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RESEARCH

Tribological Behavior of Calcium Complex Palm-Biogrease with Green Additives

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A B S T R A C T

Vegetable oils have been acknowledged to have significant potential as a substitute base fluid in grease formulation. The so-called biogreases are demanded especially in open applications where the greases are in contact with soil and water and lost to the environment. In this paper, formulation of a biogrease derived from palm-based ester as the base fluid and calcium complex soap as the thickener is discussed, with the main purpose to explore the potential of palm oil as an effective biodegradable fluid, and to investigate the possibility to improve the tribological properties by mixing calcium carbonate ($CaCO_3$) and hydroxyapatite (HA) as green additives. The wear and friction properties as well as the load carrying capacity of the formulated bio-grease were extensively studied through the four-ball wear tester. The experimental results show that the average coefficient of friction of the palm based biogrease was improved by about 6.5% when 5% of $CaCO_3$ and 3% of HA were added comparing to base grease. Besides, the load carrying capacity was also enhanced significantly by the addition of the additives. This demonstrated the good potential of palm ester as base fluid for grease and the $CaCO_3$ and HA as lubricant additives, exhibited the good anti-friction and load carrying abilities.

1. INTRODUCTION

The development of bio-based lubricants has become a growing interest among industrial players nowadays have attributed to the need for alternative environmentally friendly materials to replace crude mineral oils. The depletion of mineral oil reserves, as well as the concern for the safety of the environmental, was the main motivation for the efforts. The bio-based lubricants are basically derived from living materials mainly plant oils and animal fats. Not only environmentally benign, the bio-based lubricants are also biodegradable, cost-effective, renewable, non-toxic, and not bio-accumulative [1]. The biodegradable capacity of a material describes the degree it can disintegrated by microorganisms down to the base substances. According to Florea et al. [2], mineral oil has a biodegradability range between 20-40%, whereas synthetic esters and vegetable oils have a range between 60-100% and 90-98% respectively. The utilization of vegetable oils as bio-based lubricant, either edible or non-edible, had been successfully practiced globally. This is in parallel with the positive growth of vegetable oil's global production due to agricultural advancement [3]. The triglycerides structure of the vegetable oils, with high viscosity and viscosity index and flash point, make it the best alternative to mineral oil [4]. The excellent lubricating properties also have been proven in past studies, whether without or with presence of dedicated additives [5-8].

Semi-solid lubricant or grease is an ideal preference in a component where the lubricant needs to remain longer without drain or leak out under gravity or centrifugal action. The bio-based grease is formulated from bio-based raw materials especially plant origin base oils, which are thickened by the environmentally acceptable thickeners and added with performance additives that should contain none or fewer heavy metal and chlorinated additives. According to Nagendramma and Kumar [9], since base oil is a key ingredient of grease that contains at least 80% of the total constituent, it reflects the biodegradability of grease. However, Florea et al. [2] claimed that the thickener and additive also have an impact on the grease biodegradability.

Biogrease formulation and development utilizing vegetable oils as the base oil became one of the research interest topics that have been discussed in current studies, emphasizing on the tribological and physicochemical properties. According to Adhvaryu et al. [10], the formulation of a lubricating grease is a complicated trial-and-error process, in which the optimization of the reactants and reaction protocol is critical in order to attain the desired grease consistency. An optimum amount of metal, fatty acid, and base oil in metal soap grease formulation is significant in producing a good thermo-oxidative stability grease with the desired consistency. The authors used soybean oil in the formulation of the lithium grease and observed that the length of the fatty acid affects the grease hardness, which influences the physical performance properties including viscosity, boundary lubrication, and rheological behaviour. Sharma et al. [11] extended the study and revealed that the effectiveness of the formulated lithium biogrease with epoxidized soybean oil and several additives exhibited excellent oxidative stability with lower friction and wear, compared to the commercial greases.

In another study, Panchal et al. [12] formulated karanja oil-based lithium grease and compared the performance of the different fatty acid esters that were chemically modified with three different alcohols: hexanol, octanol, and neopentyl glycol. The study proved the competitive physicochemical and tribological properties of the formulated bio-based grease are comparable to mineral-based grease, with great improvement of load-carrying capacity. Focusing on biodegradability concern, Acar et al. [13] formulated the completely biogenic lubricating greases with high-oleic sunflower oil (HOSO) and/or castor oil and different types of biodegradable thickener agents. The extensive study investigated the tribological and rheological properties of the formulated greases, showed the lower coefficient of friction produced by the biodegradable model greases than the reference greases. Utilizing special bio-additives, Padgurskas et al. [14] investigated the tribological performance of rapeseed oil- and lard-based greases with lithium and sodium soap thickeners suspended in ethanol. The study found that the wear resistance of the rapeseed oil greases with additive which formed the wear-protective layer on the contact surfaces improved about 40-50%. However, the additive was less efficient in lard-based greases.

The additives chosen for a lubricant varied depending on the specific properties to be enhanced or improved. Oil and grease utilize similar additives except for solid additives that are mainly preferred for greases to enhance the load-carrying capacity. Unfortunately, most lubricant additives generally are organic compounds containing reactive elements such as chlorine, sulphur, phosphorous atoms, or heavy metals that cause potential traits to the environment [15]. Hence, green additives like calcium carbonate ($CaCO_3$) that are stable, non-toxic, and safe should be preferred. In a study carried by Ji et al. [15], the effectiveness of the $CaCO_3$ in lithium grease was proven. The $CaCO_3$ nanoparticles, with 5 wt% optimum compositions, formed a boundary film which deposited on the rubbing surfaces and facilitates the reduction of friction and wear, also improved the non-seizure load by 15%. Using the same additive, Zhang et al. [16] revealed the potential of the $CaCO_3$ nanoparticles in poly-alpha-olefin (PAO) in improving the lubricant's load-carrying capacity, anti-wear, and friction-reduction properties. Another potential food-grade additive is hydroxyapatite (HA) which is a calcium phosphate ceramic that has similar chemical and mechanical properties to the hard tissues' mineral components such as bone and tooth [17]. Besides from non-toxic and bio-compatible, it is also the most thermodynamically and chemically stable calcium phosphate [18]. According to Waynick [19], a combination of carbonates and phosphates additive packages may attain superior extreme pressure properties and anti-wear qualities. In addition, both additives are also low cost, less toxic, safe, stable, water-insoluble, non-corrode, and unreactive to ferrous and non-ferrous metals even at very high temperatures. These types of additives are also preferred for their excellent heat and load resistance with long life at elevated temperatures.

In the present study, palm-ester trimethylolpropane (TMP) was used as the base fluid with calcium complex soap as a thickener and the addition of the anti-wear and extreme pressure additives; $CaCO_3$ and HA in the development of new renewable and biodegradable lubricating grease. Complex soap was chosen due to the improved properties for high operating temperature, dropping point, water-resistance, and load-carrying ability. The effects of the additives and the concentration on the tribological properties of the grease were also investigated.

2. EXPERIMENTAL DETAILS

2.1 Materials

A commercial trans-esterified ester of palm oil (PALMESTER 2090) that produced by KLK OLEO (Petaling Jaya, Malaysia) was used as the base fluid. The properties of the palm-ester used are listed in Table 1. The oil is C18-unsaturated, mixed esters with oleic acid and trimethylolpropane. It was claimed to be chemically stable at environment temperature, non-toxic, and does not have any harmful effect, as well as easily biodegradable and not hazardous to water. Calcium hydroxide ($Ca(OH)_2$), benzoic acid, and $CaCO_3$ were provided by Ungerer Australia Pty. Ltd., whereas the HA ($Ca_5(PO_4)_3(OH)$) was manufactured by Thermphos (Lianyugang) Food Ingredient Co., Ltd., Jiangsu, China. Two commercial greases (GC-I and GC-II) were used as reference grease in the study. Both greases are synthetic ester-based with solid lubricant content, particularly MoS_2 and/or graphite. GC-I is thickened by bentonite clay and has semi-fluid structure grease (NLGI 00) whereas GC-II has a soft structure (NLGI 1) and is composed of inorganic thickener. The n-heptane used for cleaning of steel balls for the four-ball testing were purchased from Evergreen Engineering & Resources (Semenyih, Selangor, Malaysia). All materials were used as received without any further purifications.

Table 1. Physical characteristics of the palm-ester.

Characterization	Test Method	Palm-ester 2090
Colour		Light yellow
Kinematic Viscosity @40°C [mm^2/s]	ASTM D445	76.6
Kinematic Viscosity @100°C [mm^2/s]	ASTM D445	14.95
Viscosity Index	ASTM D2270	149.8
Density at 15 °C [g/cm^3]	ASTM D1298	0.921
Pour point [°C]	ASTM D97	-48
Cloud point [°C]	ASTM D2500	-24
Flash point [°C]	ASTM D92	326
Acid value [mg KOH/g]	ASTM D1980	1
Saponification value [mg KOH/g]	ASTM D5558	197

2.2 Preparation of calcium complex grease

Complex grease is generally formed by reacting common base oil with two dissimilar organic acid compounds, such as a long-chain fatty acid and short-chain complexing acid. The calcium complex palm biogrease was prepared in a laboratory beaker with heating and stirring apparatus. The palm ester was first pre-heated to 60-70°C before stearic acid (the long-chain fatty acid; $C_{17}H_{35}COOH$) and boric acid (the short-chain complexing agent; C_6H_5COOH) with equivalent weight ratio were added. The mixture was heated continuously and stirred for about 15 minutes to ensure the acids were completely dissolved. The calcium hydroxide (taken in 1:0.75 ratio to stearic acid) was then added slowly. The thickened mixture was formed, and the temperature was slowly raised to a maximum of 120°C. As saponification began, the mixture started to boil, and water was release as the by-product reaction. Heating continued with high-speed stirring for 60 minutes to ensure complete reaction of the thickener. The mixture was then allowed to cool; and at 60°C, the $CaCO_3$ and HA additives were added. While maintaining the temperature at 60°C, the mixture was continuously stirred at high speed for homogenization for about 60 minutes before it was cooled to room temperature.

Five samples of the palm bio-grease formulations with various composition of palm ester, calcium complex thickener, and additives are listed in Table 2. The palm ester base oil compositions varied from 75% to 85% of the total product weight while the calcium complex soap thickener composition was 10-15 wt%. The ratio of thickener and base oil represents the different liquid (oil) content in the mixture to examine the effect of thickener composition on grease consistency. The grease sample PB-I is the plain grease without any additive, while the other four grease samples varied in terms of the additive concentration ranging from 8-15 wt% for both the $CaCO_3$ and HA. This is to investigate the influence of the different additive concentration in the formulated palm bio-grease.

Table 2. Composition of the palm bio-grease formulations.

Grease samples	**Grease composition (wt%)**				**Metal soap/oil ratio**
	Palm ester	Calcium complex soap	$CaCO_3$	HA	
PB-I	85	15	0	0	1:5.67
PB-II	80	12	5	3	1:6.70
PB-III	80	10	5	5	1:8.10
PB-IV	75	10	10	5	1:7.34
PB-V	75	10	5	10	1:7.34

2.3 NLGI consistency test

The grease rheology was measured by means of the consistency. It is the relative hardness of a grease determined by National Lubricating Grease Institute (NLGI) grade, in which the lower grade represents the softer grease. In this experiment, the NLGI grade of the formulated greases was determined by utilizing the SKF Grease Test Kit TKGT 1. Only a small amount of grease sample (about 0.5 grams) was required for each test, which is suitable for simple analysis purposes. The grease sample was placed in between two glass plates and spread using a weight for 15 sec. The consistency level of the grease was then determined according to the scale provided, where the larger spread indicates softer grease.

2.4 Tribological test

Wear preventive

The anti-wear properties of greases were evaluated by employing a four-ball wear tester (Koehler Instrument Company, Inc.), according to ASTM D2266 for evaluating wear-preventing characteristics under test conditions. The four-ball test set up includes the three 14.7 mm AISI 52100 steel balls (61 to 63 HRC) that clamped together in a pot. The fourth ball was secured on a rotating spindle on the top of the pot. During the operation, grease sample that contains in the pot was heated to 75°C, and the pot was pressed upward with a force of 40 kg against the top ball which rotated at 1200 rpm speed for 60 min. The friction force and the coefficient of friction during the test, were automatically recorded from the load cell connected to the stationary pot. Upon completion of the test, the wear scar on the three stationary balls due to the three-point contact with the top ball was observed using the Alicona 3D Surface Metrology System.

Extreme pressure

The extreme pressure test was carried out according to ASTM D2596, with the purpose to determine the load-carrying capacity of the greases and the relative ability to prevent wear under applied loads. Similar sample preparation procedure as in wear preventive test was used but only 10 seconds was taken for each test, with 1760 rpm speed at 30°C. The load was increased stepwise in every run with a new set of balls until the test balls are welded together or seizure. The final seizure load where the load of the four balls become welded to each other indicates the load-carrying capacity of the applied grease.

3. RESULTS AND DISCUSSION

3.1 Characterization of the $CaCO_3$ and HA solid additives

The crystal structure and phase purity of the $CaCO_3$ and HA particles used in this study were investigated by the X-ray powder diffraction (XRD) analysis. Fig. 1 exhibits the diffraction peaks of $CaCO_3$ which correlate well to the literature pattern (JCPDS Card No. 01-080-9776). The crystalline calcite appears at 2-theta values of 23.1°, 29.4°, 31.5°, 36.0°, and 39.5°, indicating the calcite crystal compositions. Whereas the XRD pattern of the HA shown in Fig. 2 is match well with the literature pattern (JCPDS Card No. 01-071-5048).

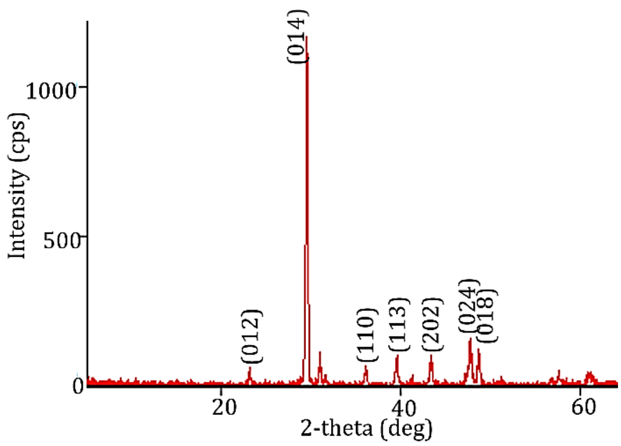

Fig. 1. XRD pattern of the $CaCO_3$

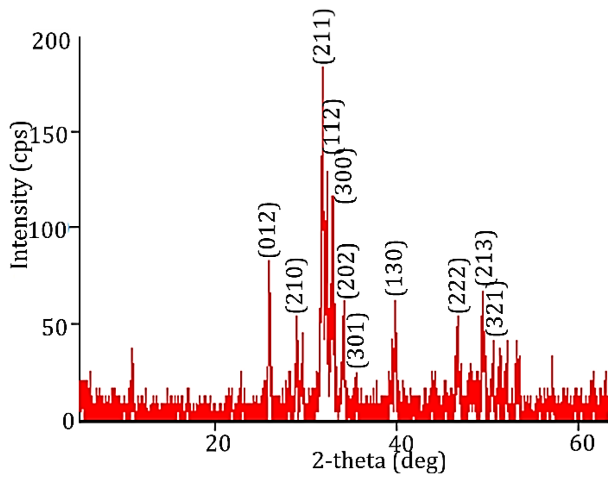

Fig. 2. XRD pattern of the HA

The major peaks of the HA pattern are mostly appearing between 2-theta 25° and 35°, which the sharper peaks positioned at 25.96°, 31.82°, 31.88°, and 32.94°, representing the good crystallinity. The pattern also corresponds with the HA patterns reported in [20-21]. These validates the purity of the $CaCO_3$ and HA particles used in this study.

3.2 Palm biogrease formulation

The preparation and formulation of an effective lubricating grease is a complex process where the mixing time and process temperature are the significant processing variables. The interactions among the base oil, thickener and additives also need to be considered. The present study applied a 1:0.75 ratio of metallic base to fatty acid, as recommended in Adhvaryu et al. [10] to achieve stable grease matrices. The optimum ratio, as claimed by the authors had better oxidative stability for the desired grease hardness. Furthermore, according to Sharma et al. [11], the stable grease matrix also depends on the selection of fatty acid, in which the fatty acid with longer chain lengths in the metal soap makes stronger interlocking fibres. The stearic acid (C18 chain length) therefore was used as fatty acid in the present study for the soap preparation. It was observed during the mixing process that an appropriate ratio of the calcium hydroxide, stearic acid, and benzoic acid is crucial to obtain a complete grease reaction. It is also known that vegetable oil, especially palm oil is very sensitive to high temperatures since the viscosity varies more with temperature [22]. Thus, keeping the mixing temperature below 120°C is crucial to ensure the complete reaction of the fatty acid and complexing agent with the oil and the calcium base. During the saponification process, a viscous structure of the mixture was developed after the calcium hydroxide completely reacted with the oil solution of stearic and benzoic acids. The viscosity however was slowly reduced when a higher temperature was applied for heat treatment for the grease. To confirm the homogeneity of the mixture, high-speed stirring is recommended. The final product had a smooth, white, cream-like texture.

Five palm biogreases were prepared in the present study with various thickener and additive concentrations and combinations. Two commercial greases (GC-I and GC-II) that were categorized as soft grease with NLGI grade 00 and 1 were used for reference purposes. The NLGI consistency determines the grease ability to resist deformation under applied load with the lower NLGI grade correspond to softer grease which has lower resistance to deformation. The base oil and the thickening agent composition have a significant contribution to grease hardness or consistency. As the base oil imparts the grease's lubricating properties, the thickener roles as a gelling agent that holds the oil in the structure and determines the hardness. In addition, according to Adhvaryu et al. [10], the grease NLGI also influences a range of grease performance characteristics including the dropping point, pump-ability, viscosity, adhesion, and rheological behaviour.

The NLGI grade of the palm biogrease (PB) formulated in the present study is depicted in Table 3. The results indicate that the synthesis of the palm oil biogrease with 10-15 wt% of calcium complex thickener produces very soft to moderate soft grease with NLGI grade 0 to 2. The PB-IV and PB-V which composed of 10% thickener of the total product weight resulted in a very soft grease (NLGI 0). Even though the PB-II had a slightly higher content of thickener compared to PB-I, the lower oil base content in the formulation resulted in harder grease with NLGI 2. It was also observed that the grease hardness is not altered significantly with the additive contents.

3.3 Wear preventive properties

The lubricating ability of the friction and wear properties as well as the weld point of the formulated palm biogrease were assessed by a series of four-ball tests, under the rotating and sliding actions. Fig. 3 illustrates the coefficient of friction trends over time for the palm biogreases and the reference greases during the tribological tests. Referring to the trends, the coefficient of friction of the palm biogreases was lower from the beginning and increased slowly until it became stable after about 15 minutes run. Conversely, the commercial greases (GC) recorded a higher coefficient of friction at the earlier run, before slowly reducing and stabilizing at a similar period. The different conditions however can be observed after about 30 minutes of run, where the PB-I palm grease sample that contains no additive demonstrate a fluctuating and increasing friction trend. At this period, the boundary layer of the lubricating greases started to reduce, resulting higher wear.

The other palm grease samples which utilized $CaCO_3$ and HA additives (PB-II, III, IV and V) demonstrate lower friction trends. The grease samples were able to maintain the stable friction trends from the 15 minutes of run until the end, explained by the formation of a metallic particle tribo-layer on the friction surface. The PB-IV mixture exhibits the most effective friction reduction palm grease that was able to maintain the tribo-layer with lowest friction and minimum fluctuation. This validates the effectiveness of the $CaCO_3$ and HA additives used for the palm greases.

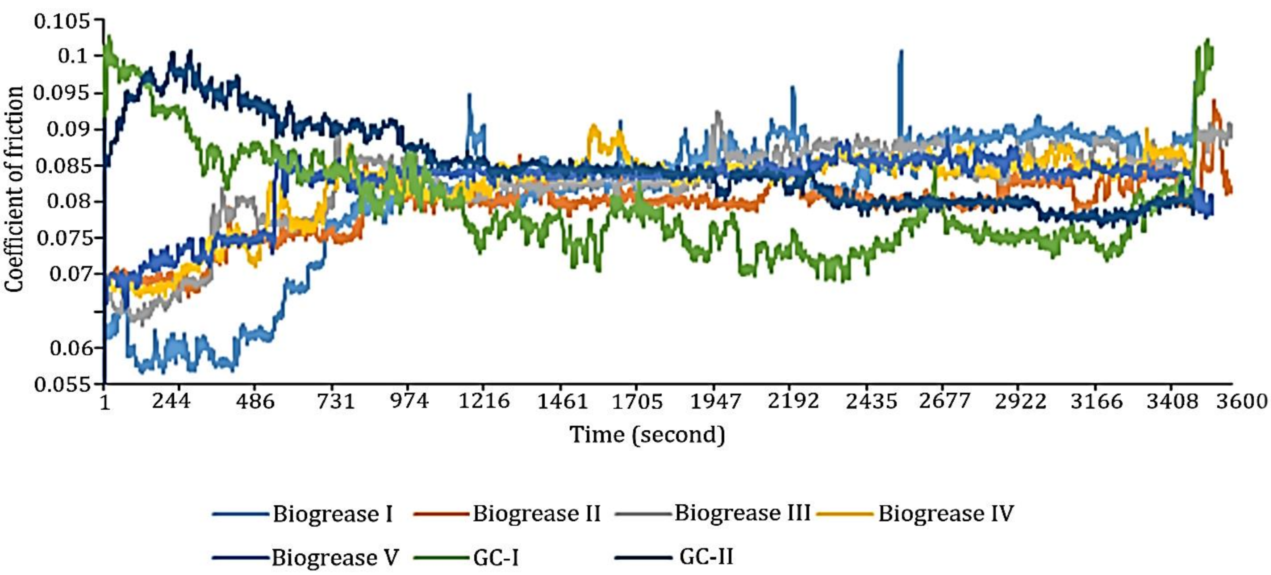

Fig. 3. The coefficient of friction curves over time

The average coefficient of friction for the grease samples is presented in Fig. 4. It was found that the coefficient of friction of the greases formulated with palm oil ester are comparable with the commercial greases, even without any additive (PB-I). For instance, the PB-I palm grease and commercial grease GC-II, both with NLGI 1 grade recorded similar coefficient of friction value, showing the good lubricating ability of the palm ester as the base fluid. This may be contributed from the triacylglycerol structure of the vegetable oil with long, polar fatty acid chains that promotes the formation of high strength and thick lubrication film between contact surfaces [23]. Additionally, according to Dandan and Samion [24], The high content of fatty acid in the palm oil, particularly palmitic acid (C16:0), oleic acid (C18:1), and stearic acid (C18:0), creates strong bonding with the lubricated surfaces.

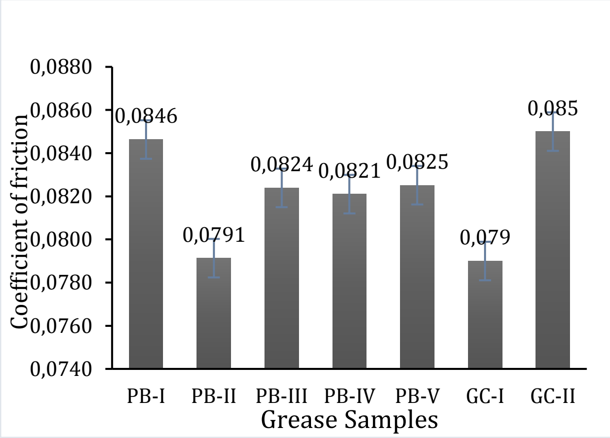

Fig. 4. The average coefficient of friction of the grease samples

This thus contributes to lower friction and better anti-wear properties. The results obtained are also in agreement with past studies that used palm-based oil for biogrease formulation. In the continuation studies, Sukirno et al. [25] and Sukirno et al. [26] have demonstrated the ability of the formulated lithium and calcium palm-greases that performed better as anti-wear protector by producing less amount of wear compared to mineral-based grease. The great performance, according to authors was, attributed from the presence of epoxy ring –COC- , ester groups -COOC- and hydroxides –OH of the modified RBDPO (Refined Bleach Deodorized Palm Oil) and the epoxy RBDPO in protecting the surface from rubbing actions. The studies however did not address the friction behaviour of the formulated greases.

The addition of combined additives of $CaCO_3$ and HA into the palm biogrease also produce superior results. The coefficient of friction values of the formulated biogrease is comparable with the commercial greases that contain MoS_2 and/or graphite solid additives. This demonstrates the efficacy characteristics of the low-cost additives of $CaCO_3$ and HA, that are able to form an effective tribo-film on the contact surfaces and perform as great as the established and well-known solid additives. Furthermore, the non-toxic and environmentally friendly characteristics give even more advantages to the said additives. Referring to Fig. 4, PB-II grease that composed of 5 wt% $CaCO_3$ and 3 wt% HA produced the lowest coefficient of friction, which the total of 8 wt% additives is improved by 6.5% compared to the plain palm grease PB-I. The harder structure (NLGI 2) of the grease might contribute to the lower friction, in which the grease formed a thicker lubrication layer that fosters friction reduction. The higher compositions of additive however did not contribute to significant friction reduction. It was also found that the higher amount of either one of the two additives with same total composition – as in PB-IV and PB-V samples – resulted in a very small friction difference, showing the similar influence of both additives.

The wear properties of the formulated biogrease were evaluated from the wear scar diameter measurement of the balls from the four-ball wear tests. The resulting average wear scar data is presented in Fig. 5, while the wear scar images are illustrated in Fig. 6.

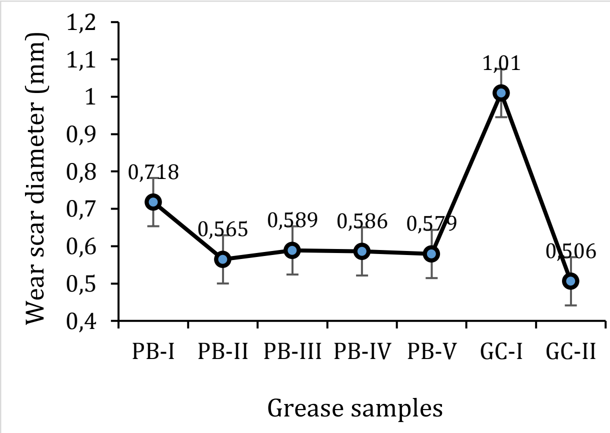

Fig. 5. The average wear scar diameter of the grease samples

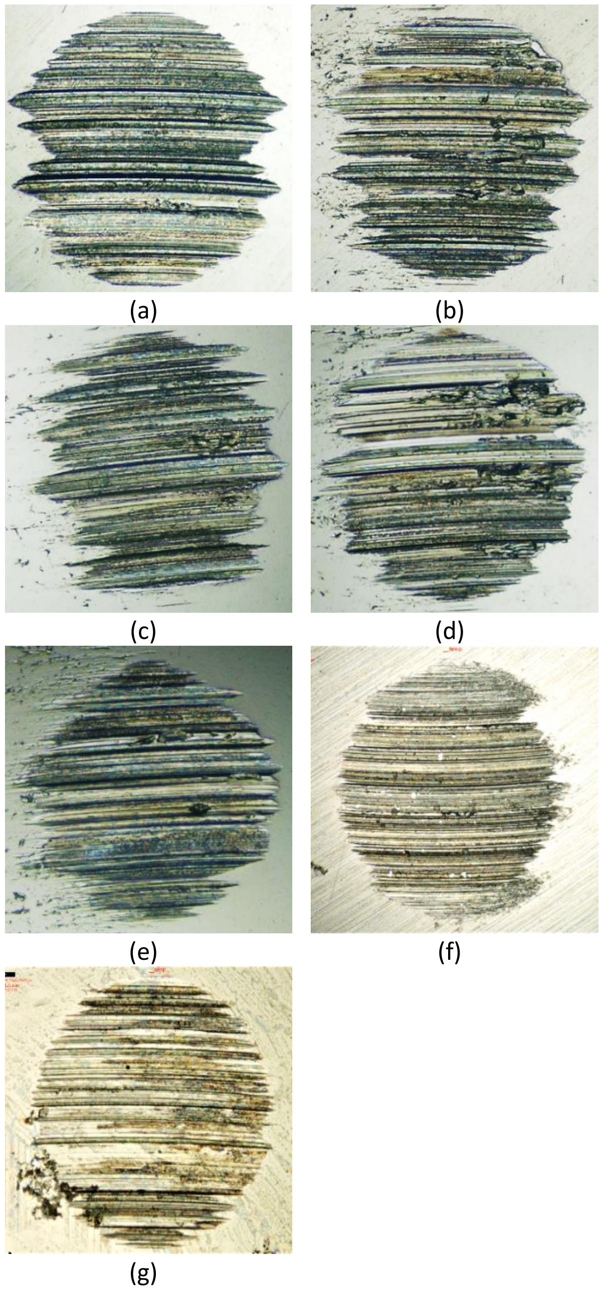

Fig. 6. Optical micrograph of wear scars for (a) PB-I, (b) PB-II, (c) PB-III, (d) PB-IV, (e) PB-V, (f) GC-I, and (g) GC-II greases

The surface analysis obtained shows that the wear scar diameter values for the formulated palm biogrease were in the range of 0.57-0.72 mm, which the addition of the identified additives into the greases had reduced the wear scar diameter to about 0.5 to 0.6 mm. According to Sharma et al. [11], in most cases, up to 0.5 mm scar diameter is an allowable limit for most industrial anti-wear applications. This therefore verifies the good lubricating ability of the palm biogrease. The PB-II grease displayed the smallest wear scar diameter which improved by 21.3% compared to the PB-I that contains no additive, owing to the ability of the $CaCO_3$ and HA additives to effectively form stable surface coatings. The morphologies of the worn surfaces of the lubricate steel balls worn as in Figure 6 also shows the plain palm grease without additive produced large and rough worn surface (Figure 6(a)), with clear edges observed. The grease samples which contain additives produce smoother edges due to the protective film formed by the $CaCO_3$ and HA particles. This shows the excellent anti-wear characteristic of the combined additives. The results obtained were in agreement with Gu et al. [27] that obtained good anti-wear and friction reduction effects of lubricant containing combined nanoparticles. The authors utilized $CaCO_3$ and CeO_2 nanoparticles and found the optimum tribological performance of the oil lubrication with an equal ratio of the two additives. However, like the friction data, the wear properties of the formulated greases also were not affected much by the additive concentration. Referring to the data in Fig. 4 and 5, the different concentrations of the $CaCO_3$ and HA additives, there was only about 1.2-4.2% improvement either with 5 wt% or 10 wt% content in the PB-III, PB-IV and PB-V greases. Thus, it is recommended to apply only 8 wt% of both additives that sufficient for effective friction and wear performance.

The bigger wear scar obtained for the commercial grease GC-1 however was probably due to the higher content of solid additives in the particular grease [28]. The presence of solid additive particles minimize friction at the sliding interfaces by providing rolling effects, resulting in a minimum coefficient of friction. Yet a high concentration of the particles in a lubricant may cause stress concentration on the contact surface which may lead to the formation of abrasive wear and rubbing actions between the contact surfaces. The same condition was also observed on the worn scar produced by the formulated palm grease containing a high concentration of $CaCO_3$ and HA additives. The grease samples PB-IV and PB-V that contain total of 15 wt% of additives producing larger wear scars than PB-II sample containing only 8 wt% of additives. The irregular abrasive grain surfaces can be observed on the surface lubricated by palm greases with a high concentration of additives (Fig. 6d and 6e), with the additive particles deposited on the worn scar surfaces. The abrasive and rubbing actions occurred on the lubricated surfaces resulting in higher friction compared to the lower additive concentrations.

3.4 Extreme pressure properties

The extreme pressure (EP) properties of the grease examine the lubricant performance under high load application, in which the grease should remain on the surface and not be squeezed out. Results for the EP properties of the formulated palm biogreases are depicted in Table 3. The commercial greases that have been assigned as a reference; GC-I and GC-II have a maximum non-seizure load of 400 kg and 315 kg respectively. This indicated that the grease applied should have the ability to maintain the lubricating film and perform its intended function up to at least 315 kg. The formulated calcium complex palm biogrease with no presence of any additive; PB-I could retain its properties up to 160 kg of load, before seizure at 250 kg load. This indicated the lubrication failure of the grease, in which the lubricant lost the ability at the critical load. The load-carrying capacity of the formulated biogrease however was greatly improved, from 160 kg of the plain grease to 400 kg of the formulated grease with $CaCO_3$ and HA additives. This shows that under relatively high applied load, the combination of the two additives produced a significant tribo-chemical reaction film, which even under very high load assigned the grease still had a good ability to retain its properties without breaking down the film. The variation of additive compositions however did not give any significant difference to the biogrease' EP properties. In other words, a total of 8 wt% of $CaCO_3$ and HA additives are sufficient for the desired EP performance. The formulated palm biogrease with the low cost, non-toxic $CaCO_3$ and HA additives therefore demonstrate the comparable EP properties with the commercial greases. Table 3 lists the summary of the experiment results throughout the present study.

4. CONCLUSION

This study presents the formulation of a new biogrease utilizing palm ester as the base fluid and thickened by calcium complex soap. It was found that the properties of the palm ester-based grease were comparable to the commercial grease products, of which the formulated biogrease with the addition of $CaCO_3$ and HA additives demonstrated good friction-reducing and anti-wear properties. In addition, the low cost, green-additives also contribute to the great load-carrying ability of the palm biogrease. According to the result findings, the combination of the $CaCO_3$ and HA additives with a total of 8 wt% composition as dual-action WP and EP additives demonstrate great lubricating potential, placing the palm biogreases on par with other commercial greases. In the further development of the palm biogrease, the optimization of additive concentration needs to be further studied with detailed analysis of the tribological and physicochemical properties.

Table 3. Result summary for the formulated palm biogrease

	The palm bio-grease					**Commercial grease**	
	PB-I	**PB-II**	**PB-III**	**PB-IV**	**PB-V**	**GC-I**	**GC-II**
Base oil (%)	85	80	80	75	75	Synthetic ester	Synthetic ester
Calcium complex soap (%)	15	12	10	10	10	Bentonite clay	Inorganic thickener
Additives						Industrial additives	Industrial additives
$CaCO_3$ (%)	0	5	5	10	5		
HA (%)	0	3	5	5	10		
NLGI number	1	2	1	0	0	00	1
Four-ball coefficient of friction	0.0846	0.0791	0.0824	0.0821	0.0825	0.079	0.085
Four-ball wear (mm)	0.718	0.565	0.589	0.586	0.579	1.010	0.506
Last non-seizure load (kg)	160	400	400	400	400	400	315
Weld load (kg)	250	500	500	500	500	500	400
Appearance	White creamy	White creamy	White creamy	White creamy	White creamy	Grey/ silvery	Grey/ silvery

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