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Comparative analysis of novel grease formulations from used transformer and engine oils

Muhammad Auni Hairunnaja [1], Mohd Aizudin Abd Aziz [1*], Mohd Azmir Arifin [1], Nur Amira Fatihah Bashari [1], Navinash Nedumaran [1], Khairuddin Md Isa [2]

[1] Faculty of Chemical & Process Engineering Technology, Universiti Malaysia Pahang Al-Sultan Abdullah, Lebuhraya Tun Razak, 26300 Gambang, Pahang, MALAYSIA.
[2] Faculty of Chemical Engineering Technology, Universiti Malaysia Perlis, Kompleks Pusat Pengajian Jejawi 3, 02600, Arau, Perlis, MALAYSIA.
*Corresponding author: maizudin@umpsa.edu.my

KEYWORDS

Coefficient of friction
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Used oil
Used transformer oil

ABSTRACT

Addressing the dwindling mineral oil supply and the improper disposal of used transformer oil (UTO) and used engine oil (UEO). This study formulates greases from these used oils (UOs) to compare their performances. UTO and UEO are considered wastes and often disposed of improperly due to a lack of awareness on their environmental impacts. Hence, the study focuses on formulating greases from UTO and UEO through several processes, involving the treatment of these UOs to eliminate contaminants, addition of thickener and additives during grease formulation, and subsequent testing for consistency, oil separation, dropping point, corrosion resistance, coefficient of friction (COF) and wear scar diameter (WSD) value for the grease performance analysis. All greases exhibit NLGI values of 2-3, oil bleeding within (-15% - 15%), dropping points ranging from 180°C to 240°C for sodium grease and over 350°C for fumed silica (F.S.) grease, oil separation below 4%, with slight tarnishing in corrosion tests. The top-performing grease, $FG_{84.7}G$, stands out with the highest dropping point with lowest COF and WSD value, making it the most optimal choice. The study concludes that UTO and UEO are effective base oils for grease due to their satisfactory performance.

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1.0 INTRODUCTION

The quest for environmentally sustainable alternatives to conventional mineral oil in various industrial applications is currently a key concern in the energy sector. This focus is primarily driven by the rapid depletion of global fossil fuel reserves and heightened environmental worries stemming from the excessive use and disposal of mineral oil. Used oil (UO) is being explored as a renewable energy source, capable of being transformed into other energy forms. Considering UO and their derivatives as potential substitutes for mineral oil in specific lubricant applications, especially those involving direct environmental contact is gaining attention. In tandem with these efforts, numerous researchers are actively engaged in extensive studies on recycling and related fields to further contribute to environmental conservation like Razali et al. (2017), which includes in formulating grease from several waste oils using red gypsum. Abdulbari et al. (2011) and also develop grease from waste cooking oil (WCO) using spent bleaching earth (SBE). Meanwhile, Nizam and Abdulbari (2010) also developed the same thing from WCO within combination of SBE and fumed silica as additive. M.A. Hairunnaja et al. (2023) also did a review on research about reutilizing WCO into grease, including the reliable formulation steps. Plus, Coroliano et al. (2024) also did the same thing by turning silkworm pupae oil and WCO (e.g. simple soybean oil and ricin oil) into greases for identifying the polyol addition effect and the frying oil re-utilization effects. However, these combined endeavours underscore a comprehensive approach towards sustainable practices and highlight the ongoing commitment to find eco-friendly alternatives in various facets of grease industries.

Numerous engines and mechanisms require lubricant oils for optimal functioning, but nearly half of the purchased lubricant oil eventually becomes waste. The remainder is lost during usage or through leaks. UO are deemed hazardous waste due to their potentially harmful properties. Just one liter of UO has the capacity to contaminate an immense quantity of water, posing a significant threat to aquatic life. If UO are left untreated, they can heavily pollute soil and the environment by emitting harmful substances. All stakeholders, including industries, consumers, and automotive repair facilities, play crucial roles in UO recovery.

In response to the escalating concerns surrounding UO, the global UO market has experienced significant growth, reaching a value of $45.0 billion in 2021 and estimated to reach $70.6 billion by 2031. However, despite its economic value, UO often becomes unsuitable for its initial purpose due to impurities or loss of original properties, rendering it classified as scheduled waste and a threat to public health and the environment. Various types of UO contain undesired impurities such as suspended solids and water, greatly enhancing the potential for environmental harm. The challenge now lies in developing efficient and sustainable methods to recover and repurpose UO, ensuring both economic viability and environmental responsibility in the face of the growing demand for lubricant oils in diverse industrial applications.

The spotlight on eco-friendly products has intensified in recent years. In Malaysia, approximately 150 million liters of UO are generated annually (A. Japar et al., 2019; N. S. A. Japar et al., 2020) along with an estimated 36 million metric tons of used transformer oil (UTO). While the reusing of UO for new products has been implemented in Malaysia (Junus, 2011; Rahman et al., 2021) , the reuse of UTO has not been pursued. Even though the amount of disposed UTO is smaller compared to used engine oils (UEO), finding potential applications for this oil is crucial to prevent an increase in its disposal. The primary motive for involvement in this field stems from environmental concerns. Despite being waste, UTO, albeit in smaller quantities than other UEO, continues to be generated without a repurposing plan. UEO, defined as petroleum-based or synthetic oil no longer performing well due to impurities like dirt, metal scrapings, water, or chemicals, is a substantial contributor to this issue.

While UO is discarded, it remains valuable as it can be recycled or reused, offering affordability and widespread availability. Utilizing UO also helps reduce dependency on dwindling fresh mineral oil reserves. Nevertheless, several treatments are necessary to eliminate undesired impurities that can negatively affect grease properties and to reduce contaminants to acceptable limits (A. Japar et al., 2019; Ismail et al., 2021). Consequently, liquid lubricants have limitations and struggle to meet the escalating performance demands in automotive and industrial sectors (Abd Aziz et al., 2017; Aziz et al., 2019). Technology faces the challenge of developing multifunctional lubricants capable of operating under higher temperatures and pressures across various contact surfaces, with the aim of reducing friction and improving system efficiency (Indah Thuraya Herman et al., 2024) . This has led to a growing focus on the development and application of greases in elastohydrodynamic regimes. The chemistry and composition of greases play a crucial role in determining the thickness and stability of lubricant films.

Hence, the development of greases based on mineral oil has been a subject of active research for decades (A. Japar et al., 2019; Uppar et al., 2022). Recent experiments have investigated grease formulations using various types of UO, as outlined in Table 1. A typical grease composition consists of 60-95% base fluid (mineral, synthetic, or vegetable oil), 5-25% thickener (fatty acid soaps of alkali or alkaline metals), and 0-10% additives (antioxidants, corrosion inhibitors, anti-wear/extreme pressure, antifoam, tackiness agents, etc.) (Abdu Rahman et al., 2019; Odusote et al., 2023). Lubricating greases, defined by the American Society for Testing and Materials (ASTM), are semi-solid colloidal dispersions of a thickening agent in a liquid lubricant matrix. Their consistency is owed to a gel-forming network where the thickening agent disperses in the lubricating base fluid (Abd Aziz et al., 2024). Greases are employed to reduce friction between surfaces by minimizing heat generation and providing a protective film that separates the touching surfaces (Japar et al., 2018; Rahman & Aziz, 2022). Additionally, additives contribute to the grease's durability and long-term performance when applied to materials.

Table 1: Previous studies on production of greases from different type of used oils.

Authors	Used Oil Types(s)	Thickener	Additives
(Hayder Abdulbari et al., 2011)	Used Cooking Oil	Spent Bleaching Earth (SBE)	Fumed Silica
(Sofi et al., 2019)	Used Transformer Oil	Bentonite Fumed Silica	-
(Akumefula et al., 2019)	Used Engine Oil	Sodium Stearate	-
(Razali et al., 2017)	Various type of used oils with similar type of thickener	Red Gypsum & Fumed Silica	-

Given the distinct properties between soap and non-soap-based thickeners, along with concerns regarding used oil-related pollution and fluctuating oil prices, this study aimed to explore the potential of converting UEO and UTO into valuable end products such as grease. The study utilized sodium stearate and fumed silica (F.S.) thickeners to discern the performance differences between various grease thickeners. The research involved identifying different ratios of UTO, UEO, and thickeners, while also examining the impact of additive inclusion on characteristics like consistency, oil bleeding, dropping point, oil separation, corrosion, and tribology test values. The additives incorporation like Polytetrafluoroethylene (PTFE), graphite and Molybdenum disulfide (MoS_2) will exhibit distinguishes between these greases' performances at their presence. However, the goal was to assess how these elements interplay and affect the properties of the resulting grease products.

2.0 MATERIALS AND METHODS

The subchapters covered every material utilised, the grease preparation procedure, and the grease characterization.

2.1 Materials

Grease was formulated using three primary ingredients in different ratios: thickener, additives, and base oil. UEO and UTO were utilized as the base oils in this study. The UEO was obtained from car workshop in Gambang, Pahang. Meanwhile, UTO was obtained from Tenaga Nasional Berhad Research (TNBR), Bangi, Selangor. At first, the oil was first pre-treated to remove contaminants. One type of thickener that is non-soap based, called "F.S.," and one that is soap-based, called "Sodium stearate," were both utilized as thickener in this study. Molecules, polymers, or particles that are only partially soluble in lubricating fluid are known as thickeners. Grease performance will be impacted by various thickeners, primarily in terms of dropping point, water resistance, and grease consistency. In the meantime, graphite, MoS2, and PTFE were the kinds of adding agents that were used.

2.2 Pre-Treatment of Used Transformer Oil and Used Engine Oil

This process is a crucial step in the study, involving the assurance of UEO and UTO being free from undesired impurities and in good condition before proceeding with the grease formulation process. Two significant steps are involved in this process: filtration and heating. The filtration process was conducted using a vacuum filter to ensure that the utilized oils (UOs) were devoid from any undesired impurities or particulates, including solids and inorganic materials (A. Japar et al., 2019; N. Suhaila A. Japar et al., 2020). Next, the UOs undergone second pre-treatment process via heating process. During this step, the UOs were heated to high temperatures, reaching up to 120°C, with continuous stirring for 2 hours to remove and minimize moisture content.

2.3 Preparation of Grease

Tables 2, 3, and 4 present the ratios used in formulating both non-soap and soap-based greases with two different thickeners: F.S. and sodium stearate, along with various additives. The formulation involves four main steps. Firstly, the UO undergoes a pre-treatment process. Subsequently, the WO, thickener, and additives are weighed according to the specified ratios. The UO was heated to a temperature of 80 – 90 °C while being stirred using a homogenizer to ensure the removal of any water content. Then, the thickener was gradually introduced into the solution, and the homogenizer's speed is adjusted to facilitate homogenization, which takes at least one hour. The addition of F.S. was done incrementally to ensure thorough homogenization (Abdulbari et al., 2008; M. A. Hairunnaja, M. A. A. Aziz, et al., 2023). Similarly, in the grease formulation process using sodium stearate, the steps are similar, but it necessitates heating the UO to 180 °C before blending it with the thickener. Then, the grease produced was stored and cooled down for a week. Figure 1 depicts the steps in formulation of grease.

Table 2: Grease percent composition within addition of PTFE.

Code	**Type of grease**	**Used Engine Oil (%)**	**Thickener (%)**	**Additives (%) PTFE**
$SG_{92.13}P$	Sodium Grease (Soap based thickener)	92.13	5.87	2
$FG_{84.7}P$	Fumed Silica Grease (Non-soap-based thickener)	84.7	13.3	2

Table 3: Grease percent composition within addition of graphite.

Code	**Type of grease**	**Used Engine Oil (%)**	**Thickener (%)**	**Additives (%) Graphite**
$SG_{92.13}P$	Sodium Grease (Soap based thickener)	92.13	5.87	2
$FG_{84.7}P$	Fumed Silica Grease (Non-soap-based thickener)	84.7	13.3	2

Table 4: Grease percent composition within addition of MoS_2.

Code	**Type of grease**	**Used Engine Oil (%)**	**Thickener (%)**	**Additives (%) MoS_2**
$SG_{92.13}P$	Sodium Grease (Soap based thickener)	70	28	2
$FG_{84.7}P$	Fumed Silica Grease (Non-soap-based thickener)	92	6	2

Homogenization was carried out continuously for the subsequent three hours, maintaining a minimum speed of 4000 rpm with the homogenizer. The homogenization process was considered complete when the mixture attained a gel-like structure. Following this, the addition of additives took place within 30 minutes. The mixture was heated until it achieved full homogenization. An additional hour of homogenization without heating was then conducted. Simultaneously, additives were introduced at a final concentration of 2% in this study to examine potential variations in grease properties concerning different additives and thickeners.

2.4 Characterization of Grease

To determine the properties of the grease itself, the formed grease will be subjected to several physical tests, including tribology, oil bleeding and separation, dropping point, corrosion, and consistency tests.

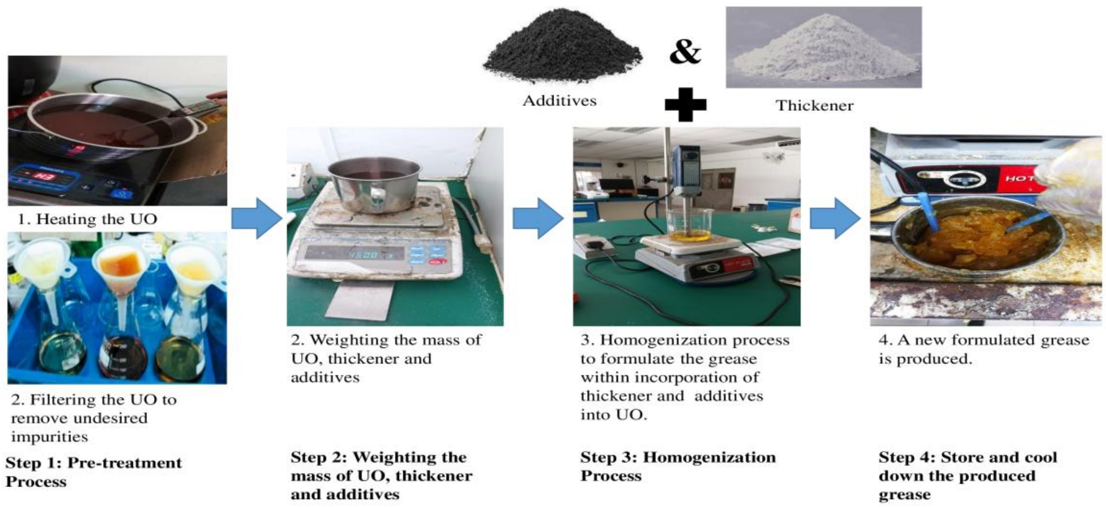

Figure 1: Steps in formulation of grease.

2.5 Consistency Test

The consistency test, as per ASTM D-217, assesses how well a grease maintains its shape when subjected to external force. In the context of lubricating greases, it gauges the relative firmness or softness, and this property is related to flow and dispensing characteristics. The SKF Grease Test Kit TGKT 1 was used to conduct this test on each grease. A fixed volume of grease was applied between two glass plates and compressed under a weight for 15 seconds. The resulting strain in the grease's consistency was observed and measured using a calibrated NLGI grade scale. This testing method adheres to ISO 2137, which outlines procedures for determining the consistency of lubricating greases when dealing with small sample sizes. The NLGI results for each grease provide insights into their consistency levels and help define various characteristics of the greases themselves. Figure 2 shows the way of using the SKF grease test kit and NLGI scale of grease sample.

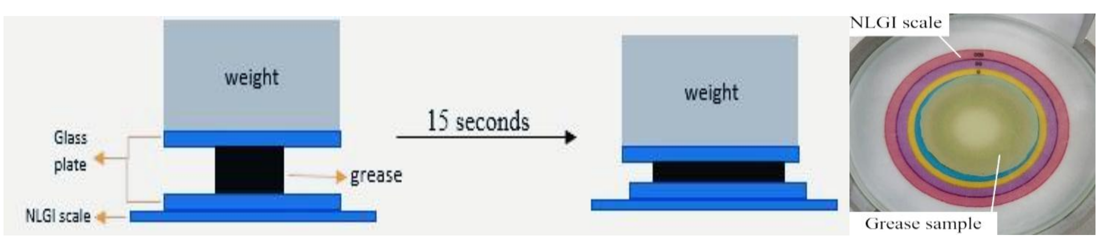

Figure 2: Way of using the skf grease test kit and nlgi scale of grease sample.

2.6 Oil Bleeding Test

The oil bleeding test was executed using SKF's grease bleeding kit, and the percentage of oil bleed was determined by following the instructions outlined in SKF's test kit manual (SKF, 2009). This method required only a small sample volume, offering an alternative approach. The sample was placed on blotter paper and subjected to a 2-hour heating process on a hot plate set at 60°C, adhering to SKF's provided manual as fresh grease (SKF, 2009). The extent of oil staining on the paper was quantified based on the bleed area, and the percentage difference in bleed area between fresh and used samples was calculated using equations (1) and (2). "Used greases" refers to greases that had been subjected to controlled aging conditions for 10 days at both room or ambient temperature and at an extreme temperature of 70°C (Gonçalves et al., 2015; Isa et al., 2017).

a) Oil bleeding of greases at room temperature after being left for ten days at room or ambient temperature

b) Oil bleeding of greases above hot plate at 60°C for two hours

c) Oil bleeding of greases in oven for extreme temperature category at 70 °C for ten days

Figure 3: Oil bleeding analysis at three different types of conditions.

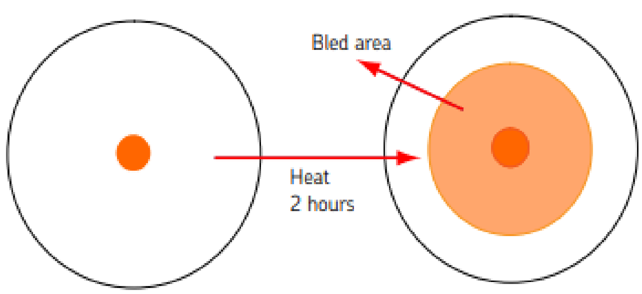

Figure 4: Schematic diagram of oil bleeding process.

$$S_i = 0.785 \times (D_{AV}^2 - 100) \quad (1)$$

$$\% \, Diff = 100 \times \frac{S_{Used} - S_{Fresh}}{S_{Fresh}} \quad (2)$$

In this case, S_i denotes the bled area from both fresh and used samples, D_{AV} is the bled area's average diameter, and %Diff denotes the difference in bled area between fresh and used samples.

2.7 Dropping Point Test

The determination of the dropping point was conducted in accordance with ASTM D2265. The dropping point represents the temperature at which grease transitions from a semi-solid to a liquid state. This phase change is especially typical in sodium-based greases. This test is great significance as it aids in identifying the types of grease and is a vital component of quality control.

Furthermore, the dropping point serves as a valuable indicator of the maximum temperature at which grease can be exposed without undergoing complete liquefaction or excessive oil separation (ASTMD2265-22, 2022). Figure 5 depicts the equipment used for this test and the result values were subsequently calculated using Equation (3).

Figure 5: HK-3498 high temperature dropping point equipment (ASTM D2265).

$$DP(°C) = ODP + \left[\frac{BT - ODP}{3}\right] \quad (3)$$

Here, ODP is the thermometer reading at the moment the drop first touches the test tube's bottom, BT is the block oven temperature at that moment, and DP is the dropping point value.

2.8 Oil Separation Test

In compliance with ASTM D-1742 (Standard Test Method for Oil Separation from Lubricating Grease during Storage), an oil separation test was conducted to determine the tendency of oil to be separated from grease during storage. For a month, the grease sample was kept at room or ambient temperature in a closed container (M. A. Hairunnaja, M. A. Abd Aziz, et al., 2023). The amount of oil that separated that defined as oil puddle was gathered and expressed as weight percentage via equation 4. Figure 6 below shows the systematic diagram of oil separation process on grease.

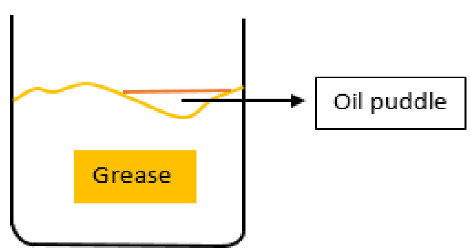

Figure 6: Systematic diagram for oil separation process on grease.

$$\%\ Oil\ separation = 100 \times \frac{Mass_{Oil\ Separated}}{Mass_{grease\ sample}} \quad (4)$$

2.10 Tribology Test

The tribology properties of the lubricating grease play an important role in the grease's performance during operation. This test followed the procedure outlined in ASTM D2266, which involved conducting wear tests at a rotating speed of 1200 rpm (equivalent to a linear speed of 33.38 meters per minute) and applying a load of 392 N for a duration of 30 minutes. The ambient temperature was maintained at approximately 75°C. The friction coefficient of the formulated grease was automatically recorded using a strain gauge integrated into the four-ball tester operated by the SRV test machines. This test will determine the coefficient of friction (COF) and wear scar diameter value (WSD). However, the tribology properties of the grease were evaluated with help from General Tribology Lab, University Malaya (UM) in Malaysia. Figure 7 below is the picture of four ball tester FBT3 equipment.

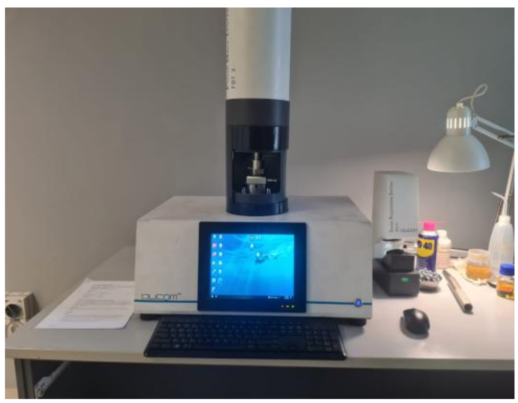

Figure 7: Four ball tester FBT3 for ASTM D2266.

3.0 RESULTS AND DISCUSSION

The study's findings were discussed in this section. The results of the consistency, oil bleeding, dropping point, oil separation, and corrosion tests that were part of the grease properties analysis are displayed in Table 5 below.

3.1 Consistency Test

The consistency of grease is primarily determined by the National Lubricating Grease Institute (NLGI) class, which serves as a measure of its relative hardness (NLGI 6) or softness (NLGI 000). This property is closely related to the flow and dispensing characteristics of the grease. Stiffer greases, such as NLGI 6, are typically used in applications with very high speed and low load requirements (Mortier et al., 2010). Greases with higher NLGI numbers are well-suited for bearing applications with low speeds, high operating temperatures, and the need to resist water washout. On the other hand, greases with lower NLGI numbers are typically used for low-speed rolling element bearings operating at lower temperatures (Mortier et al., 2010).

Table 5 presents the results obtained regarding the consistency class of each formulated grease. None of the greases fell below an NLGI value of 1. Those with an NLGI value of 2 include $FG_{84.7}G$, $SG_{70}M$, $FG_{92}M$, and $FG_{84.7}P$, which fall into the category of medium soft to medium texture greases. $SG_{92.13}P$ and $SG_{92.13}G$ fall under NLGI 2-3, sharing a similar structure description with NLGI 2 greases. No greases in this study were classified as NLGI 4 to 5. NLGI grade 2 to 3 greases are commonly used in various industries (Akin, 2009). NLGI grade 2 greases are suitable for moderately loaded rolling bearings in medium-speed applications, designed to provide a good balance of properties needed for easy pumping through dispensing systems (A. Japar et al., 2019).

Most commercially available greases fall within the NLGI grade range of 1 to 4. NLGI grade 2 is the most common consistency because it exhibits the typical semi-solid appearance of grease. Many multipurpose greases also fall into the NLGI 2-3 category. Some of the formulated greases had a consistency softer than NLGI 2, likely due to an insufficient amount of thickener in the formulation to hold the oil within the thickener system. However, the consistency of these softer greases can be increased by adjusting the oil-to-thickener ratio (Suhaila et al., 2018).

Softer consistency greases can still be used in specific applications, particularly those with low operating temperatures and high-speed requirements. Thus, grease consistency can be tailored in formulations to match the operating conditions of the application. The present study observed that all the sample greases exhibited varying consistency levels, although further research is necessary to determine the most suitable option.

Table 5: Overall results for the formulated greases after testing.

Code Greases	NLGI Consistency Values	Oli Bleeding Values		Dropping Point Values	Oil Separation Values
		At ambient (%)	At 70 °C (%)		
$SG_{92.13}P$	2-3	6.7963	0.8854	225.33	1.0423
$SG_{92.13}G$	2-3	7.0089	0.6567	231.3	1.1420
$SG_{70}M$	2	-6.8200	-14.6000	185.9	0
$FG_{84.7}P$	2	3.4863	5.5006	>350	2.4780
$FG_{84.7}G$	2	2.7712	5.4223	>350	2.2801
$FG_{92}M$	2	-8.8100	-11.5000	>350	0

3.2 Oil Bleeding Test

The oil bleeding characteristics of grease refer to its tendency to release oil. A controlled amount of oil bleeding is desirable in greases because it provides continuous oil lubrication under specific temperature and conditions. Uncontrolled oil bleeding can lead to the loss of grease lubricity and cause it to dry out. The bleeding properties depend on the grease's thickener structure, base oil viscosity, and firmness. To assess oil bleeding properties, the difference in oil bleeding between fresh and used grease was evaluated at both room or ambient temperature and 70°C. According to Muhammad Auni Hairunnaja et al. (2023), when the bleeding difference falls within the range of plus or minus 15 percent (-15% > X > 15%), the grease can still be used without changing the re-lubrication intervals.

Referring to Table 5, all the formulated greases exhibited acceptable oil bleeding values that did not exceed the (-15% > X > 15%) range. Greases formulated with MoS_2 demonstrated the lowest percentage of oil bleeding at ambient temperature, with -6.82% for $SG_{70}M$ and -8.81% for $FG_{92}M$. At 70°C, both greases recorded -14.6% ($SG_{70}M$) and -11.5% ($FG_{92}M$). Subsequently, the oil bleeding rate increased for greases formulated with $FG_{84.7}G$ (2.7712%), $FG_{84.7}P$ (3.4863%), $SG_{92.13}P$ (6.7963%), and $SG_{92.13}G$ (7.0089%) at ambient temperature. At 70°C, the oil bleeding rate showed a significant increase for $SG_{92.13}G$ (0.6567%), $SG_{92.13}P$ (0.8854%), $FG_{84.7}G$ (5.4223%), and $FG_{84.7}P$ (5.5006%). The highest percentage was recorded by $SG_{92.13}G$ (7.0089%) for oil bleeding difference at ambient temperature and $FG_{84.7}P$ (5.5006%) at 70°C. A negative result in greases formulated with MoS_2 indicates that used grease bled less than fresh unused grease.

Higher oil bleeding in grease suggests that it cannot retain base oil within its structure due to intense shearing or vibrations, indicating poor mechanical stability. The addition of MoS_2 in all grease formulations significantly improved the oil bleeding difference at both ambient and 70°C temperatures, extending the grease's lifespan. Lower oil bleeding in the presence of MoS_2 suggests delayed base oil viscosity increase due to high-temperature oxidation. These findings align with A. Japar et al. (2019), who found that MoS_2 could delay grease deterioration. Similarly, (Bhardwaj et al., 2017) indicated that greases blended with MoS_2 experience less damage to the thickener structure and enjoy a longer grease life. In contrast, graphite and PTFE did not have the same oil bleeding rate-reducing effects, consistent with results from (Rahman & Aziz, 2022).

3.3 Dropping Point Test

Table 5 displays the results of the dropping point test for the formulated greases using UTO and UEO as base oils. The results indicate that the dropping point temperature of the formulated greases increases with higher levels of F.S. in the formulation. Additionally, the addition of additives in the formulation has an impact on the grease's dropping point, with MoS_2 known for its superior performance across a wide temperature range, both low and high (Makowski et al., 2022). F.S. and MoS_2 are recognized for their high melting points, reaching up to 1600°C and 1700°C, respectively.

These findings align with previous studies by Abdulbari et al. (2008) and Razali et al. (2017), where no dropping point was observed for greases formulated with F.S., even at temperatures exceeding 350°C respectively. Therefore, the high dropping point of the formulated greases indicates that these greases exhibit thermal stability and excellent heat resistance at elevated temperatures.

Regarding sodium greases, their operating temperature is typically expected to reach up to 175°C. However, in this study, the dropping point values of sodium greases exceeded 175°C. It's plausible that the amount of thickener is closely related to the dropping point value, as this test primarily assesses the thickener's durability under elevated temperatures. The dropping point of grease may increase if the ratio of base oil to thickener (sodium stearate) significantly decreases. A higher amount of sodium stearate corresponds to a higher dropping point value, suggesting that an increased amount of thickener adequately fills the base oil into its pore structure, making WO completely entrapped in grease's structure.

Graphite and PTFE are additives with distinct functions and purposes. Graphite is commonly used as a friction reducer in grease, serving as an effective extreme pressure and anti-wear additive. It also provides higher operating temperatures over the grease, much like MoS_2. PTFE is known as a friction modifier and anti-wear additive. The study's results demonstrate that additives play a significant role in enhancing the maximum operating temperature of the grease. Even though both $SG_{92.13}P$ and $SG_{92.13}G$ have similar grease composition values, the presence of graphite notably raises the grease's dropping point up to 231.3°C. It's important to note that PTFE is not utilized for temperature resistance purposes. Despite graphite and MoS_2 having similar functionalities as additives, the thickeners play a pivotal role in determining the dropping point values of the greases. F.S. grease is considered the best choice for high-temperature grease applications.

3.4 Oil Separation

After one month of storage, all grease samples were examined for oil separation on the grease's surface. The oil separation test is designed to monitor the separation of oil from grease at elevated temperatures for a defined period. The rate at which oil separates from the grease increases with time and varies depending on the storage temperature (Suhaila et al., 2018). When the base oil begins to separate from other components in the grease, it can alter the grease's consistency and potentially affect its ability to perform as intended. According to the ASTMD1742-20 (2020) standard, greases with an NLGI grade of less than 1 are not suitable for the standard test method, but thankfully, none of the greases in this study had such a low consistency value. The amount of oil separated from the greases is presented in Table 5.

Sodium soap, which is a stearate salt, is known for its strong emulsifying properties that can lead to oil entrapment due to its emulsification characteristics (Dayton & Galhardo, 2014). This means it tends to retain the base oil without separating until it reaches a certain operating temperature. In contrast, standard silica possesses oil-retaining capabilities and exceptional heat resistance, which require a longer time for the oil separation process (Abdulbari et al., 2008).

As per Sofi et al. (2019), the amount of oil separated from the grease remained within an acceptable range of less than 4%. The results indicate that all the greases had oil separation values less than 4%. The oil separation values for $SG_{92.13}P$ and $SG_{92.13}G$ were 1.0423 and 1.1420, respectively. However, $SG_{70}M$ showed no oil separation. The values for $FG_{84.7}P$ and $FG_{84.7}G$ were 2.4780 and 2.2801, respectively, while $FG_{92}M$ exhibited no oil separation. F.S. demonstrated a strong ability to retain and attract oil within its thickener system. $FG_{92}M$, with only 6% F.S. content, appeared insufficient to retain 92% of the oil, yet it still experienced no oil separation compared to $FG_{84.7}G$. It's believed that the different base oils exhibit varying abilities to be entrapped within the microstructure of the thickeners. While the pore size matrix of similar thickeners may be the same, it depends on the particle size of the base oil to be emulsified with (Henríquez, 2009). Another factor may be the soot content in both base oils. Higher soot levels lead to higher base oil viscosity. It's possible that the viscosity of UEO is higher than UTO due to the greater soot content, which may have caused some discrepancies in the UEO volume to be entirely entrapped within the thickener structure. In this case, perhaps the UEO was not fully filtered to reduce the soot content compared to UTO. Additionally, an inconsistent homogenization process could impact the oil separation phenomenon due to incomplete mixing between the base oil, thickener, and additives.

The percentage of oil separation increases when the UEO content is raised. While the desirable value for oil separation is below 4%, the sodium grease samples remain well within this desired range. Therefore, $SG_{70}M$ exhibited no oil separation due to the sufficient amount of thickener. Overall, a higher UEO content in the grease leads to an increased level of oil separation, depending on the type of thickener used. This is because of the lack of a thickener matrix that can retain the UEO within the grease, causing the grease to bleed. Even when oil separation occurs in the grease, it can still be used as long as the amount of oil separation is not excessive. Excessive oil bleeding can lead to changes in grease viscosity that may hinder its performance.

3.6 Tribology Test (Coefficient of Friction)

Grease is a substance that is applied to surfaces in contact with moving bodies to control or lessen wear and friction. To investigate and comprehend the frictional performance of these greases, final grease tribological characteristics were assessed (Figure 8).

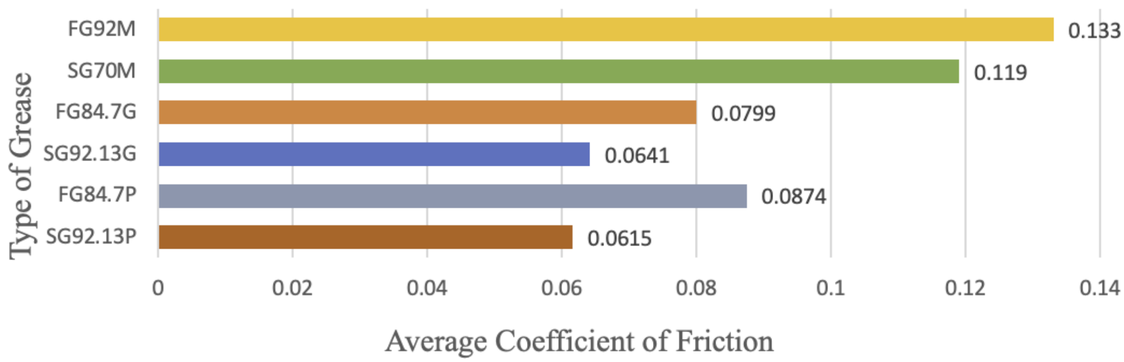

Figure 8: Comparison of COF values with different type of greases.

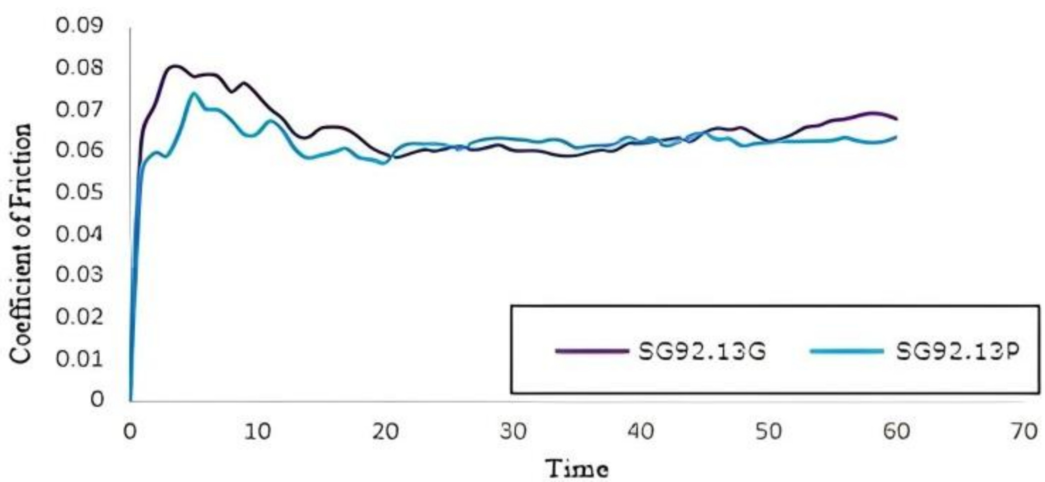

Figure 9: Comparison of COF values between $SG_{92.13}G$ and $SG_{92.13}P$.

The COF of sodium and F.S. grease with additives was measured over a duration of 60 minutes. Figures 9 to 11 illustrate the fluctuations in the COF of the grease. At the outset of the test, all the greases exhibited an initial peak and subsequent fluctuations. In the case of $SG_{92.13}P$ and $SG_{92.13}G$, these fluctuations persisted for 18 minutes before stabilizing. This contrasted with $FG_{84.7}P$ and $FG_{84.7}G$, where the fluctuations only lasted for 6 minutes before reaching stability. The COF remained steady for the remainder of the test. However, greases containing MoS_2 exhibited a different trend. $SG_{70}M$ displayed the first peak and fluctuations from the first minute to the 8th minute, and the COF began to stabilize for 15 minutes. Subsequently, the COF fluctuated briefly from the 23rd minute to the 28th minute, followed by another period of stability for 23 minutes. $FG_{92}M$ demonstrated a similar frictional pattern for the initial 8 minutes, after which the COF stabilized until the 44th minute. Towards the end of the test period, both of these final greases experienced a brief spike in COF before dropping again.

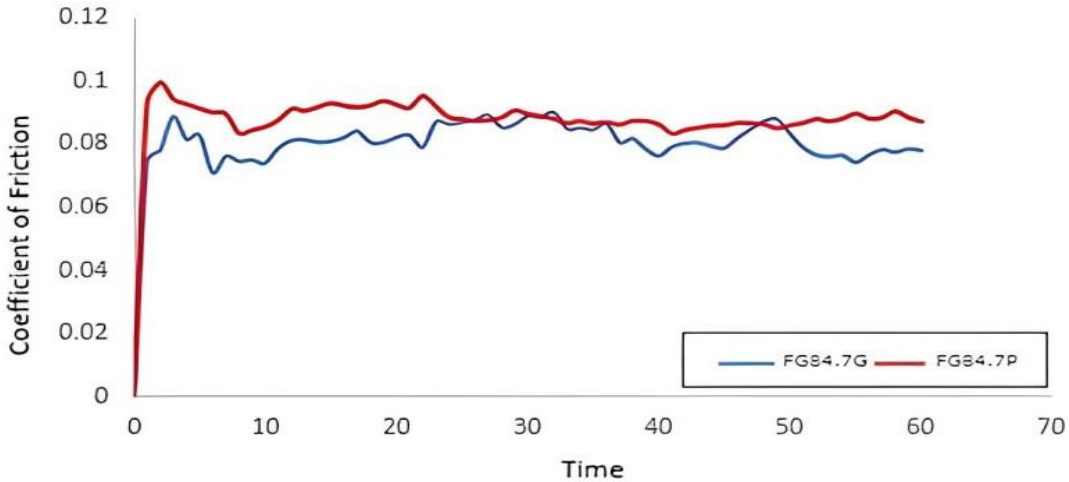

Figure 10: Comparison of COF values between $FG_{84.7}P$ and $FG_{84.7}G$.

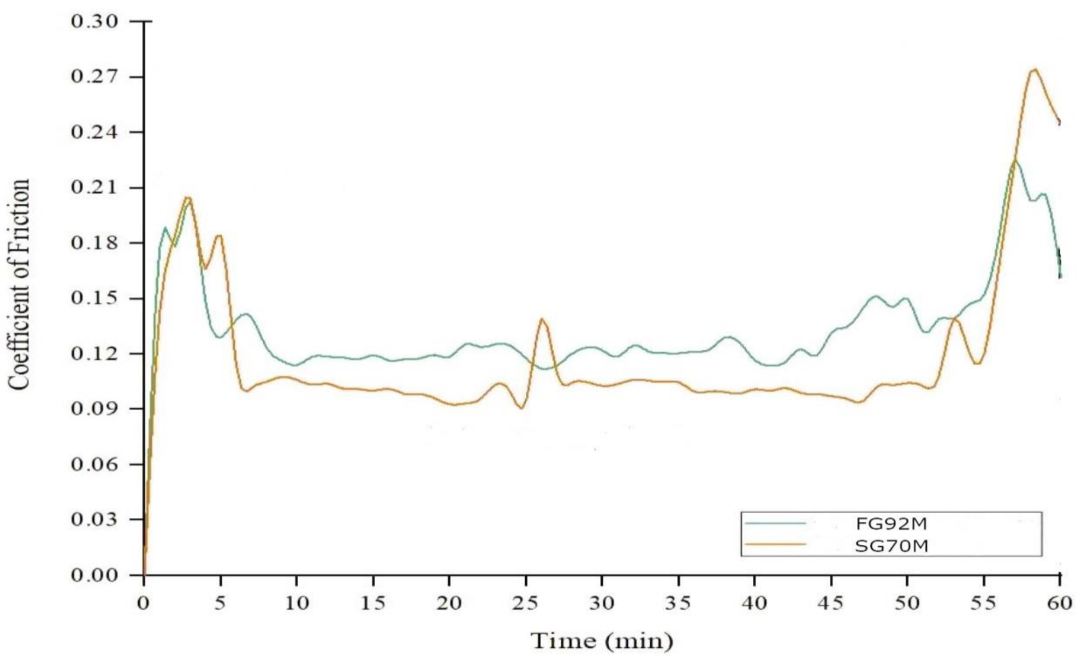

Figure 11: Comparison of COF values between $SG_{70}M$ and $FG_{92}M$.

The average COF for $FG_{84.7}G$, $FG_{84.7}P$, and $FG_{92}M$ were 0.0799, 0.0874, and 0.133, respectively. Conversely, the addition of additives to sodium grease lowered the COF, with an average COF of 0.0815. Notably, $SG_{92.13}P$ exhibited the lowest COF value with an average of 0.0615, followed by $SG_{92.13}G$ with the second-lowest COF, and the third lowest $SG_{70}M$ with an average of 0.119 for sodium grease. These results indicate that the additives enhanced the grease's anti-wear properties. Figure 8 provides an overview of the average COF for various selected greases.

In summary, sodium grease with additives achieved the lowest COF. When comparing sodium to F.S. greases with additives, sodium greases consistently demonstrated lower COFs, signifying superior lubrication. Despite MoS_2 being considered a good additive for reducing frictional forces between sliding surfaces, the combination of UTO and MoS_2 did not significantly impact the COF value. This suggests that the additives did not substantially affect the grease's anti-wear properties. PTFE and Graphite are highly correlated in minimizing the greases' COF values when emulsified with UEO. Instead, changes in COF may be attributed to chemical reactions among the grease components, causing the additives to lose their ability to reduce COF. The results highlight the superior lubricating properties of sodium grease with additives compared to F.S. grease with additives.

3.7 Tribology Test (Wear scar diameter)

The term WSD refers to the degradation of surface structure caused by frictional force created between two contact surfaces due to the grease's minimal lubricity. It is evident that the contact surfaces of the WSD contain some scratches, which might result in lower-quality equipment after consumption. This is how various lubricant additives provide anti-wear and anti-frictional characteristics. The stability of the lubricating film under pressure in the direction of the minimising effects may also be explained by the WSD values. Grease for lubrication is expected to reduce the WSD by reducing friction between the equipment's surface, according to Sivebaek et al. (2003). Studies comparing the WSD of grease at various thickeners and additives are visible in Figure 12. Figure 13 displays the WSD image for each grease presence after the COF analysis.

For $FG_{92}M$, the analysis average of the WSD value is 866 μm, while for $FG_{84.7}P$, it is 760.68 μm. In the meantime, $FG_{84.7}G$'s WSD value—which stands for the lowest value among F.S. greases—was 587.05 μm. These findings follow theory, which states that the lowest WSD value is derived from the lowest COF value.

Unfortunately, the results are still contradicted to the results obtained by sodium greases. Grease with higher COF has higher average WSD value, but grease with lower COF has second highest of average WSD value. The average WSD value for $SG_{70}M$, $SG_{92.13}P$, and $SG_{92.13}G$ are 540 μm, 432.78 μm, and 415.65 μm respectively. Meanwhile, the $SG_{92.13}G$ has the lowest WSD value among the sodium greases even though has second highest of COF value. This is due to the three-body abrasive wear system, where the presence of incomplete removal of nanoparticles in base oil of $SG_{92.13}P$ lead to the abrasive issue. In theory, an excessive number of nanoparticles will eventually form into a large cluster and cause surface damage to the equipment. As a result of having a minimum COF value, the SG92.13P should has the lowest WSD value.

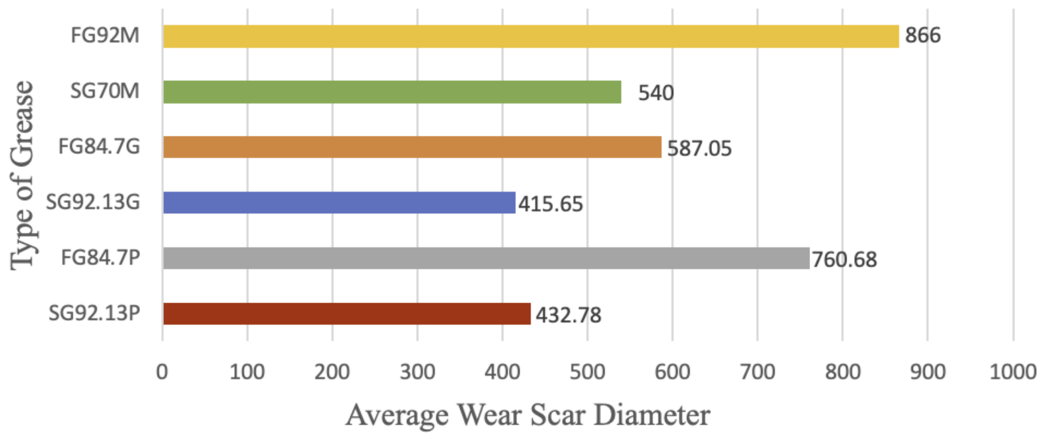

Figure 12: Comparison of WSD values with different type of greases.

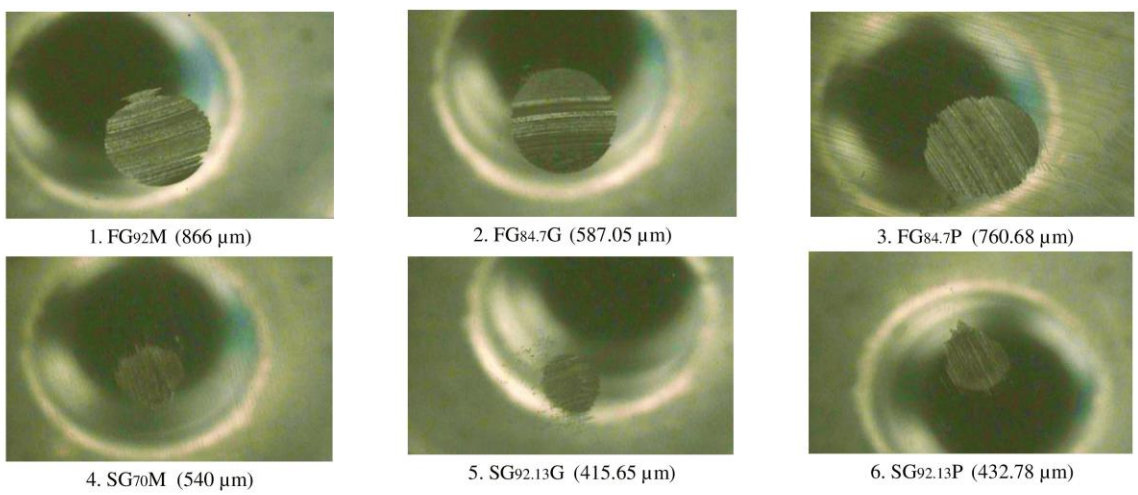

Figure 13: WSD images for each grease presence after the COF analysis.

Graphite was found to be able to lower the WSD value of the lubricating grease based on the overall average data of WSD values. But in this instance, it was found that PTFE and MoS_2 had no effect on the greases' ability to minimise their WSD values. The reason for the expectation in this instance is the inadequate quantity of MoS_2 and PTFE nanoparticles, which prevented them from completely forming a layer of physical deposition film. This resulted in the contamination of the nanoparticles being able to pass through the films and influence the degradation of the contact surfaces.

CONCLUSIONS

The formulation of grease using UTO and UEO is an innovative idea. Transformer oil and engine oil are derived from petroleum resources which were in the same categories as lubricating oil. The possibility of these UOs that being utilized as new product (grease's base oil) could be a promising base oil alternative to replace the conventional fresh petroleum oil which currently decreasing due to the declined of petroleum reserves and to create a green product. However, the grease made from both base oils demonstrated good properties with the addition of additives. These base oils were formulated successfully by using two different types soap thickener (Sodium stearate) and non-soap thickener (F.S.). Incorporation of additive like MoS_2, Graphite and PTFE are only slightly affecting the grease properties. In this case, graphite and PTFE had a tendency to reduce the COF value of grease compared with MoS_2 which is known as the best anti-wear and anti-frictional agent. Thus, F.S. is able to enhance the grease thermal stability by improving the grease dropping point value over 350°C, meanwhile the sodium stearate was unable. Fortunately, due to the selection of sodium thickener in this study, the full potential of additives to improve grease thermal stability is visible when utilize the graphite compared than using MoS_2, as the thickener starts to collapse below the melting point of additive. According to the data obtained for all the formulated greases, they are more suite for bearing application due to have 2-3 of NLGI values. Thus, the UOs need to be heated and filtered completely to remove undesired impurities for obtaining the superior properties especially in term of tribology performance (lower COF and WSD values). However, $FG_{84.7}G$ was considered the best grease due to achieve the highest predominant properties.

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