TRIBOLOGICAL PERFORMANCE OF PERFLUOROPOLYETHER (PFPE)-BASED GREASE FOR POTENTIAL APPLICATION IN AUTOMOTIVE BEARINGS

Article history
Received
12th June 2023
Received in revised form
17th September 2023
Accepted
19th September 2023
Published
1st December 2023

Nur Aisya Affrina Mohamed Ariffin,[a,*] Hong Pui Yee,[a] Chiew Tin Lee,[a] Mei Bao Lee,[a] Siti Hartini Hamdan,[b] and William Woei Fong Chong,[a,c]

[a]Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[b]Technical Foundation Centre, University Kuala Lumpur Malaysian Institute of Chemical & Bioengineering Technology (UniKL MICET), Malaysia
[c]Automotive Development Centre (ADC), Institute for Vehicle System & Engineering (IVeSE), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

*Corresponding author
nuraisyaaffrina@gmail.com

ABSTRACT

This study aims to evaluate the tribological performance of various commercially available perfluoropolyether (PFPE)-based greases in order to assess their potential application in automotive bearings. The composition and specification of the greases are taken into consideration for the analysis. The thermal oxidative stability of the selected grease is determined through oxidative thermogravimetric analysis test. Subsequently, the greases are evaluated for their frictional and wear behaviour using a ball-on-disk tribometer. To assess the wear condition of the ball and disk, a digital microscope with Amscope software is employed. The study reveals a scarcity of PFPE lubricant applications in the automotive sector. Among the greases tested, GPL 205 exhibits the highest thermal oxidative stability, characterized by the highest onset temperature of 321°C. Conversely, GPL 215 demonstrates superior friction reduction and anti-wear properties.

KEYWORDS

Perfluoropolyether; Grease; Automotive; Tribology

INTRODUCTION

Friction brings advantages in our lives, like enabling us to walk freely, piling off objects without slipping and stopping a vehicle with the brake system provided. However, friction and wear often initiate issues in industrial applications, such as greater energy consumption and power loss, corrosion and rusting, shortened service life of industrial machinery, and downgrading the safety and reliability of equipment.

To address these challenges, lubricants and greases are commonly used to minimize friction and wear by feeding them into the contact zone between the moving pairs. Grease, defined by the National Lubricating Grease Institute (NLGI) and the American Society for Testing and Materials (ASTM) in ASTM D288, grease is defined as “a solid to a semi-fluid product of a thickening agent in a liquid lubricant with other ingredients imparting special properties may be included.”

Grease comprises three main components: 1) base oil, 2) thickener and 3) additive packages. Grease lubrication is widely applied in rolling bearings [1], railway tracks [2] and electrical contacts [3]. The broad utilization of grease compared to lubricants in industrial applications is due to its extraordinary characteristics. Grease provides better leakage resistance and protects the moving parts against contaminants due to its consistency. Grease can remain in place even under long-term severe operating conditions under its solid features. Besides, grease is a lubricant reservoir, indicating that the grease-lubricated part is expected to have longer relubrication intervals than lubricating oil [4].

Among different types of greases, mineral oil-based grease is preferable for lubrication due to its stability in lubricating performance and lower cost. Despite the good lubricity, this type of grease has raised a threat to our environment due to its non-biodegradable properties. Triglyceride, animal or vegetable oil has become the alternative to cope with the problem. Nevertheless, the poor thermo-oxidative stability of vegetable oil-based grease limits its application in high-temperature working conditions. Thus, the recent selection criteria of grease should not only focus on performance cost but also consider toxicity and biodegradability.

Considering the need for environmentally friendly lubricants, it is crucial to investigate the tribological behaviour of bio-based components to improve grease lubrication. This study evaluates the tribological performance of various commercially available PFPE-based greases for potential use in automotive bearings based on their composition and specifications. Additionally, a bibliometric analysis is conducted to examine the potential application of PFPE lubricants in the automotive sector. The investigation compares the thermal oxidative stability, frictional properties, and wear characteristics of PFPE-based greases with typical mineral oil-based greases.

The proper lubricant for the automotive industry application is found to be limited, creating a research gap to explore new alternative grease. This study gives an insight into the tribological performance of PFPE-based greases and analyses the research trends regarding PFPE lubricants in the automobile industry. It has been found to have superior tribological performance over traditional mineral-oil-based lubricants in terms of improved thermal stability and water resistance, apart from being biodegradable and non-toxic.

METHODOLOGY

Grease Selection

The grease selection criteria concentrate on the potential application in automotive bearings. Five (5) types of grease have been chosen to study their tribological properties. The base oil of three (3) out of the five (5) greases selected is mineral oil because mineral oil-based grease is favourable in the automotive sector (Toyo®), and the other two (2) greases are PFPE-based (Krytox™). Several, consisting of extreme pressure (EP) additives, exhibit different tribological performances. The selected greases and the chemical properties are in Table 1 and Table 2, respectively.

Table 1: Selected Greases

Grease		Base oil	Thickener
A	EAJ 7000EP	Lithium 12-hydroxy stearate	Mineral oil
B	EAJ 101EP	Calcium 12-hydroxy stearate	Mineral oil
C	EAJ 8000	Clay	Mineral oil
D	GPL 205	Polytetrafluoroethylene (PTFE)	PFPE
E	GPL 215	Polytetrafluoroethylene (PTFE)	PFPE

Table 2: Chemical Properties of Selected Greases

Grease	Base oil viscosity at 100ºC (cSt)	Dropping point (°C)	Useful temperature range (°C)
A	Min 15	210	-20 to 136
B	Min 10	125	90
C	Min 15	Non-melt	-20 to 150
D	18	-	-36 to 204
E	18	-	-36 to 204

Thermogravimetric Analysis

A TA Instrument Q500 with an autosampler was used for the TGA experiment. The atmosphere was purified with oxygen, and the heating rate was 10°C/min. The sample size of each grease is set according to standard, raging around 15 – 20 mg.

Friction Test

A ball-on-disk tribometer was used for the friction test using an 8mm diameter stainless-steel ball and a JISSKD-11 tool steel disk. The wear track radius was fixed at 20mm. The test was repeated with different rotating speeds, from 20 to 100 rpm, with increments of 20 rpm and from 200 to 2000 rpm, with increments of 200 rpm [5,6]. The applied normal load was varied from 1 to 5 kg, with an increment of 1 kg for each load. For each configuration, the duration is 3.5 minutes to achieve 200m of distance with three times repeatability with an error of less than 5%. The wear measurements of the ball and disk were observed after the friction test.

RESULTS AND DISCUSSION

TGA Curves

The TGA curves for every grease sample tested are illustrated in Figure 1. Referring to Figure 1, the TGA of grease EAJ 7000EP is divided into two stages. The first stage is from 110°C to 390°C, in which the peak decomposition of the mineral base oil occurs at 318°C, resulting in 70% weight loss. The lithium 12-hydroxy stearate degrades in the second stage, peaking at 448°C. Approximately 1% residue is left after the test, which could be the amount of solid or charcoal from the TGA. The TGA of grease EAJ 101EP, separated into four phases, is displayed in Figure 1(b). The first phase is mainly due to the evaporation of water at 99°C, which is close to the boiling point of water. The mineral base oil volatilizes in the second phase and peaks at 299°C. The third phase occurs from 400°C to 540°C, where calcium 12-hydroxy stearate decomposes and peaks at 436°C and 481°C, respectively. Calcium 12-hydroxy stearate is formed by calcium hydroxide and 12-hydro stearate acid saponification. Calcium carbonate decomposes last at 613°C. The residue left is about 5% of the total weight. The TGA results of grease EAJ 8000 are shown in Figure 1**Error! Reference source not found.**(c). First, the mineral base oil's evaporation occurs from 170°C to 400°C, hitting a maximum at 327°C. Then, clay thickener degrades completely in the second stage at 446°C, with the residual remaining being about 4%.

Figure 1(d) presents the TGA outcome of grease GPL 205. The first phase indicates the volatilization of PFPE base oil, leading to 80% weight loss. Next, the decomposition of PTFE occurs from 500°C to 600°C, maximum at 556°C, with no residue left after the experiment. Figure 1(e) illustrates the TGA curve of grease GPL 215. It is divided into two phases. Initially, PFPE degrades completely at 391°C and subsequently, PTFE decomposes, peaking at 542°C. The decomposition of the additive MoS_2 is not observed on the TGA curve because the temperature range employed for the TGA test does not affect MoS_2. According to the TGA of MoS_2 from the literature, it is very thermally stable up to 800°C [7]. The residue left, MoS_2 is approximately 3% of the total weight.

Thermal Oxidative Stability

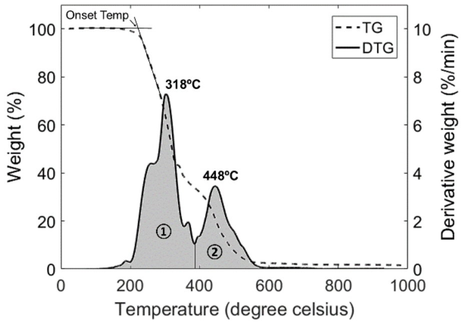
The thermal oxidative stability of the tested grease is determined based on the onset temperature, as shown in Table 4. Experimentally, GPL 205 exhibits the highest thermal oxidative stability by having the highest onset temperature, 321°C. It is believed that the PFPE base oil of grease GPL 205 and GPL 215 could be different, causing their onset temperature variation. The PFPE may be different in terminal group or molecular weight. According to Hoshino et al. [7], PFPE with hydroxyl or carboxyl-terminal group has a gradual decomposition rate compared to PFPE with carboxyl methyl ester terminal group. Besides, smaller molecular weight PFPE lose weight earlier than bigger molecular weight PFPE. The onset temperature is quite similar for typical automotive greases made of mineral base oil, which is reported in the range of 221 to 248°C). The mineral base oil for the grease is mostly aromatics oil, with the onset temperature of aromatics oil being approximately 230°C [8].

Table 4: Onset Temperature of Studied Greases

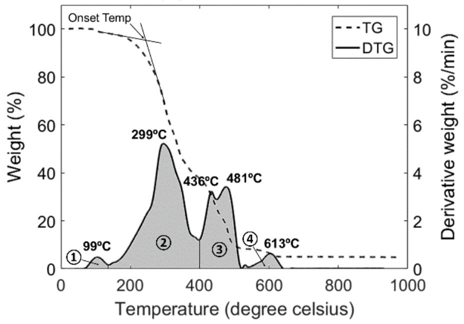
Grease		Onset Temperature (°C)
A	EAJ 7000EP	221
B	EAJ 101EP	248
C	EAJ 8000	235
D	GPL 205	321
E	GPL 215	295

Friction Force against Load Applied

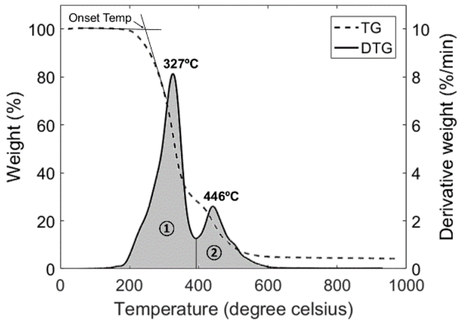
The relationship between the load applied and friction force for selected greases is studied. The graphs of friction force against load applied are displayed in Figure 2 at 1800 rpm. Generally, studied greases have a linear relationship between friction force and load applied, agreeing with Amonton's friction law.



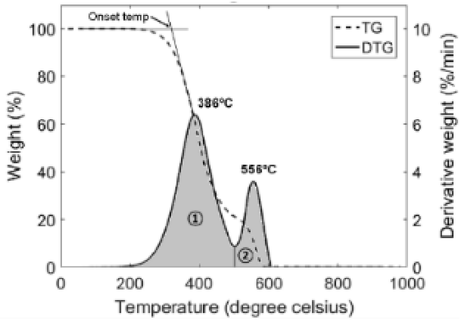
(a) EAJ 7000EP



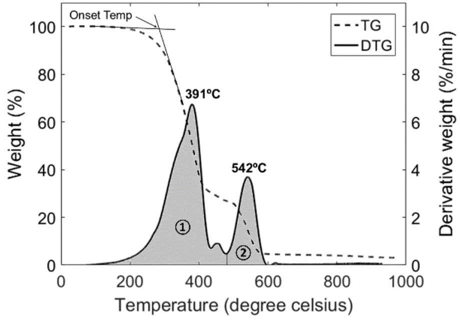
(b) EAJ 101EP



(c) EAJ 8000

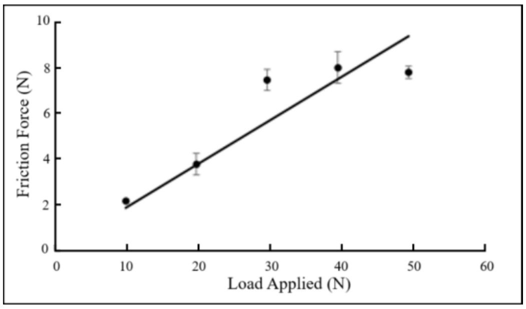


(d) GPL 205

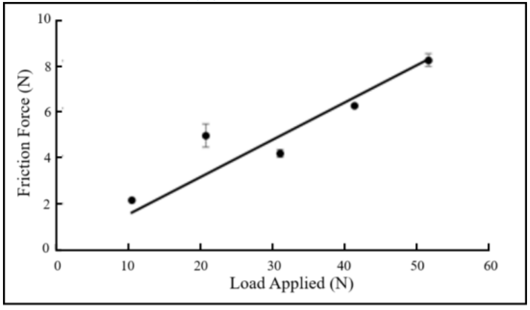


(e) GPL 215

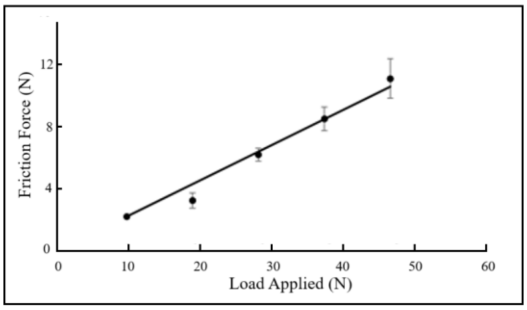
Figure 1: TGA Curves for Studied Greases



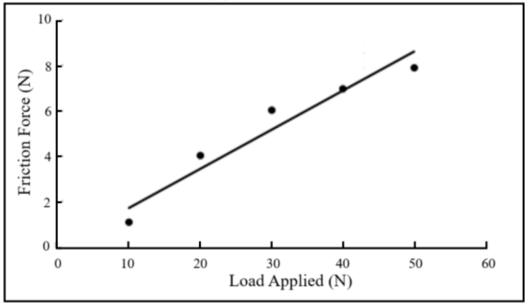
(a) EAJ 7000EP



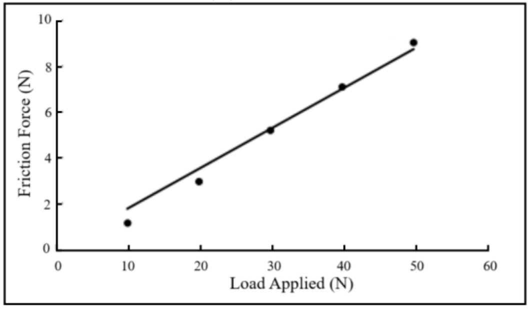
(b) EAJ 101EP



(c) EAJ 8000



(d) GPL 205



(e) GPL 215

Figure 2: Friction Force Against Load at 1800 rpm

Friction Coefficient against Sliding Speed

The Stribeck curves for the tested grease samples, plotted using friction coefficient and sliding speed, are illustrated in Figure 4. The friction coefficient is relatively low at slow sliding speeds. When the sliding speed increases, the friction coefficient increases until a maximum value decreases. As stated by Kanazawa et al. [9], this trend indicates that the tribological properties of the thickener dominate the grease at slow sliding speeds before transitioning to the base oil-dominated region at higher sliding speeds. It is to be noted that the peak friction coefficient signals the transition between these two phases.

The friction coefficient of grease EAJ 7000EP rises from the beginning until it reaches the value of 0.2675 at a speed of 600 rpm. Grease EAJ 8000 also exhibits the same behaviour as grease EAJ 7000EP because the friction coefficient saturates at 600 rpm. The saturation value of the friction coefficient is 0.2341. For grease EAJ 101EP, the peak friction coefficient, 0.5323, occurs at a speed of 400 rpm. While for PFPE-based greases, the maximum friction coefficient of grease GPL 205 and GPL 215 are 0.4267 at 400 rpm and 0.2010 at 600 rpm, respectively.

Based on the literature, low-speed lubrication is dominated by thickener [10]. The friction coefficient is low for the speed range of 20 to 100 rpm due to the contact between the ball and disk being lubricated with enough grease supply. When the sliding speed increases to 600 rpm, the friction coefficient is observed to be increased. This phenomenon happens under the high shear stress and decreased viscosity of grease, resulting in high mobility of grease and is easy to be expelled from the contact. At a faster speed, the grease is expected to be base-oil dominant [11]. The effect of hydrodynamic lubrication, where the contact is separated from direct contact by a full film of lubricant, increases beyond the speed of 600 rpm. Thus, the shear stress is lowered, and the friction coefficient decreases.

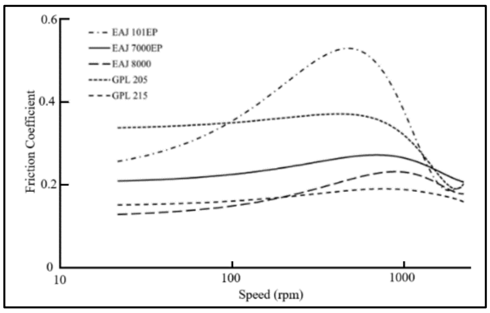


Figure 4: Stribeck-Curve for Studied Greases

It is noticed that studied greases with additives, namely EAJ 8000 and GPL 215, possessed a low friction coefficient. Thus, it can be concluded that grease with additives possesses better friction-reduction properties. Above all, it can be observed that grease GPL 215 demonstrates the greatest overall lubrication performance by maintaining the friction coefficient value low and steadier as compared to the other four greases.

Wear Measurement

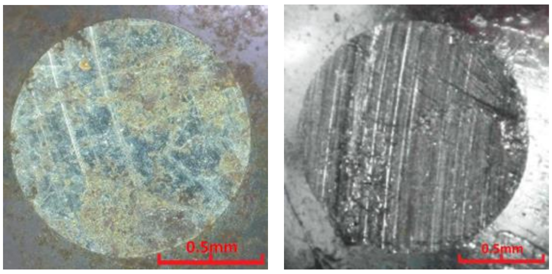
The wear measurement of the scar diameter of the ball and the track width of the disk is shown in Table 5. The ball wear scar diameter and disc wear track width images are captured and displayed in Figure 5 and Figure 6, respectively. The wear life of the grease-lubricated contact is believed to be greater than that of the non-lubricated contact, meaning that the wear measurement value of the grease-lubricated contact is expected to be lower. However, the wear scar and wear track of grease EAJ 7000EP, EAJ 101EP and GPL 205 are higher than that of non-lubricated surfaces. The sliding distance of the experiment can explain this. The dry contact test is conducted at only one speed, 1000 rpm, while the grease-lubricated contact test is run from speed 20 to 2000 rpm.

Table 5: Wear Measurement of Ball And Disc

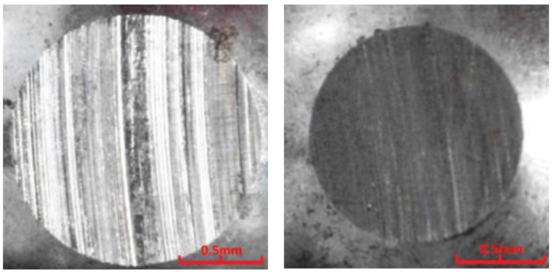
Grease	Wear scar diameter on the ball (mm)	Wear track width on disk (mm)
None (dry contact)	2.44	1.45
EAJ 7000EP	2.68	2.50
EAJ 101EP	2.90	2.56
EAJ 8000	2.23	1.77
GPL 205	3.07	3.00
GPL 215	1.34	0.65

It is found that grease GPL 215, composed of PFPE, PTFE and MoS_2, is the most effective in wear durability enhancement. The presence of the solid additive MoS_2 has adversely augmented the tribological properties of grease GPL 215 as compared to grease GPL 205. This is attributed to the weak van der Waals forces between the sulphur atom and molybdenum atom layers (S- Mo-S). Thus, the formation of tribofilm on the interacting surface becomes easier. The same concept is applied to grease EAJ 8000, made of mineral oil, clay thickener and MoS_2, which exhibited less wear than grease EAJ 7000EP and EAJ 101EP.

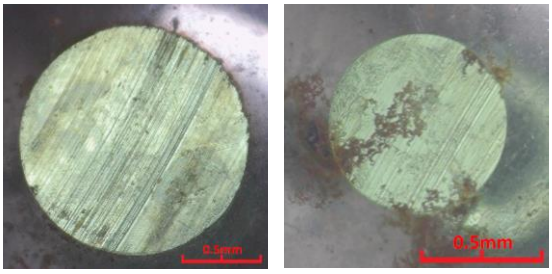
The wear measurement outcome shows that the solid additive MoS_2 plays a crucial role in decreasing wear, and the PFPE-MoS_2 grease works the best in extending wear life for steel-to-steel contact.



(a) Dry **(b) EAJ 7000EP**



(c) EAJ 101EP **(d) EAJ 8000**



(e) GPL 205 **(f) GPL 215**

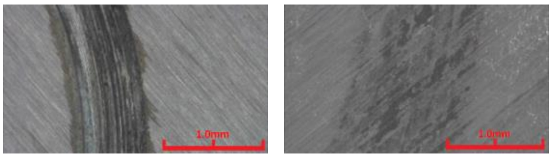
Figure 5: Wear Scar Diameter on the Ball

CONCLUSION

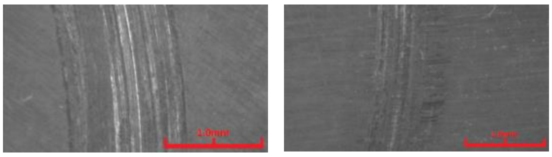
Most of the reported studies focus on the fundamental characterization of PFPE as a lubricant, with a lack of focus on its potential use in the automotive sector. Therefore, further exploration needed to be done to uncover the potential of PFPE in the industry.

Based on the TGA test, compared to mineral oil-based greases, PFPE-based grease possesses higher thermal-oxidative stability. Typical automotive greases composed of mineral base oil have similar onset temperatures, ranging from 221 to 248°C. Among the tested greases, GPL 205 demonstrates the highest onset temperature of 321°C, while GPL 215 has the second-highest onset temperature of 295°C.

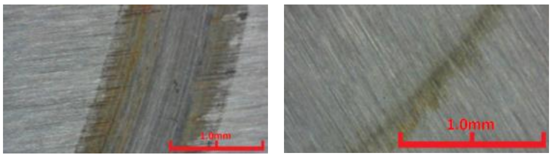
By comparing the coefficient of friction, grease containing additive possesses better friction-reduction properties than mineral oil-based and PFPE-based grease. GPL 215 shows the most favourable overall lubrication performance among the tested greases, maintaining a consistently low friction coefficient. It also effectively protects the ball and disk surfaces from wear, as evidenced by the smallest wear scar diameter of 1.34 mm and the narrowest wear track width of 0.65 mm. Thus, it can be concluded that PFPE-based grease with additives is a good alternative for grease in the automotive industry.



(a) Dry **(b) EAJ 7000EP**



(c) EAJ 101EP **(d) EAJ 8000**



(e) GPL 205 **(f) GPL 215**

Figure 6: Wear Track on Disc

ACKNOWLEDGEMENT

The authors would like to acknowledge the support provided by the UTM Fundamental Research Grant, Universiti Teknologi Malaysia (UTM) under Project vot No. Q.J130000.3851.22H02 (PY/2022/00419).

REFERENCES

[1] *De Laurentis, N., Kadiric, A., Lugt, P., and Cann, P., 2016. The influence of bearing grease composition on rolling/sliding concentrated contact friction. Tribol. Int., 94, 624–632.*

[2] *Temple, P. D., Harmon, M., Lewis, R., Burstow, M. C., Temple, B., and Jones, D., 2018. Optimisation of grease application to railway tracks. Proc. Inst. Mech. Eng. Part F J. Rail Rapid Transit, 232(5), 1514–1527.*

[3] *Achanta, S., and Drees, D., 2008. Effect of lubrication on fretting wear and durability of gold coated electrical contacts under high frequency vibrations. Tribol. - Mater. Surfaces Interfaces, 2(1), 57–63.*

[4] *Lugt, P. M., and Baart, P., 2012. Grease Lubrication Mechanisms in Bearing Seals.*

[5] *Chong, W.W.F., Hamdan, S.H., Wong, K.J. and Yusup, S., 2019. Modelling transitions in regimes of lubrication for rough surface contact. Lubricants, 7(9), p.77.*

[6] *Lee, C.T., Lee, M.B., Hamdan, S.H., Chong, W.W.F., Chong, C.T., Zhang, H. and Chen, A.W.L., 2022. Trimethylolpropane trioleate as eco-friendly lubricant additive. Engineering Science and Technology, an International Journal, 35, p.101068.*

[7] *Hoshino, M., Kimachi, Y., and Terada, A., 1996. Thermogravimetric behavior of perfluoropolyether. J. Appl. Polym. Sci., 62(1), 207–215.*

[8] *Sharma, B. K., Adharyu, A., Perez, J. M., and Erhan, S. Z., 2008. Effects of hydroprocessing on structure and properties of base oils using NMR. Fuel Process. Technol., 89(10), 984–991.*

[9] *Kanazawa, Y., Sayles, R. S., and Kadiric, A., 2017. Film formation and friction in grease lubricated rolling-sliding non-conformal contacts. Tribol. Int., 109, 505–518.*

[10] *Cann, P. M., 2007. Grease lubrication of rolling element bearings — role of the grease thickener. Lubr. Sci., 19(3), 183–196.*

[11] *Ariffin, N. A. A. M., Ang, H. H., Hamdan, S. H., Ahmad, H. A. I. A., and Chong, W. W. F., 2022. A numerical algorithm for grease-lubricated point contact at different regimes of lubrication. Jurnal Tribologi, 35, 50-67.*