based on previous studies. According to Table 3, the range of speed was varied from 1200 rpm to 2400 rpm.

3.1 Viscosity Analysis

Viscosity is an important measure of flow for fluids, where steep slope indicates a significant change in viscosity with temperature, conversely a gentle slope indicates a smaller change in viscosity with temperature. Generally, a gentle slope is recommended because it exhibits predictable performance across varying temperatures. From Figure 8, TBHQ displays a low viscosity at low temperatures which ensures good lubrication and protection during startup in cold conditions. Conversely, a higher curve of viscosity means that the oil is able to provide a better film strength and load-carrying capacity and are less likely to thin out at higher temperatures. Across the graph, all three antioxidants show relatively similar trends whereas the temperature increases, the kinematic viscosity also decreases. Adequate viscosity at high temperatures prevents thinning of the lubricant, resulting a protective film under operating conditions. According to Figure 8, the best performance of viscosity depends on the application of machinery, but ultimately where little changes in viscosity over time remain stable is preferred. From a cold climate standpoint, the best antioxidant performance is TBHQ, followed by BHT, and lastly BHA.

As shown in Table 8, the viscosity range at 40°C for various vegetable oils spans from 28 cSt to 52 cSt, while mineral oil has the highest viscosity at 103 cSt, and biodiesel has the lowest at 3.845 cSt. Compared to other vegetable oils, oleic acid has a relatively lower viscosity than sunflower oil, which can be attributed to differences in fatty acid composition. Being a monounsaturated fat, oleic acid generally has a lower viscosity compared to oils with higher polyunsaturated fatty acid content, such as soybean oil.

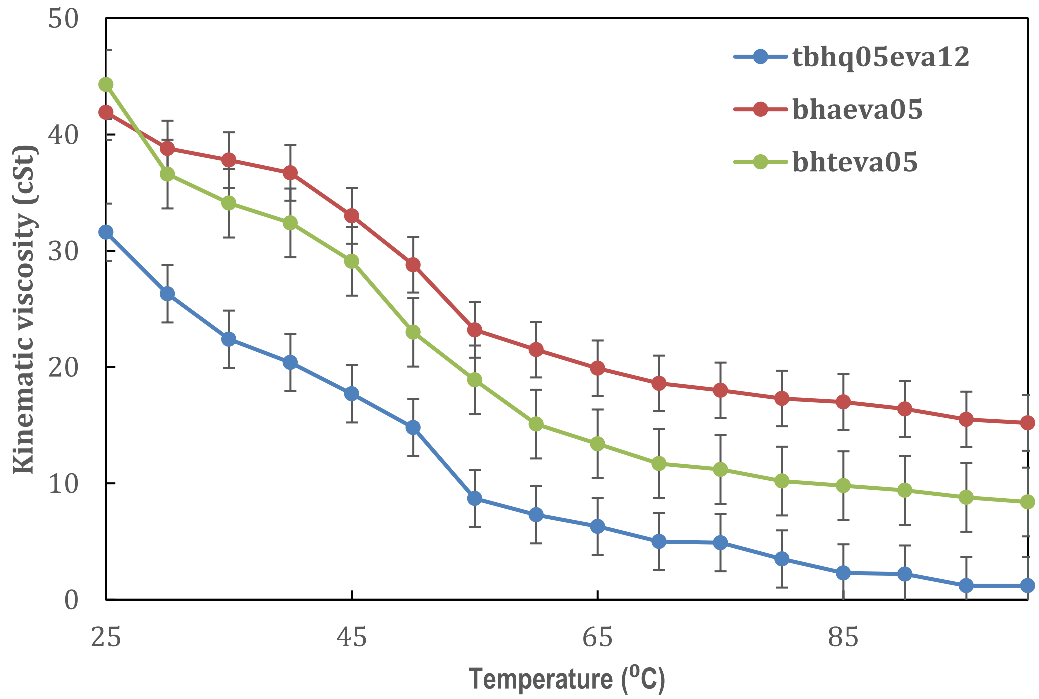


Figure 8: Graph of kinematic viscosity vs temperature for TBHQ, BHA, and BHT.

Table 7: Viscosity of different oils.

Oil	**Kinematic viscosity at 40°C (cSt)**	**Kinematic viscosity at 100°C (cSt)**	**Source**
Olive oil	37.042	7.814	(Kalam et al., 2017)
SAE15W40	103	16.09	(Kalam et al., 2017)
Babassu biodiesel	3.845	-	(Girardi et al., 2020)
Soybean oil	28.86	7.55	(Salih and Salimon, 2022)
Sunflower oil	40.05	8.65	(Salih and Salimon, 2022)
Rapeseed oil	45.60	10.07	(Salih and Salimon, 2022)
Palm oil	52.4	10.2	(Salih and Salimon, 2022)

3.2 Thermal Property Analysis

Differential Scanning Calorimetry (DSC) is a technique used to observe changes in enthalpy, revealing the exothermic and endothermic behaviors of a material, which correspond to its heat-releasing and heat-absorbing properties (Hettiarachchi et al., 2023).

Two types of thermal analysis profiles were studied, specifically cooling and heating experiments with the following procedures: the analysis is defined by using nitrogen as purge gas; 1) exothermic tests: the sample is held at 60°C isotherm for 10 min to eliminate the thermal history of the samples, then cooled at 2 ◦ C/min to −60 ◦ C and held for 2 min. 2) endothermic tests: The sample is heated from −60 to 100 ◦ C at 10◦ C/min.

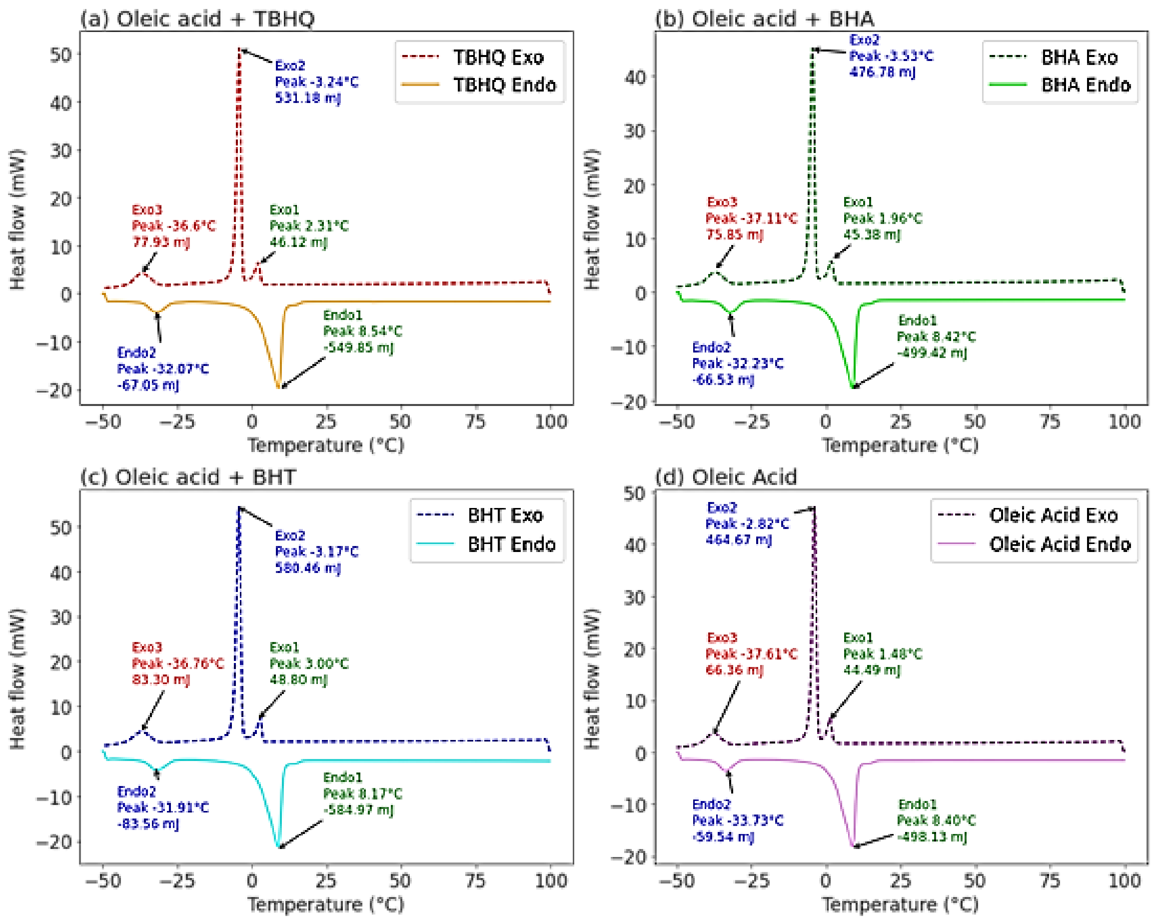


Figure 9: DSC thermograms for different antioxidants, (a) TBHQ, (b) BHA, (c) BHT, including comparison to (d) oleic acid.

Figure presents the DSC thermogram for the antioxidants TBHQ, BHA, BHT in comparison to oleic acid. Exothermic peaks are labelled with 'Exo' following each biolubricant mixture name, such as TBHQ Exo, BHT Exo, and BHA Exo while endothermic peaks are labelled with 'Endo', for instance, TBHQ Endo and BHT Endo. Each graph displays three exothermic peaks and two endothermic peaks. The maxima of these peaks are indicated by the total area of energy under the curve, measured in milliJoules (mJ), along with the peak temperature in degrees Celsius.

In Figure 9(a), the exothermic peaks Exo1, Exo2, and Exo3 have maxima at 2.31°C, -3.24°C, and -36.76°C, respectively, with crystallization points observed before the change in curve concavity. The variation in crystallization points may be due to the presence of different crystal phases forming polymorphs of the complex triglyceride molecules in oleic acid (Ajithkumar *et al*, 2009 cited in Hettiarachchi *et al.*, 2023). Additionally, two endothermic peaks, Endo1 and Endo2, appear at at 8.54°C and -32.07°C, respectively. The onset temperature at the concavity change corresponds to the melting point.

From the DSC thermograms of oleic acid formulations with different antioxidants, sample OA displays an endothermic peak maximum at -33.73◦ C (Figure 9(d)). Minimal variation is observed among the four thermograms in Figure 9, indicating that the antioxidants do not significantly impact the thermal properties of the formulations. The endothermic peak transition temperature (EPTT), which corresponds to the pour point (PP) of the fluid, remains nearly the same across all formulations and closely matches that of oleic acid (Figure 9(d)). The onset temperature, marking the beginning of melting, is also similar for all antioxidants when compared to oleic acid. Comparable trends are noted in the exothermic regions of DSC thermograms, which signify the onset of solidification, as indicated by the exothermic labels in the graphs.

In reference to oleic acid, the endothermic peaks differ by 1.65%, 0.24%, and 2.78% in temperature for TBHQ, BHA, and BHT, respectively. Cedeno-Sanchez *et al.*, (2023) reported similar DSC thermogram trends, where high oleic palm oil shows three exothermic peaks and two endothermic peaks. The first exothermic peak often represents an initial crystallization process or a partial oxidation reaction, potentially indicating the formation of new phases or the onset of chemical reactions. The second exothermic peak may correspond to a more pronounced oxidation reaction, where the material undergoes further transformation, such as degradation or cross-linking, or it could signal a secondary crystallization process. This transition reflects the recrystallization of low melting polymorphs to higher melting forms, advancing from a hexagonal subcell (α) to an orthogonal subcell (β') and eventually to a triclinic subcell (β), prior to melting. The third exothermic peak might represent the final stages of oxidation or thermal degradation, indicating continued reaction with oxygen or breakdown into simpler components. For material with multiple phases or components, this peak could also signify distinct parts of the material reacting or decomposing.

The first endothermic peak likely corresponds to the melting of specific crystalline phases within the material, marking the initial phase transition from solid to liquid. A second endothermic peak could indicate an additional melting process or another type of phase change, especially if the material contains a complex mixture of components or multiple additives. This second peak may also suggest the volatilization of a component or a phase transition related to decomposition products formed during the exothermic reactions.

3.3 Analysis of Coefficient Of Friction

The run-in period in the first 10 seconds represent an instability, because the machine has just started operating. The transition period from run-in to steady state (10 seconds to 200 seconds) is where the COF has started to stabilize. After 200 seconds, the steady state starts and continues until 3600 seconds. Based on Figure 10, almost all the biolubricants at the run-in period start at highest value, before achieving a relatively stable COF. TBHQ has achieved the lowest COF of all three antioxidants, followed by BHA, then BHT. Additionally, TBHQ has relatively small peaks of change, which is the opposite of BHA, having large peaks of change across time. This may be due to TBHQ's antioxidant activity in comparison to other synthetic and natural antioxidants in the market (Tang et al., 2010).

For vegetable oils, which are composed of triglycerides, surface protection is achieved by the adsorption of the polar portion of the oil molecules (the ester groups in triglycerides) onto the metal surface. This forms a barrier that helps reduce the coefficient of friction (Panchal et al., 2017 cited in Jedrzejczyk et al., 2021).

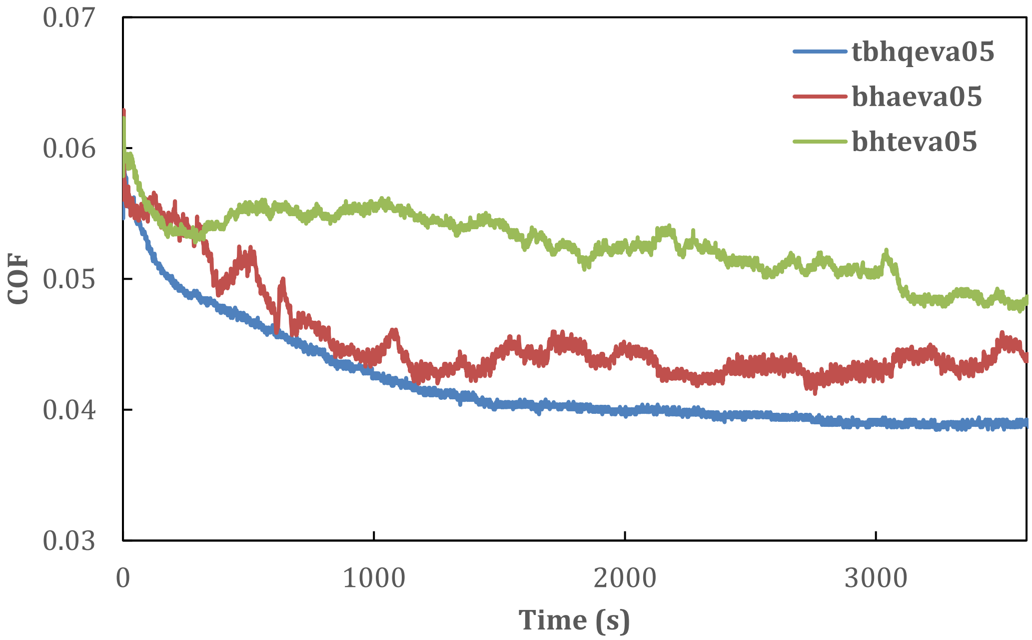


Figure 10: COF vs Time graph for 1200rpm.

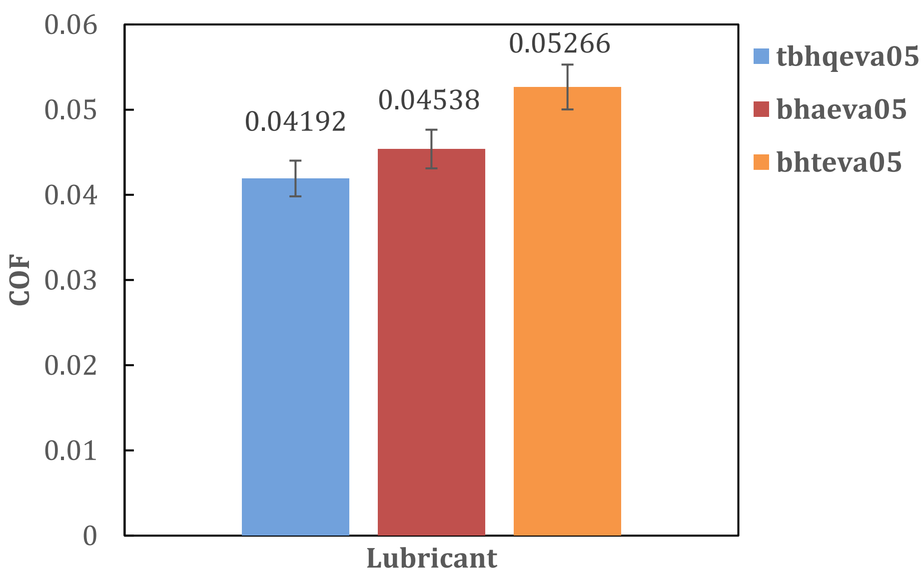


Figure 11: Average COF for 1200rpm.

The average COF is obtained after 1 hour of the fourball test, as shown in Figure 11, 'tbhqeva05' represents TBHQ, 'bhaeva05' for BHA, and similarly 'bhteva05' for BHT. Out of the three antioxidants, TBHQ proved to be the lowest value at 0.04192, followed by BHA at 0.04538 and lastly BHT at 0.05266. Comparing TBHQ to BHA and BHT, TBHQ yielded a lower COF at 7.9% and 22.7%, respectively.

In comparison to prior studies, the COF values observed in this study, ranging from 0.03 to 0.05, are significantly lower than the typical COF values reported for vegetable oils and lignin-based additives. Jedrzejczyk *et al.*, (2021) documented COF values between 0.10 and 0.13 using lignin-based additives, aligning with other research findings on vegetable oils (McNutt & He, 2016; Ortega-Alvarez *et al.*, 2019 as cited in Jedrzejczyk *et al.*, 2021). This stark contrast suggests that the formulations tested in this study demonstrate enhanced anti-friction properties, indicating potential advantages in applications requiring lower friction coefficients.

This outcome emphasizes the effectiveness of the current formulations in reducing friction, potentially attributable to the unique combination of oleic acid with antioxidants and viscosity improvers like EVA.

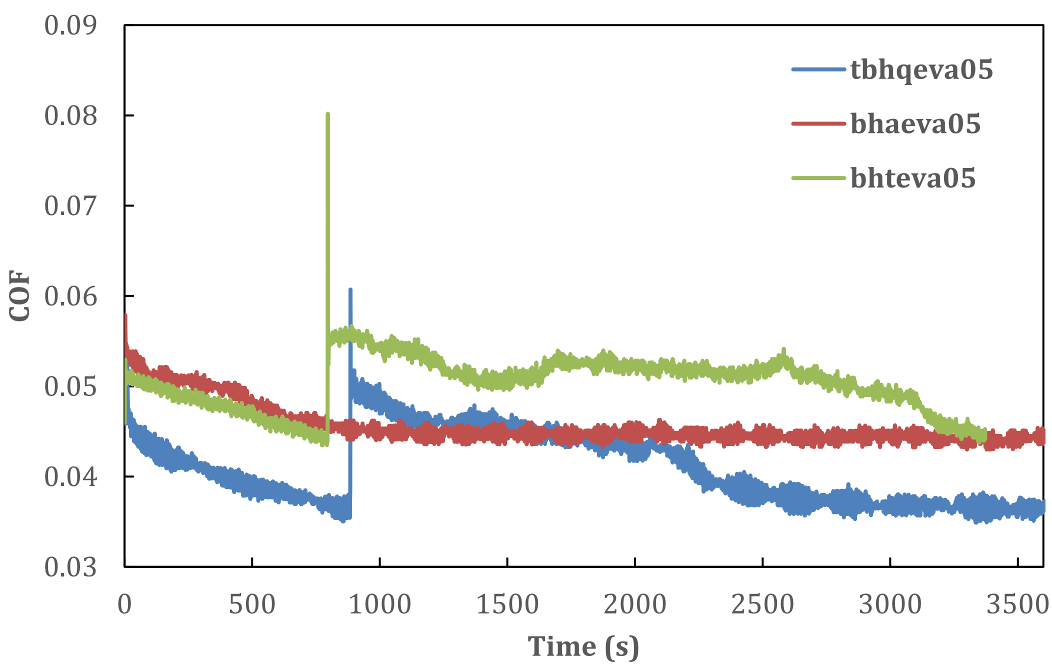


Figure 12: COF vs Time graph for 1600 rpm

Contrary to the graph for 1200rpm, BHT and TBHQ showed a sudden spike in the steady state region in Figure 12. They both show similar trends, a relatively stable decrease in value, a sudden increase, and then a slow decrease almost similar to the first 700 seconds. On the other hand, BHA display a relatively stable variation in COF. In the first 10 seconds it has a spike, before decreasing steadily. Despite this trend, TBHQ still managed to surpass all the other two antioxidants, providing the lowest COF value which is followed by BHA, and then BHT. The study by Hou *et al.*, (2024) utilized the same percentage of TBHQ which show the lowest COF among all lubricant combinations using castor oil as base oil. It is found that the addition of TBHQ increased the dispersion stability, synergistic effect between TBHQ and other particles exist in the oil to improve the tribological properties.

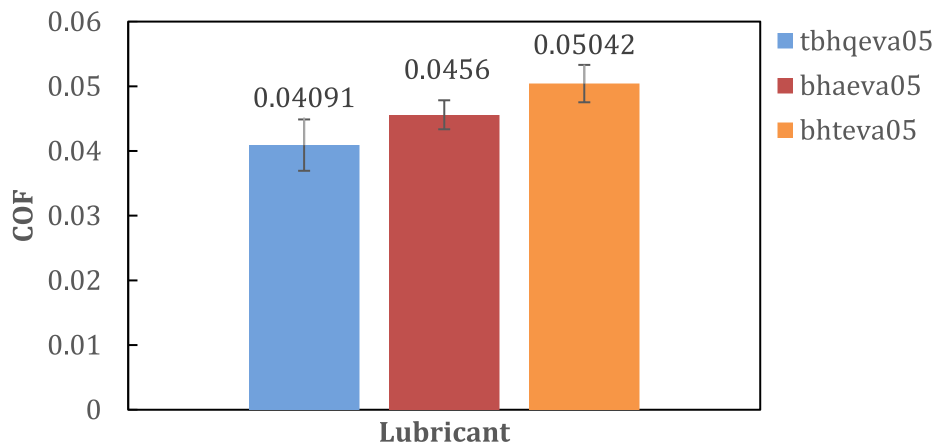


Figure 13: Average COF for 1600rpm.

Similarly to 1200rpm, the mean COF values follow the same trend, where TBHQ has the lowest value, followed by BHA, and lastly BHT. For TBHQ, the average COF is 0.04091, while BHA is at 0.04560, and BHT at 0.05042 as seen in Figure 13. Comparing TBHQ to BHA and BHT, which TBHQ yielded a lower COF at 10.8% and 20.8% respectively. Girardi *et al.*, (2020) also reported that TBHQ yielded the most percentage improvement compared to other antioxidants such as BHT and BHA in biodiesel.

According to Figure 14, the behavior of TBHQ is very erratic, where huge variations of COF occur, although it remains the lowest COF. However, BHA and BHT share the same behavior, where little changes occur in its COF values and remain very stable over time. Despite the huge variation in COF values of TBHQ, its mean COF remained the lowest out of the three antioxidants as in Figure 14. The erratic lines may be due to thermal effects, because at high speeds friction generates more heat along with insufficient lubricant, causing more metal-to-metal contact.

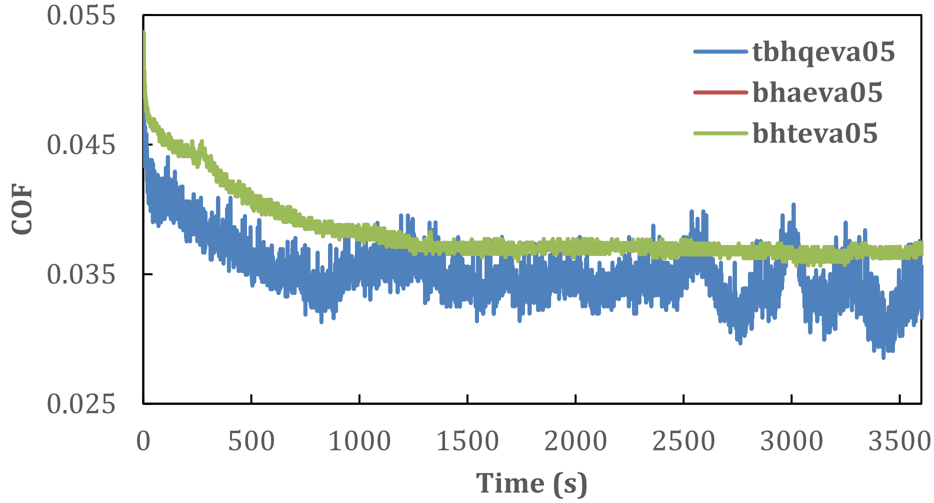


Figure 14: COF vs Time graph for 2000rpm.

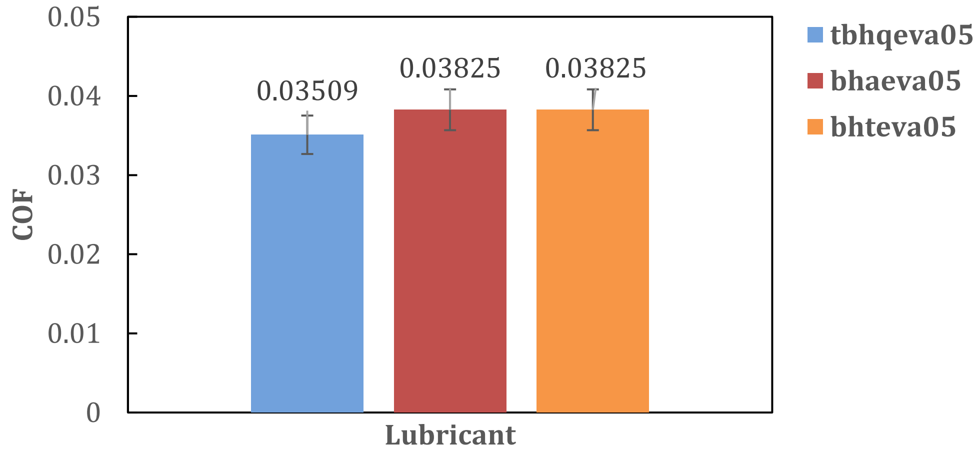


Figure 15: Average COF for 2000rpm.

Similarly to other speeds (1200rpm and 1600rpm), TBHQ holds the longest record for achieving the lowest TBHQ value in Figure 15. Its COF value is the lowest out of the three antioxidants, at 0.03509, followed by BHA and BHT, which share similar values of 0.03825. Comparing TBHQ to BHA and BHT, which TBHQ yielded a lower COF than BHA and BHT at 8.6%.

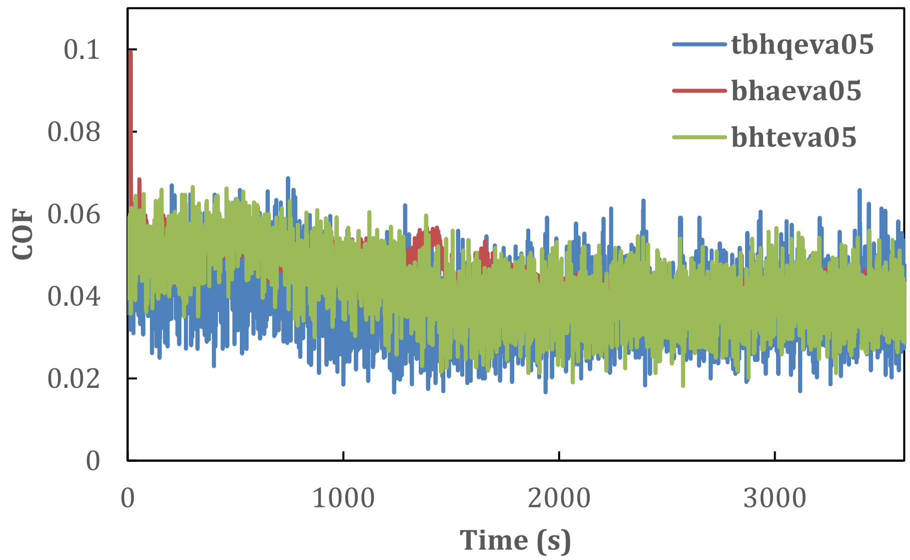


Figure 16: COF vs Time graph for 2400rpm.

Based on Figure 16, the speed of 2400rpm has resulted in a different behavior of trend compared to previous speeds. All of the antioxidants have very erratic lines, spanning huge widths, and behave very noisy. Despite this, TBHQ remains the lowest COF, followed by BHT, then BHA. Although the patterns are erratic, BHA behaves relatively stable compared to others. As higher speeds generate more friction, the temperature likely increased, causing more vibrations and trapped debris during rotation.

According to Figure 17, TBHQ remains the lowest COF value, followed by BHT and then lastly BHA. TBHQ's COF is at 0.03879, while BHT at 0.04254 and lastly BHA at 0.04510. Comparing TBHQ to BHA and BHT, which TBHQ yielded a lower COF at 15% and 9.2%, respectively. It is evident that TBHQ is the best antioxidant that show the lowest COF values even across different speeds. This is due to TBHQ's higher antioxidant activity compared to others. This is evidenced by Hou et al., (2024) that utilized 0.5wt.% TBHQ show the lowest COF values when compared to 1.0wt.%, 1.5wt.%, and even 2.0wt.% TBHQ.

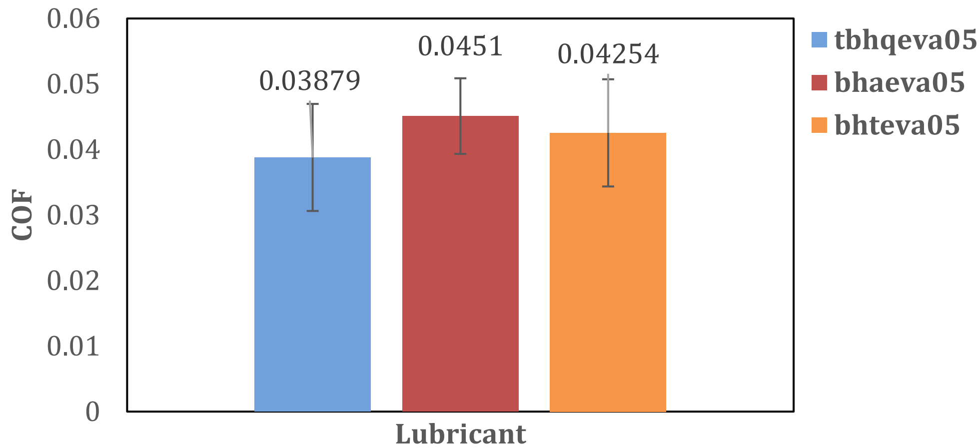


Figure 17: Average COF for 2400rpm.

3.4 Analysis of Wear Scar Diameter

The WSD is taken from the steel balls, where the scar is captured into a microscope program in a computer. The results are shown in Table 9. The table is shown graphically in Figure 16:

Table 9: Results for WSD.

No.	Load (N)	Speed (RPM)	Temp (°C)	Composition	Average WSD (mm)
1	392	1200	75	0.5 wt.% TBHQ + 1.2 wt.% EVA	0.3596
2	392	1200	75	0.5 wt.% BHA + 1.2 wt.% EVA	0.5319
3	392	1200	75	0.5 wt.% BHT + 1.2 wt.% EVA	0.5015
4	392	1600	75	0.5 wt.% TBHQ + 1.2 wt.% EVA	0.5868
5	392	1600	75	0.5 wt.% BHA + 1.2 wt.% EVA	0.3777
6	392	1600	75	0.5 wt.% BHT + 1.2 wt.% EVA	0.6730
7	392	2000	75	0.5 wt.% TBHQ + 1.2 wt.% EVA	0.5563
8	392	2000	75	0.5 wt.% BHA + 1.2 wt.% EVA	0.3806
9	392	2000	75	0.5 wt.% BHT + 1.2 wt.% EVA	0.5086
10	392	2400	75	0.5 wt.% TBHQ + 1.2 wt.% EVA	0.4753
11	392	2400	75	0.5 wt.% BHA + 1.2 wt.% EVA	0.6606
12	392	2400	75	0.5 wt.% BHT + 1.2 wt.% EVA	0.6038

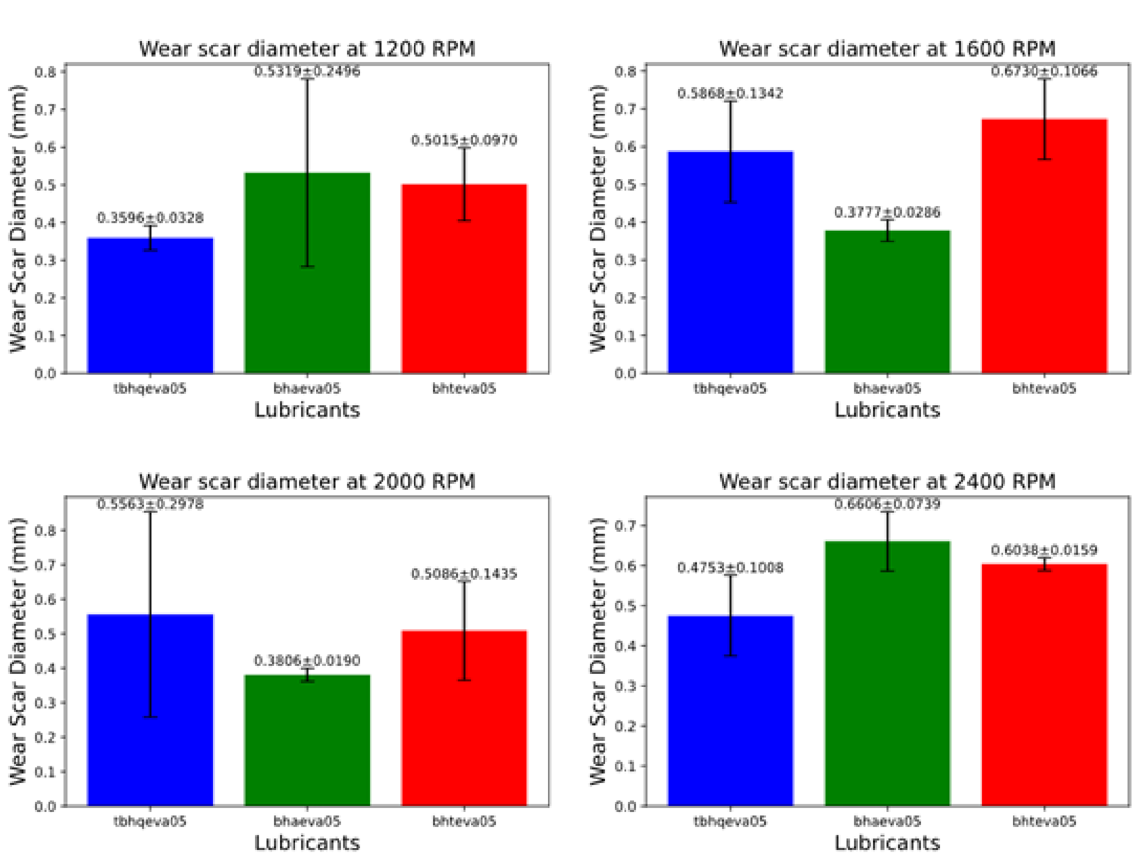


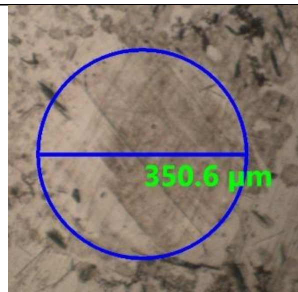
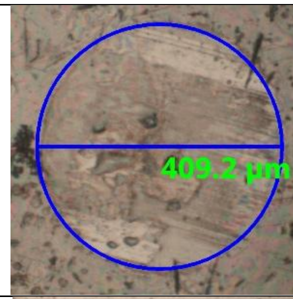
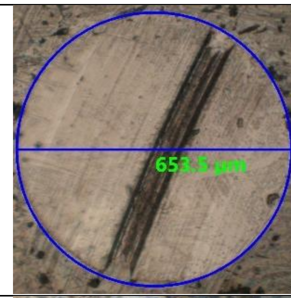
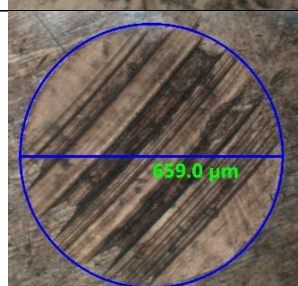
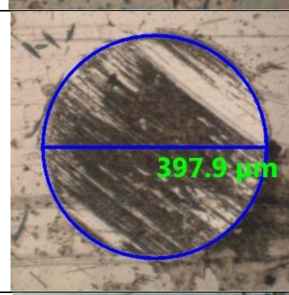
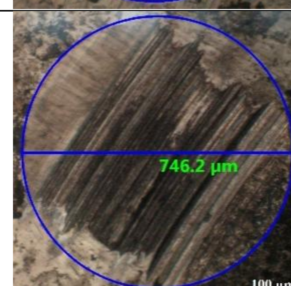
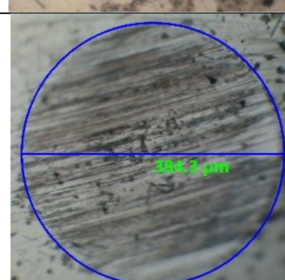
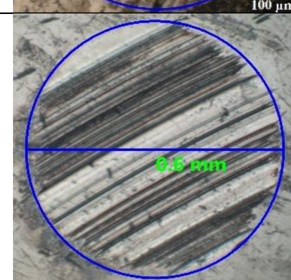
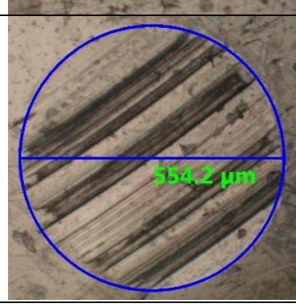
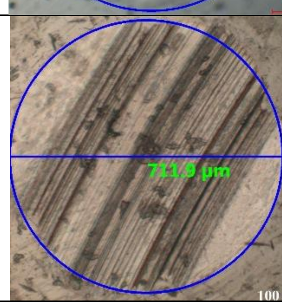
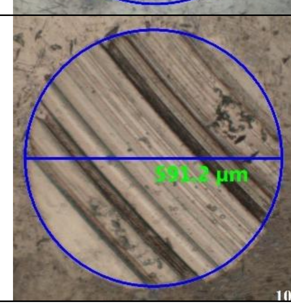
Figure 18: WSD vs lubricants graph for different speeds.

According to Figure 18, TBHQ provided the lowest WSD (0.3596mm) at 1200rpm compared to other biolubricants in different speeds; this is due TBHQ providing better protection to the wear surface (Hou *et al.*, 2024). BHA also proved its anti-wear properties next to TBHQ, with the lowest WSD at 0.3777mm in 2000rpm. Although BHT has the highest wear scar compared to all, its highest at 0.6730mm, it is only about 25% higher compared to its lowest wear scar at 0.5015mm. In the study of Jedrzejczyk *et al.*, (2021), the wear volume of BHT was reported to have reductions by 30-40%. Additionally, the study by Hou *et al.*, (2024) show one of the lowest value of WSD by 0.5wt.% TBHQ in castor oil.

3.5 Physical Wear Appearance

Images of the wear scar are analyzed under a microscope, as shown in the Table 10 below. The wear scars on the metal balls exhibit varying depths, which correspond to their coloration—shallow scars appear lighter, while deeper scars are darker. These scars consist of parallel lines, or furrows, with most appearing sparse. Darker lines indicate rougher, more pronounced furrows, whereas smoother, lighter furrows, represent less severe wear. Each wear scar is circled to highlight the primary area of wear concentration, making it easier to observe where significant scarring has occurred. This visual differentiation aids in assessing the wear severity and distribution across the tested metal surfaces.

Table 10: Wear scar images for all lubricants in different speeds.

Speed (RPM)	**Lubricant**		
	0.5wt.% TBHQ + 1.2wt.% EVA	0.5wt.% BHA + 1.2wt.% EVA	0.5wt.% BHT + 1.2wt.% EVA
1200			
1600			
2000			
2400			

Further inspection of wear scar is shown in images Figure 19-Figure 24 of antioxidants TBHQ, BHA, and BHT and speed 1200 RPM and 2400 RPM. According to Figure 19, TBHQ at 1200 RPM show a clear perimeter of scar compared to the perimeter at 2400 RPM. Although defined, at 1200 RPM the abrasive tracks are not as many at the higher speed (2400 RPM). The same pattern also applies to BHA (Figure 21-Figure 22), a higher speed corresponds to many abrasive tracks although not as clear of a wear scar perimeter. The tracks are lighter, and run in parallel lines to each other. Examining BHT in Figures 23 and 24, the abrasive tracks are lighter in 1200 RPM compared to 2400 RPM. It is apparent that in the higher speed, BHT show several pitting spots where the scars run deeper, indicating a higher wear scar.

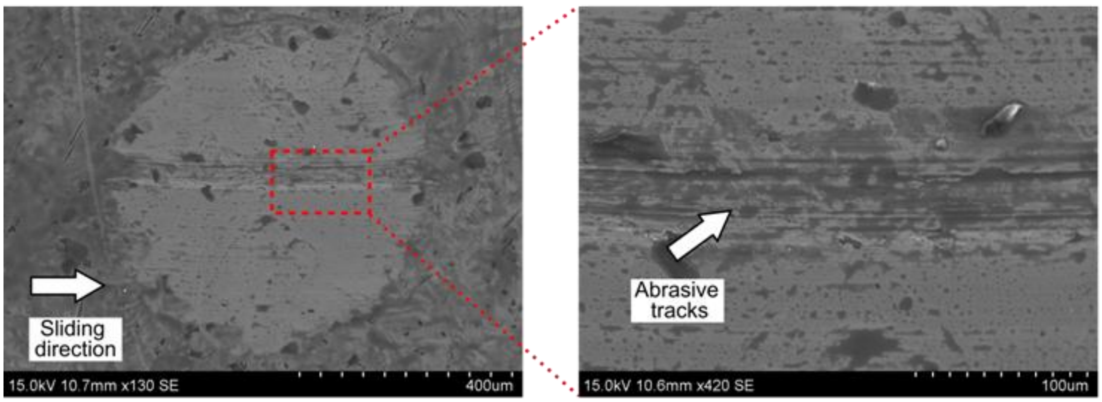


Figure 19: Morphology of wear scar obtained by TBHQ at 1200 RPM.

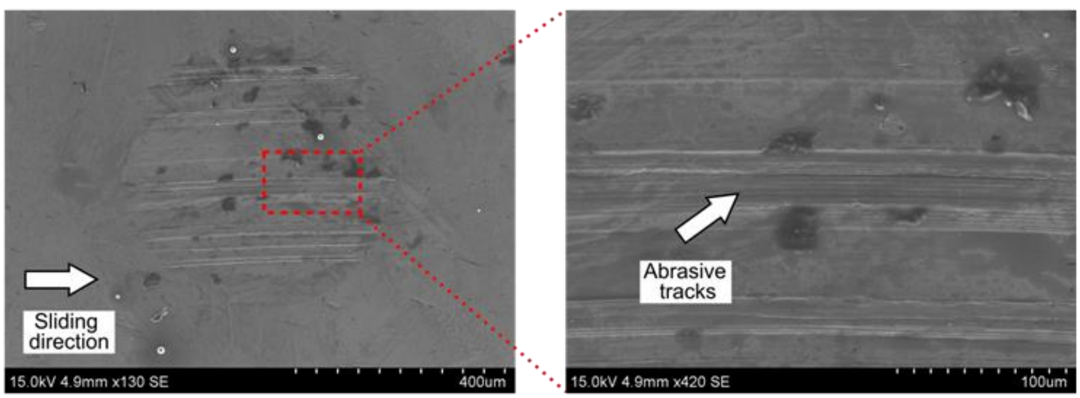


Figure 20: Morphology of wear scar obtained by TBHQ at 2400 RPM.

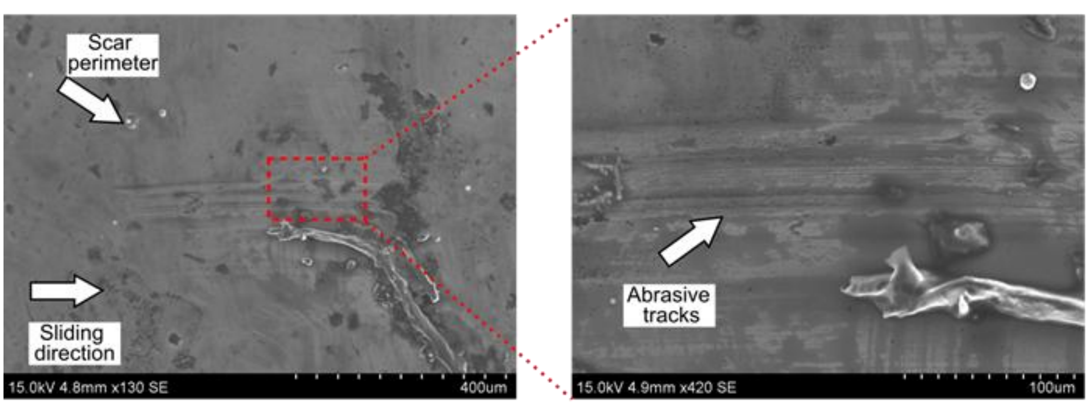


Figure 21: Morphology of wear scar obtained by BHA at 1200 RPM.

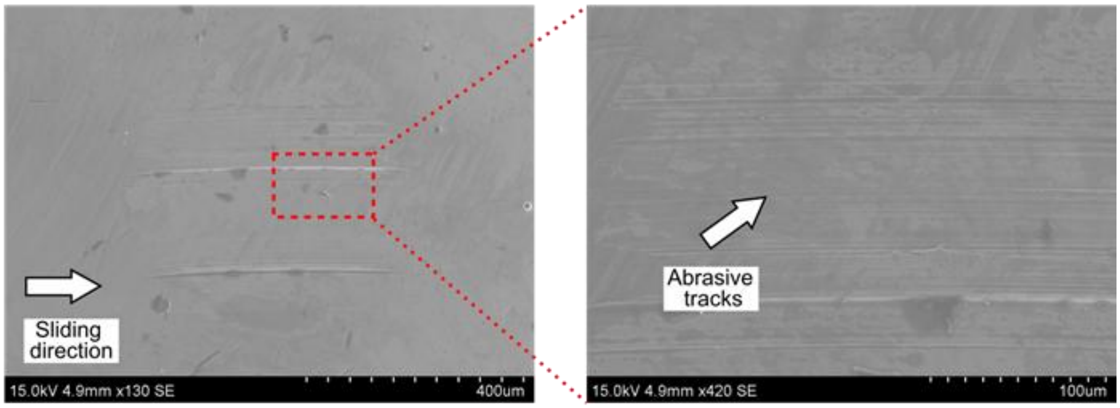


Figure 22: Morphology of wear scar obtained by BHA at 2400 RPM

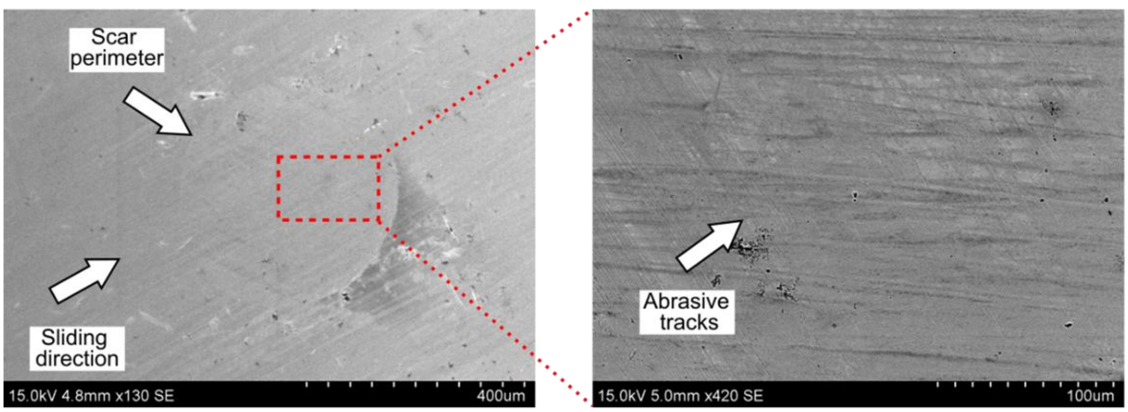


Figure 23: Morphology of wear scar obtained by BHT at 1200 RPM

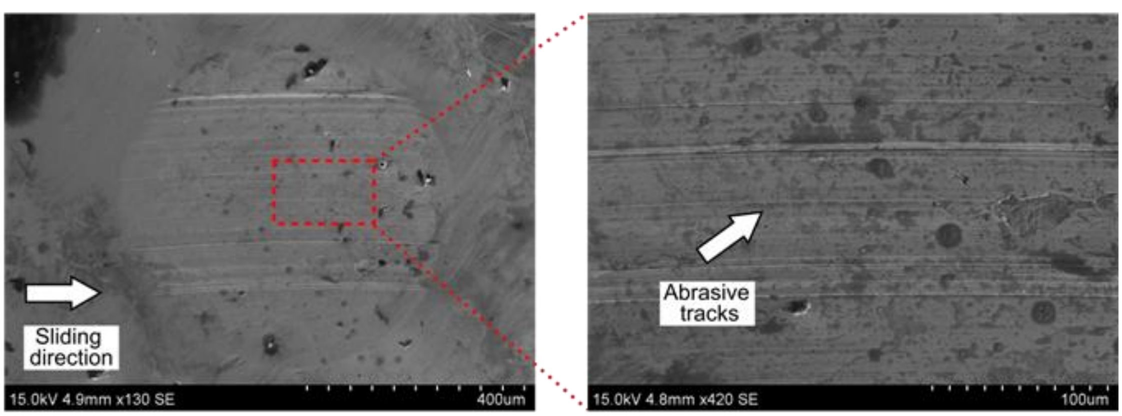


Figure 24: Morphology of wear scar obtained by BHT at 2400 RPM

3.6 Friction Behavior at Various Speed

Figure 25 show the dependence of COF to speed for all three antioxidants. Across the graph, BHA, BHT and TBHQ show similar trends, a steady decrease in COF, and especially the lowest at 2000rpm, before increasing at 2400rpm. On the other hand, the highest COF occur at speed 1200rpm. The optimum performance of the biolubricants occur at 2000rpm compared to the other speeds.

Changes in COF and wear performance directly affect the efficiency and lifespan of machinery in real-world applications using palm oil-based lubricants. A lower COF reduces friction, leading to improved energy efficiency and reduced heat generation, while better wear performance minimizes material degradation, extending the operational life of components. This makes palm oil-based lubricants more attractive for industries like automotive and heavy machinery, where both energy savings and durability are critical.

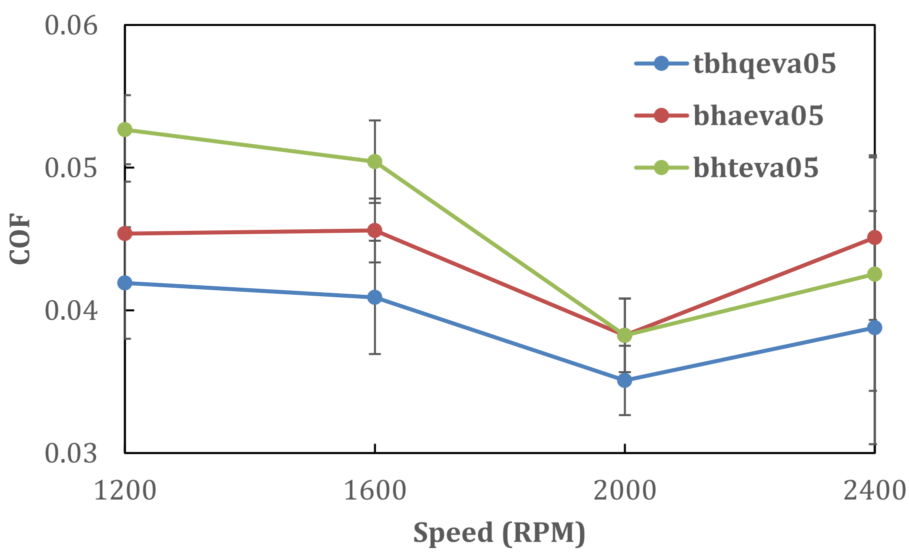


Figure 25: Graph of COF vs speed for all antioxidants.

CONCLUSIONS

Based on the discussion of oleic acid base stock with the addition of TBHQ, BHT, and BHA along with EVA, the following conclusions are yielded:

a. The addition of antioxidants provided relatively similar values of COF, although the performance yielded best using TBHQ.
b. Among the four speeds (1200 rpm, 1600 rpm, 2000 rpm, 2400 rpm), the best performance was achieved by fourball test using 2000rpm, 75°C, and 392N.
c. WSD for TBHQ remains one of the lowest across four speeds, followed by BHA and then lastly BHT.
d. In terms of cold climate performance, the order of best to worst performance of antioxidants are: TBHQ > BHT > BHA.

Due to financial constraints, future work could focus on comparing the biolubricant with commercial mineral oil to broaden the scope of comparison. Additionally, experimenting with varying concentrations of antioxidants, rather than keeping them constant, may provide further insights. Conducting TGA analysis would also be beneficial for assessing weight loss in the oil after heating with antioxidants. Performing DSC in an oxygen environment instead of nitrogen could reveal any differences attributable to the gas used. Finally, a more detailed analysis incorporating SEM and EDX imaging would enhance the understanding of surface characteristics and elemental composition.

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