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TRIBOLOGICAL AND THERMAL CHARACTERISATION OF PALM GREASE WITH ORGANIC THICKENER

MUHAMMAD FAKHRUR RAZZI ZULKIFLI[1]; NURUL FARHANA MOHD YUSOF[1]*; NADIAH AQILAHWATI ABDULLAH[1] and ZAIDI MOHD RIPIN[1]

ABSTRACT

Grease is widely utilised in machinery to provide essential lubrication during component contact. However, the concern regarding the toxicity linked with mineral-based grease has attracted notable attention. With a thickener content of up to 30%, the properties of the thickener can likewise impact the characteristics of the grease. Organic or green thickeners have gained significant attention from researchers due to their biodegradability and environmentally friendly properties. In this study, a refined, bleached and deodorised (RBD) palm olein grease was formulated utilising a cellulose and chitosan organic thickener and subsequently, its tribological and thermal properties were assessed. The palm grease formulated with organic thickeners exhibited a lower coefficient of friction than lithium-based and food-grade grease. Mass loss analysis of the pin indicated comparable values among all grease samples. The grease containing organic thickener exhibited a shallower wear groove depth on the plate with white particles, resulting from elevated levels of carbon and oxygen attributed to the organic thickener material. Thermal analysis indicated good thermal stability of the organic thickener grease. The finding discovered the potential of organic thickener formulated with RBD palm olein as a sustainable grease.

Keywords: grease, organic thickener, palm oil.

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INTRODUCTION

Grease plays a vital role in ensuring the smooth functioning of machinery components by providing essential lubrication to the components in contact. Typically derived from mineral or synthetic oils, conventional grease has been a staple in various industries. However, it is worth noting that the constituents of conventional grease, while effective, can sometimes pose toxicity concerns. Recently, major concerns about grease toxicity in industrial applications, particularly in food processing, agriculture and manufacturing sectors, have led to an increase in the number of research and development efforts focused on green lubricants.

[1] School of Mechanical Engineering,
Universiti Sains Malaysia,
14300 Nibong Tebal, Pulau Pinang, Malaysia.

* *Corresponding author e-mail: mefarhana@usm.my*

Green lubricants, also known as environmentally friendly or sustainable lubricants, are designed to address the environmental and health issues associated with traditional lubricants, including their potential toxicity and negative impact on users, labour and ecosystems. Previous work (Barboza *et al.*, 2023) found that the use of epoxidised gingelly methyl ester with 1.00% w/w alumina nanoparticle biolubricant leads to an 8.64% improvement in brake thermal efficiency compared to the baseline operation using B20 biodiesel fuel-mineral lubricant. In addition, significant decreases in emissions had been identified with reductions in the percentage of CO, NOx and HC concentrations, as well as smoke opacity, when compared to the baseline operation for the mentioned combination. Through a combination of biodegradability and reduced toxicity, green lubricants aim to lessen the harm caused by lubricant residues that might find their way into processed food in machinery and finally safe disposal into soil and water systems.

One approach to produce a green lubricant involves substituting mineral oil-based substances or materials with environmentally friendly alternatives such as vegetable oils. Several research have evaluated vegetable-based oil performance including their rheological, tribological and thermal characteristics (Hamnas *et al.,* 2023). Grease generally is a semi-solid lubricant that contains 70%-90% base oil, 10%-30% thickener and 5%-10% additives (Japar *et al.*, 2019). Therefore, the portion of the thickener also played a crucial role in producing a safe and environmentally friendly lubricant. Organic or green thickeners have gained significant attention from researchers due to their biodegradability and environmentally friendly properties. Several works have been done to evaluate the performance of vegetable oil-based grease, such as castor oil (Ahme *et al.*, 2023; García-Zapateiro *et al.,* 2014; Japar *et al.*, 2019; Sanchez *et al.,* 2011; Vafaei *et al.,* 2022), sunflower oil (García-Zapateiro *et al.,* 2014), karanja oil (Panchal *et al.,* 2015), soybean oil (Sanchez *et al.*, 2011; Saxena *et al.,* 2021) and palm oil (Muhammad *et al.*, 2023; Yap *et al.,* 2022), however there is very limited literature available on the topic of green thickeners. Sánchez *et al.* (2011) formulated grease using chitosan, chitin and acylated derivatives as organic thickener agents while castor oil and soybean oil as base oil and found that the greases formulated with the organic thickeners oleogel exhibited a comparable coefficient of friction value with standard lithium greases, however, these greases generally display quite poor mechanical stability. The authors observed that, as the thickener concentration increases, the NLGI grade tends to rise, whereas the use of soybean oil instead of castor oil in the formulation leads to a decrease in the NLGI grade. It was observed that 23% of chitin, 25% and 27% of chitosan with castor oil exhibit NLGL greater than 1. Another work by Japar *et al.* (2019) used polypropylene, chitosan and chitin thickener with a percentage range of 25% - 35% in the formulation and observed that the formulation produced greases with NLGI 000 to 1. It was found that these formulated greases could be used for low-temperature applications only as the greases start to lose their stability at high temperatures. Sanchez *et al.* (2014) observed that the formulated acylated chitosan-thickened grease poses better thermal resistance than lithium and calcium greases with good mechanical stability behaviour. However, the friction coefficient at low speeds was slightly higher than the others and had larger wear marks. In the study by Acar *et al.* (2018), biodegradable greases were developed using sunflower oil and castor oil with various thickeners including natural cellulose fibre and traditional lithium hydro stearate. It was observed that the biodegradable grease from cellulose fibre posed a lower friction coefficient and wear volume. Abdulbari *et al.* (2011) used spent bleaching earth obtained from natural clay as a thickening agent in grease formulation and found that the addition of a fumed silica additive is required to increase the penetration number of the grease to NLGI 1.

With the limited literature available regarding the advantages of green thickeners, the unexplored potential of utilising such thickeners in the creation of biodegradable and environmentally sustainable green grease remains to be investigated. This study aims to pioneer sustainable alternatives that reduce environmental impact while maintaining or even enhancing performance levels. Through innovative research and development in this field, it is expected to improve lubrication practices, setting new standards for eco-friendly, high-performance lubricants that align with our global commitment to a greener environment. In this work, the palm olein grease is developed utilising organic thickeners, and the tribological and thermal performance of the resulting grease in comparison to both conventional grease and thickeners were accessed. Through this endeavour, it is anticipated that the produced grease will contribute to secure disposal practices of grease waste in the environment, while also ensuring the safe process of food machinery and workers while handling the machine.

MATERIALS AND METHODS

Grease Preparation

In this study, the grease is developed by using refined, bleached and deodorized (RBD) palm olein as the base oil and added with three different thickeners which were cellulose microcrystalline powder 20 µm $(C_6H_{10}O_5)$n, chitosan (Deacetylated chitin, Poly(D-glucosamine)) and lithium hydrostearate. Chitosan and cellulose were categorised as organic thickeners, whereas lithium is a widely used conventional inorganic thickener with improved superior performance (Zhou *et al.*, 2022). The RBD palm olein viscosity at 40°C and 100°C is 46.76 mm^2/s and 8.91 mm^2/s respectively. The performance of the developed greases was then compared with commercial food-grade grease available in the market as a benchmarking performance for the greases. The greases were prepared following the sets of compositions as shown in *Table 1*. The composition was selected based on previous work of producing greases with a green thickener (Japar *et al.,* 2019; Muhammad *et al.*, 2023) with the aim of producing NLGI grade 1 to grade 2 grease. These grades are widely used as multipurpose grease applied in machinery. For a set of 100 g final weights of grease using lithium thickener, 87 wt.% of palm olein (87 g) and

13 wt.% of lithium thickener (13 g) were needed. Meanwhile, the organic grease requires higher thickener compositions as stated in *Table 1*.

Firstly, the beaker was placed on a hotplate and heated above the water's boiling temperature for about 10 min to clear all the moisture content in the base oil. Subsequently, the thickener was added to the base oil. The hotplate temperature was adjusted accordingly, either raised or lowered, based on the thickener's melting point. The solution was then stirred for 10 min to completely dissolve the thickener. Then, the remaining base oil was poured into the mixture to cool it down. The mixture temperature was slowly reduced and the stirring process was continued for 30 min for homogenisation.

A penetration test was performed according to ASTM D217 standard to evaluate the NLGI grade consistency of the developed grease. The HK-2020 cone and needle penetrometer were used. Before the test, the grease underwent 60 double strokes using an HK-269G mechanical grease worker machine. A stainless steel-tipped brass cone, weighing 102.5 grams, was used to penetrate the grease for 5 s. From the test, the lithium grease penetration value was in the range of 265 to 295 dmm which was under NLGI grade 2 consistency.

Tribological Testing

The tribological test for each grease was performed using a pin-on-plate tribometer machine (DUCOM TR-20). Before testing, the pin and plates were cleaned using acetone to remove all the unwanted material that was deposited on the samples. During the test, the pin was fixed to the machine holder while the plate was undergoing reciprocating motion continuously at a specific time as shown in *Figure 1*. The normal load during the test was set to 98.1 N by applying a 10 kg mass of weight. The test was run at room temperature conditions with the 100-rpm speed of the reciprocating plate for 60 min.

Figure 1. Pin-on-plate experiment set up.

Both pin and plate were fabricated using a mild steel material as shown in *Figure 2*. The pins have a hemispherical shape with a 12 mm diameter and a 25 mm cylindrical body length. The plates were machined in cuboid shape with the dimensions of 100 × 28 × 6 mm. All the pins and plates were fabricated according to the ASTM G99 for the standard pin-on-plate test.

TABLE 1. COMPOSITIONS OF THE GREASE FORMULATIONS

Thickener agent	Thickener concentration (%)	Base oil	Name	Type of thickener
Cellulose	30	Palm olein	POCl	Organic
Chitosan	30	Palm olein	POCt	Organic
Lithium	13	Palm olein	POLi	Inorganic
Food grade grease	unknown	unknown	FG	unknown

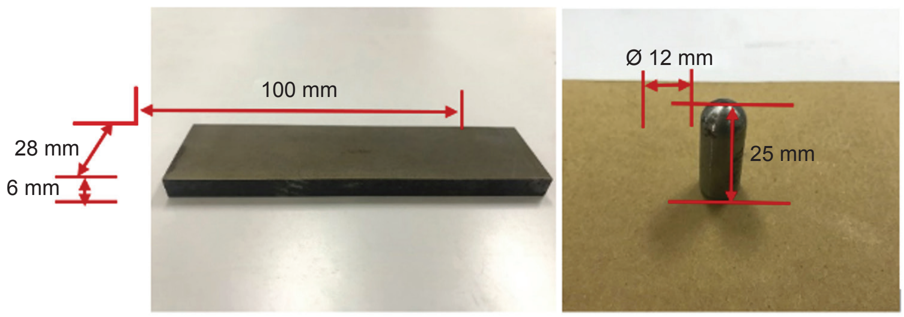

Figure 2. Plate and pin used in testing.

Surface Characterisation

Surface characterisation was performed by using Hitachi S3400N Scanning Electron Microscope and Zeiss LSCM 700 Laser Scanning Confocal Microscope. Using this technique, the surface micrograph, morphology and wear profile of the tested pin and plate were evaluated. Elemental analysis was carried out using Energy-dispersive X-ray (EDX) analysis to further evaluate the material elements in the surface micrograph.

Thermal Analysis

Thermal analysis was performed to evaluate the thermal resistance of the grease before undergoing degradation. The grease's thermal resistance was explored through thermogravimetric analysis, which provides insights into the thermal decomposition of the utilised materials. The interception between the temperature at which the grease starts to degrade and the tangent of the curve is denoted by T_{onset} while T_{max} is the temperature of the grease when there is a rapid change in mass (from the temperature derivative graph). T_{offset} is the interception between the temperature of grease at which the mass does not change as the process continues and the tangent of the curve. These values were taken from the weight against the temperature graph. The thermogravimetric analysis was performed with a thermogravimetric Analyzer Pyris 1 (Perkin Elmer) where each sample was weighed at around 10 mg (±5 mg), and the temperature was scanned from 25°C to 700°C at a heating rate of 10°C min^{-1}. TGA was performed by placing the substance in platinum crucibles under the nitrogen flow of 100 mL min^{-1}.

RESULTS AND DISCUSSION

Grease Characteristics

The physical appearance of palm grease with thickeners is shown in *Figure 3*. From *Figure 3a*, it can be seen that the grease formulated with cellulose thickener (POCl) has a brownish-cream colour. The POCl grease also has a soft like ketchup consistency, providing relatively low viscosity. The physical appearance of palm grease with chitosan thickener (POCt) is shown in *Figure 3b*. It is observed that the completed POCt grease has a brownish-cream colour with a soft texture. From *Figure 3c* the formulated POLi grease turns into a soft white cream with colour. The POLi grease also has a soft semi-solid consistency with relatively high viscosity. From visual inspection, the POLi grease also has high or uniform homogeneity compared to other greases. Previous work employing chiton, chitosan-based thickeners exhibited values of COF comparable to standard lithium greases, however, these greases generally display quite poor mechanical stability (Sanchez *et al.*, 2011).

Tribological Properties

Coefficient of friction. *Figure 4* shows the specific average of the coefficient of friction (COF) obtained from the pin-on-disc test lubricated with various greases. Grease with organic thickener (POCl and POCt) recorded the lowest COF value at 0.0840 followed by POLi and FG with their corresponding value of 0.1015 and 0.1250 respectively. Theoretically, a higher COF value means that higher force is required to initiate and maintain the motion between the surfaces thus increasing the resistance while making it harder to move (Zeren *et al.*, 2007). The coefficient of friction obtained in this study shows that the newly developed greases using organic substances which are cellulose and chitosan thickeners give a promising result as compared to the others. This result indicates that organic thickeners used in this experiment pose excellent friction and better performance than the lithium and food-grade grease in terms of COF.

Pin wear mass loss analysis. The mass of the pin subjected to contact during the pin-on-disc test was measured before and after testing to evaluate the amount of material removed after the pin and

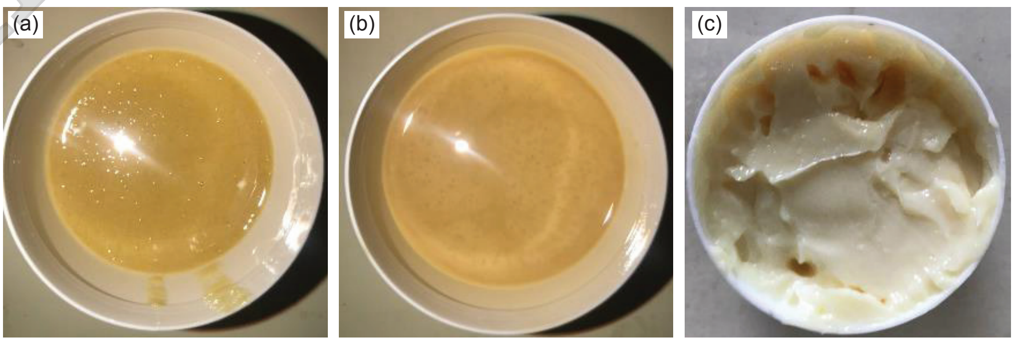

Figure 3. Palm grease developed with (a) cellulose, (b) chitosan and (c) lithium thickener.

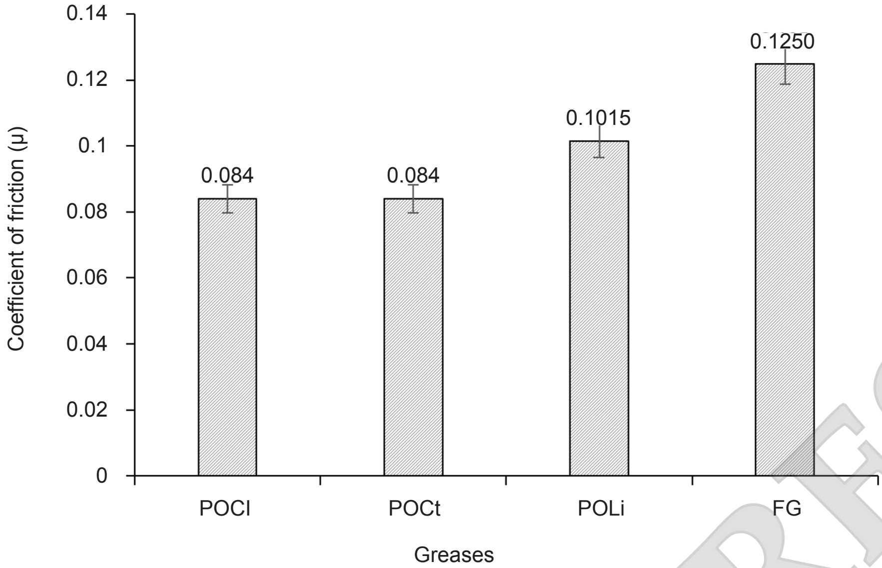

Figure 4. Coefficient of friction for all greases.

plate sliding contact. *Figure 5* shows the pin mass loss analysis for all greases. Overall, all pins show a minuscule and almost similar mass loss percentage in a range of 2 to 6 x10^{-5} %. The pin lubricated with POCl and POCt has a slightly higher mass loss percentage with a value of 6.1388 x10^{-5} % and 6.1388 x10^{-5} % respectively. FG grease shows the lowest mass loss with a value of 2.6486 x10^{-5} % followed by POLi with 4.6158 x10^{-5} % mass reduction. This finding shows that the formulated palm olein grease with organic thickener shows comparable properties compared to the conventional lithium thickener and commercial food grade grease. Lithium thickener has been proven to have superior performance in mineral grease application (Nasikin *et al.*, 2009; Zhou *et al.*, 2022). Meanwhile, the food grade grease (FG) has been commercially used and the properties have been enhanced with additives by the manufacturer.

Plate wear groove depth analysis. *Figure 6* shows the three-dimensional (3D) morphology of the plate groove formed during the pin-on-plate test and the related c value is shown in *Figure 7*. From the figure, the POCl and POCt grease show almost identical surface topography with the lowest wear depth of 13.7 μm. Most of the groove regions in POCl and POCt are in light blue colour which indicates that it has lower depth as compared to POLI and FG greases. The highest groove wear depth occurs on the FG grease plate with a value of 16.1 μm. The dark blue colour on the height map shows the highest depth region while the red one indicates the upper surface of the plate. The high depth indicates that more material was removed during the contact. POLi grease shows a slightly lower depth groove formed on the surface with a depth of 15.6 μm. The deep groove formed on the plate surface indicates the lubricant failure in protecting the surface from wear. This finding shows that the developed palm grease with organic thickener can protect the surface from wear by forming a shallower groove depth. In addition, the COF of the contact lubricated by these lubricants are shown to be low in *Figure 4*. This shows the good agreement between friction and wear analysis where low COF during contact causes less wear or material removal.

Surface characterisation. The SEM micrographs of the plates are shown in *Figure 8. Figure 8a* displays the initial plate surface condition before contact as a reference and *Figure 8b - 8d* show the images of the samples after being subjected to sliding contact applied with different greases. The image on the left displays the surface micrograph at 100x and the right side displays the enlarged micrograph image at 500x magnification. The yellow arrow shows the movement of the sliding plate during the test. The wear groove produced on the plate is highlighted in the rectangular area. For the POCl plate in *Figure 8b,* the groove marks can be seen at the area of contact with the pin. A brighter colour is observed at the area where a groove formed on the plate for all the samples after the test. Apart from groove formation, we can see that the area which is in contact with the pin is smoother than the other part of the plate as expected due to surface smoothening by sliding of the surface asperities (Mohd Yusof and Ripin, 2014; 2016). This deformation can be attributed to the contact stress developed during the surface contact (Adman and Mohd Yusof, 2019). The enlarged image

Samples	Mass before (g)	Mass after (g)	Total mass loss (g)	Reduction (x10^{-5} %)
POLi	25.9978	25.9966	0.00113	4.6158
POCl	26.0637	26.0621	0.00160	6.1388
POCt	26.0433	26.0416	0.00170	6.5275
FG	26.0429	26.0422	0.00070	2.6486

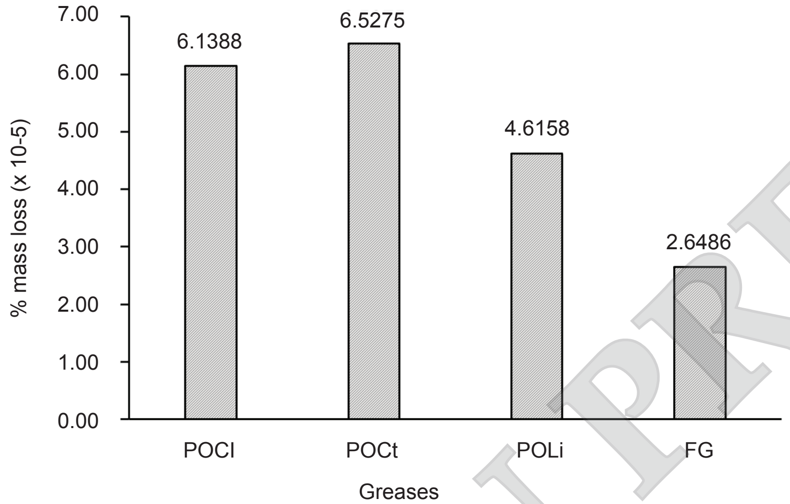

Figure 5. Percentage of mass loss of the pin for all greases.

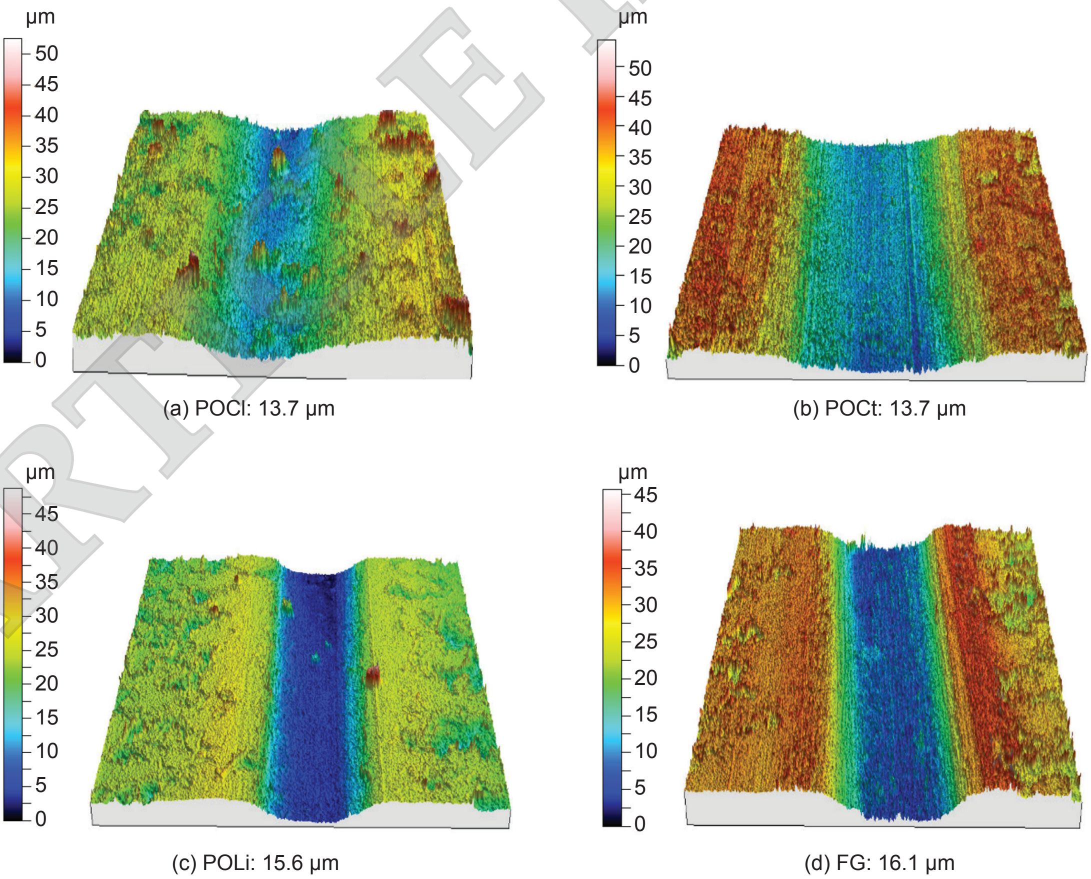

Figure 6. 3D surface topography of the plate groove for (a) POCl, (b) POCt, (c) POLi and (d) FG greases.

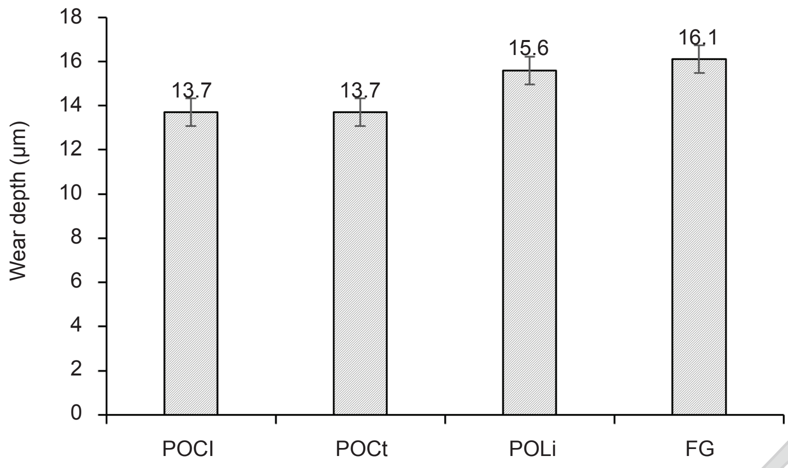

Figure 7. Groove wear depth.

in *Figure 8b* shows the presence of white particles deposited on the surface of the plate. It is expected that these particles are from cellulose thickener that protects the plate surface by acting as a pressure absorber hence reducing the groove wear depth produced on the plate. According to Kumar Gupta *et al.* (2019) cellulose is a complex polysaccharide composed of repeating units of glucose molecule with the material composition mainly consisting of carbon, hydrogen and oxygen atoms arranged in a specific molecular structure. This was also observed by Akram Hussein *et al.* (2019) where some portion of the pressure was carried by fine particles and reduced adhesion wear. This could be a reason why there is no distinct groove formed on the plate with the lubrication using POCl grease. *Figure 8c* shows the image micrograph for the POCt sample. The enlarged image shows medium-sized white particles presumably originating from the chitosan thickener. Chitosan is a naturally occurring polysaccharide derived from chitin, which is found in the exoskeletons of crustaceans such as shrimp, crabs and lobsters with the material composition of carbon, hydrogen, oxygen and nitrogen atoms, with a specific arrangement (Ramawat and Mérillon, 2015). Elemental analysis of both white particle substances indicates the presence of a high quantity of carbon and oxygen elements, hence resulting in a high percentage of carbon and oxygen detected on the surface.

Figure 8d shows the groove formation on the surface of the plate using POLi grease lubrication. It is observed that the sliding movement of the pin has generated a significant wear depth on the plate and initiation of micro crack. Based on the previous surface topography analysis in *Figure 6*, this image is taken at the groove depth of 15.6 µm. It is expected that at this depth, continuous sliding contact has started to disrupt the materials by crack initiation and severe material loss. *Figure 8e* shows the groove formation on the surface of the plate using FG grease lubrication. The sliding marks are obvious and the enlarged image shows that the surface starts to form a lot of microcracks at the groove depth of 16.1 µm. In addition, it is observed that white colour tiny particles are present, expected from the additive that is used in the food grade grease. Further elemental analysis revealed the presence of silicon in the area of the white particles. Based on existing literature, the silicon derivative is incorporated into the material composition as an additive during the grease preparation process, fulfilling its role in enhancing the grease's properties such as increasing thermal resistance (Liang and Ji, 2022).

Thermal analysis. *Figure 9* shows thermogravimetric analysis curves in terms of weight against temperature for all greases and *Table 2* shows the related temperature. It is observed that the *Tonset* of the grease that is made of organic thickeners (POCl and POCt) is slightly lower than grease using lithium thickeners which is within the range of 370.00°C-390.00°C. FG sample shows the lowest T_{onset} around 338.31°C which means that the grease starts to degrade earlier than the others. The T_{onset} is often used as an indicator of the thermal stability of a material. A lower onset temperature suggests that the material cannot withstand higher temperatures and undergo significant decomposition or weight loss early. In the previous work, Sanchez *et al.* (2014) observed that the formulated acylated chitosan displayed better thermal resistance than the lithium and calcium thickened grease.

The decomposition temperature range for both POCl and POCt are 264.08°C-544.66°C and 253.77°C-543.22°C respectively. These ranges are close to the grease made from lithium thickeners (POLi). In addition, three of them

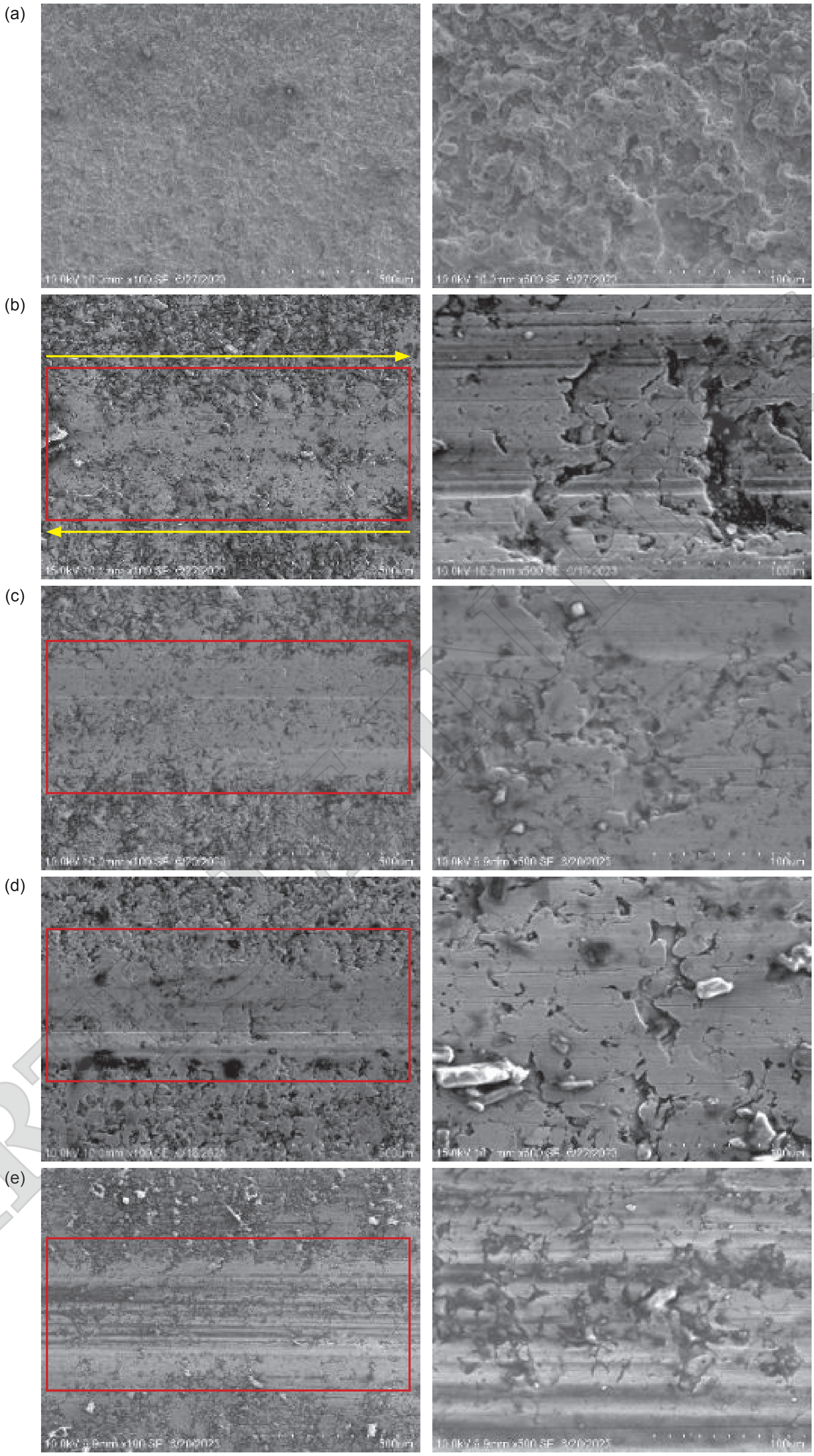

Figure 8. (a) New, (b) cellulose, (c) chitosan, (d) lithium and (e) food grade grease.

also show the widest range of T_{onset} and T_{offset} as compared to FG grease. Thermal characteristics for both organic thickeners show a slightly similar behaviour in which their decomposition range is in between 250.00°C - 545.00°C. Besides, the thermal decomposition of greases that contain cellulose and chitosan also exhibits a two-stage process, whereas the other two greases undergo thermal decomposition in a single stage. Based on the existing literature, in the case of organic thickeners, the derivative function of weight loss against temperature reveals the presence of two distinct peaks, which indicates the thermal decomposition process occurs in two stages. According to Sánchez *et al.* (2011), the first stage of decomposition-belonged to the degradation of the thickener itself while the second one is associated with the thermal degradation of the base oil (palm olein).

Implications and Limitations of Study

The study of green lubricants holds significant implications for both environmental sustainability and technological advancement in renewable energy. Based on the findings of this study, the grease performance in the aspect of tribological and thermal characteristics shows the potential of grease as an alternative to conventional mineral-based lubricants. This supports the need for a safe and sustainable lubricant, consequently leading to a decrease in pollution and ensuring a safe working environment. While the findings of this study focused on the performance of the developed grease, further work needs to be done to evaluate the level of biodegradability of the developed grease. This requires a commitment from multidiscipline studies with the aspiration to create lubricants of exceptional performance and unwavering reliability.

TABLE 2. THERMOGRAVIMETRIC ANALYSIS OF TEMPERATURES

Item	T_{onset} (°C)	T_{max} (°C)	T_{offset} (°C)	Decomposition range (°C)
POCl	387.38	429.68	448.04	264.08-544.66
POCt	378.83	435.80	454.09	253.77-543.22
POLi	395.78	429.64	454.88	274.50-540.27
FG	338.31	400.55	418.90	228.04-462.66

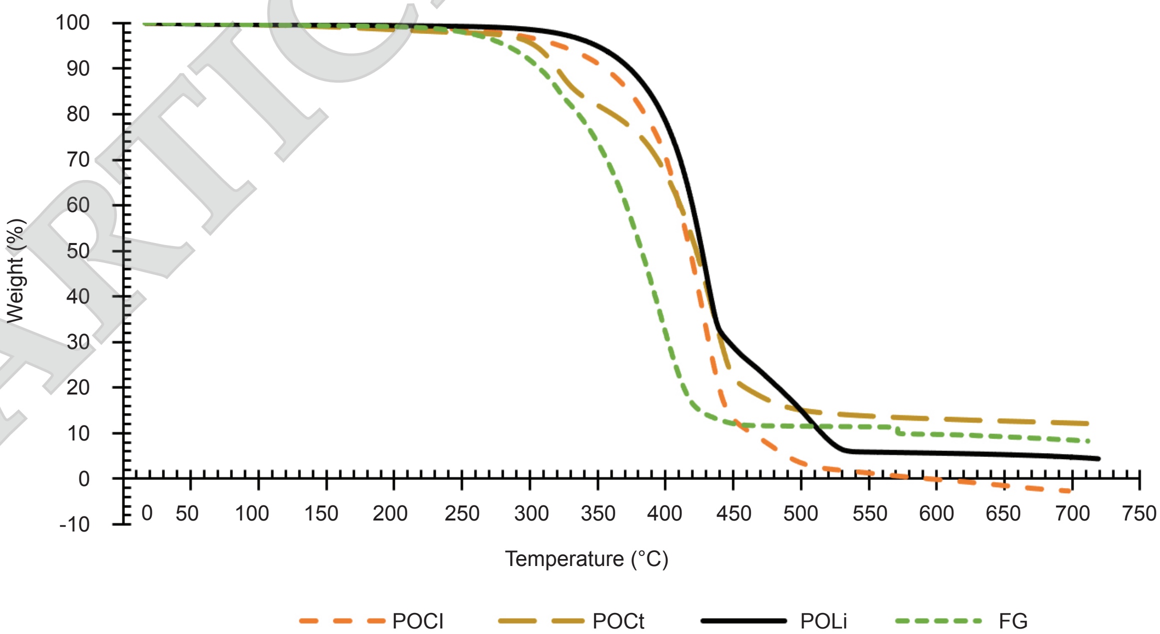

Figure 9. Weight (%) as a function of temperature.

CONCLUSION

This study revealed that the palm grease formulated with organic thickeners exhibits a lower coefficient of friction (0.0840) in comparison to both lithium-based (0.1015) and food-grade grease (0.1250). Mass loss analysis of the pin indicates comparable mass loss among all grease samples in a range of 2 to 6 x10^{-5} %. The grease containing organic thickener exhibits a shallower wear groove depth on the plate with the existence of white particles. Elemental analysis indicated that the particles result from elevated levels of carbon and oxygen which is attributed to the organic thickener material. The grease formulated with organic thickeners exhibits a slightly lower onset temperature (378.83°C) compared to the grease containing lithium thickeners (395.78°C), yet superior to the food grade grease (338.31°C), indicating good thermal stability. The finding indicated that RBD palm olein greases formulated with organic thickeners demonstrate positive performance attributes, in terms of coefficient of friction, groove wear depth, and thermal stability.

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