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THE INGENIOUS STUDY ON THE FORMULATION OF THERMAL STABILITY BIO-BASED GREASE

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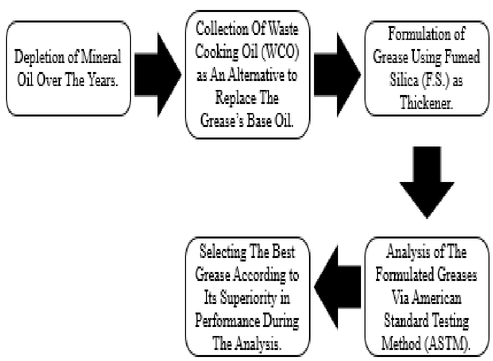
Graphical abstract



Abstract

Concerns about the dwindling supply of mineral oil, which is heavily used in products like grease, have prompted the exploration of Waste Cooking Oil (WCO) as a viable alternative. WCO, derived from improperly disposed vegetable oil, is the focus of this study, which aims to produce grease using Fumed Silica (F.S.) as a thickener. The process involves heating and filtering WCO to reduce moisture content and remove impurities, followed by formulation with additives using a homogenizer at 5000 rpm for three hours, and then cooling for a week. The resulting greases are tested for consistency, oil bleeding, separation, dropping point, and corrosion rate. All lubricants exhibit NLGI values between 00 and 4, oil bleeding from -15% to 15%, oil separation below 4%, a dropping point exceeding 350℃, and a slightly tarnished appearance against copper strips for corrosion classification. An exception is made for $F.G_{94}$ due to its abnormal grease structure. Despite this anomaly, $F.G_{90}$ emerges as the top-performing grease, meeting all specifications. This study highlights the effectiveness of WCO as a base oil for grease production.

Keywords: Copper strips, fumed silica, grease, homogenizer, waste cooking oil

Abstrak

Kebimbangan mengenai bekalan minyak mineral yang semakin berkurangan, yang banyak digunakan dalam produk seperti gris, penerokaan segera Minyak Masak Sisa (WCO) sebagai alternatif yang berdaya maju. Berasal daripada minyak sayuran yang tidak dilupuskan dengan betul, WCO menjadi fokus kajian ini bertujuan untuk penghasilan gris menggunakan Fumed Silica (F.S.) sebagai pemekat. Proses ini melibatkan pemanasan dan penapisan WCO untuk mengurangkan kandungan lembapan dan menghilangkan kekotoran, diikuti dengan perumusan dengan bahan tambahan menggunakan homogenizer pada 5000 rpm selama tiga jam, dan penyejukan seterusnya selama seminggu. Gres kemudiannya diuji untuk konsistensi, pendarahan minyak, pemisahan, titik jatuh dan kadar kakisan. Walaupun semua pelincir mempamerkan nilai NLGI antara 00 dan 4, pendarahan minyak (-15% hingga 15%), pemisahan minyak di bawah 4%, titik jatuh melebihi 350 °C dan klasifikasi kakisan dengan penampilan yang sedikit ternoda pada jalur tembaga, pengecualian telah dibuat untuk F .G_{94} kerana mempunyai struktur gris yang tidak normal. Walaupun terdapat anomali ini, F.G_{90} muncul sebagai gris berprestasi terbaik, memenuhi semua spesifikasi. Kajian ini menggariskan keberkesanan WCO sebagai minyak asas untuk pengeluaran gris.

Kata kunci: Jalur Tembaga, silika berasap, gris, penghomogen, minyak masak sisa

1.0 INTRODUCTION

Vegetable oil (VO) is a term that includes fats from various sources like seeds, nuts, grains, and fruits. Extracting these materials is crucial for their production process. Apart from human consumption, VO and fats are also used in animal feed, medicine, and technical applications [1]. Lipids connect during a dilution process in organic solvents due to the natural breakdown of plant samples or animal tissues. Major vegetables primarily consist of triacylglycerols, which make up about 92% to 98% of their composition. Meanwhile, the remaining components include monoacylglycerols, diacylglycerols, polar lipids, and other minor elements [1].

When VO is no longer good for cooking, it becomes waste cooking oil (WCO). This leftover oil is what remains after cooking. The increase in WCO is connected to more people using edible oils, including both vegetable and animal oils [1]. Typically originating from food service sectors and household applications for daily needs, these oils are collected by various authorities to address environmental pollution concerns [2, 3]. If WCO isn't disposed of properly, it could pollute water sources [4-6].

WCO isn't suitable for reuse because it lacks important nutrients and contains harmful chemicals that can potentially cause cancer-related issues [1]. According to Ganesan, Sukalingam [7], the repeated heating of VO may cause the generation of polycyclic aromatic hydrocarbons (PAH), a carcinogenic agent. This compound has a propensity to bio-accumulate in soft tissues of living organisms via synergetic action [8]. Plus, according to Ifegwu and Anyakora [9], the ability of PAH to attach to DNA has been associated with their carcinogenicity. This binding process can have a number of disruptive effects, including the potential to start tumor growth. On the other hand, the chemical changes that VO undergoes make it more appropriate for use in secondary products rather than for cooking again. The fatty acids in VO, such as palmitoleic acid, linolenic acid, and oleic acid, categorized as unsaturated fatty acids (USFA) group, play vital roles in providing essential nutrients for human health [10, 11]. Typically, palmitoleic acid helps in reducing the body's inflammation, which may aid in preventing from long-term disease including diabetes and heart disease [12]. For the body's health, linolenic acid helps provide important fatty acids like alpha-linolenic acid (ALA), which is beneficial for body health [13]. Reduced low-density lipoproteins (LDL) cholesterol and increased high-density lipoproteins (HDL) (good) cholesterol are two ways that oleic acid can provide and help to improve cardiovascular health. It also helps minimize the risk of getting some types of cancer by preventing the formation of cancer cells [14, 15]. Unfortunately, the amount of USFA will be reduced after repetitive consumption of VO. This is due to the breakdown of chemical structure bonding from thermal forces during consumption [16]. USFA has a double bond of chemical structure which cannot endure several thermal forces during heating, changing into a single bond structure known as saturated fatty acid (SFA) [17, 18].

Besides the mentioned factors, the effectiveness of WCO as a lubricant relies on certain substances that improve its stability under different conditions, especially by providing abilities to reduce friction, microbial properties, environmental friendliness, resistance to water, and emulsification. These abilities play a significant role in ensuring the overall dependability of WCO in lubricant applications, as highlighted by Herman, Md Isa [19]. Many of these substances come from SFA, which is formed through repeated consumption of VO. The formation of SFAs during repeated use of VO is the reason WCO is not recommended for human consumption.

Many researchers worry about the decreasing supply of mineral oil, which is a crucial ingredient in various products like grease. According to Japar *et al*. [25], Mineral oil is often used as grease's base oil due to its cost-effectiveness issue, compatibility with various thickeners and additives, thermal stability which helps in enhancing the grease endurance over time, lubricity effect, and versatility ability. The versatility ability means the interchangeability of its viscosity at various conditions and applications especially in grease composition during operation. Therefore, in order to minimize the utilization of this dwindling raw material, WCO can be utilized as an alternative solution in the grease formulation process. Previous studies by Abdulbari *et al*. [20], had shown that WCO can be used instead of mineral oil in formulating grease. A high thermal grease will be formed and may be applied in various extremely conditions when wco is mixed with non-soap thickeners [20]. Plus, Hairunnaja *et al.* [1] also mentioned that the high amount of kinematic viscosity, viscosity index (VI), Flash Point Value (FPV), Coefficient of Friction (COF) value, interchange of saturated fatty acid before and after VO consumption and potential of resistant from the corrosive issue are the key factors that lead to its eligibility as grease's base oil.

Grease is a mix of solid to semisolid substances made by combining base oil, thickener, and additives. Typically, the proportions are around 80-95% for base oil, 2-15% for thickener, and 0-10% for additives [21, 22]. In this study, WCO is used as the base oil, and fumed silica soap is the thickening agent. The thickener increases the viscosity of the liquid without changing other properties. Different amounts of extra components are also used to enhance positive qualities or reduce negative ones in the grease formulation.

However, this study investigates turning WCO into useful products, specifically a type of grease that doesn't use soap. The research is driven by soap's ability to thicken, concerns about environmental impact from waste oil, and chemical changes in vegetable oil during the conversion process. The study tests different combinations of WCO and fumed silica (F.S.) without extra additives for cost-effectiveness. Various analyses, such as consistency, oil bleeding, oil separation, and corrosive testing, are conducted to understand the interaction between base oil and thickener [23, 24]. The focus is on creating environmentally friendly soap-like grease, developing a production method, and analyzing the results of greases chemically. The main purpose of this study is to clarify the performance of produced greases based on established standards and find the best-formulated grease among those tested. Plus, this study introduces the formulation of lubricating grease from WCO as a sustainable and environment-friendly solution for the disposal and reuse of WCO as a base oil in lubricating grease.

2.0 MATERIALS AND METHODS

2.1 Materials

Grease was made by combining two main ingredients, thickener, and base oil, in different amounts. In this study, WCO was used as the base oil. The WCO underwent a pre-treatment to remove impurities. A non-soap-based thickener called F.S. was used. The WCO was obtained from the cafeteria at University Malaysia Pahang (UMP), and F.S. was purchased from Sigma-Aldrich and R&M Chemicals (Malaysia). A rotor-stator homogenizer was used to mix all the grease components during the formulation process.

2.2 Grease Formulation

2.2.1 Pre-Treatment of WCO

The WCO goes through a pre-treatment to remove impurities and water. This process involves two steps: filtration, using a 1.2µm pore size filter paper to remove small impurities, and heating the oil up to over 90˚C while stirring to minimize the moisture content. This heating stage lasts about one hour, and the temperature must be kept above 150˚C. After this treatment, the WCO becomes classified as treated waste cooking oil (TWCO).

2.2.2 The Formulation Process of the Fumed Silica Grease

Abdulbari *et al*. Abdulbari, Zuhan [20] outlined a process for making F.S. grease, which is corresponded in this study. In early beginning, the TWCO was heated to 120℃ for an hour to minimize the moisture content. Then, it was cooled down at a temperature of 80℃ or 90℃. The F.S. was gradually added into TWCO using specified amounts (Table 1). Meanwhile, a homogenizer ran for three hours at a minimum speed of 5000 rpm. Afterward, the temperature was adjusted to 80℃ until a gel-like paste formed. This ensures that the heat extraction and additional homogenization for 30 minutes completely occurred. The grease was stored in a closed container and left to cool for a week at ambient temperature. Table 1 and Figure 1 show the F.S. grease formulation ratios and steps in the grease formulation process.

Table 1 Grease formulation ratio for sodium grease

Sample		TWCO (%)	Thickener (%)
Fumed Silica Greases	$F.G_{94}$	94	6
	$F.G_{91}$	91	9
	$F.G_{90}$	90	10
	$F.G_{89}$	89	11
	$F.G_{87}$	87	13

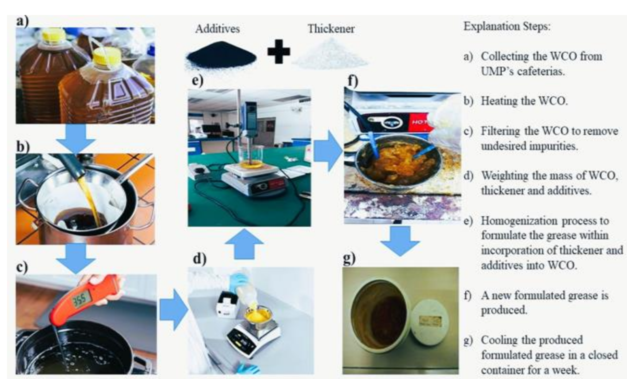


Figure 1 Steps in grease formulation process

However, $F.G_{94}$, $F.G_{91}$, $F.G_{90}$, $F.G_{89}$, $F.G_{87}$ are the fumed silica greases with 94%, 91%, 90%, 89% and 87% of TWCO respectively. These abbreviations will be used in the following discussion.

2.3 Grease Analysis

Several studies were done on the produced grease to understand its natural properties. The analysis included tests for consistency, oil separation, oil bleeding, dropping point, and corrosion. The replication for each test was done for three times.

2.3.1 Consistency Test

The test used the SKF Grease Test Kit TGKT 1 set. Two glass plates were placed between a specific amount of grease, and then a 15-second force was applied using a weight. After that, a calibrated measuring scale with an NLGI grade was used to examine and evaluate the consistency of the grease strain. The test followed the ISO 2137 protocol, which gives guidelines for assessing lubricating grease viscosity when there's a limited sample quantity. However, the NLGI result values show the consistency level for each type of grease. Best grease falls on NLGI 2-3 [23]. Figure 2 shows the method to utilize the SKF grease test kit, including the NLGI scale for the grease sample.

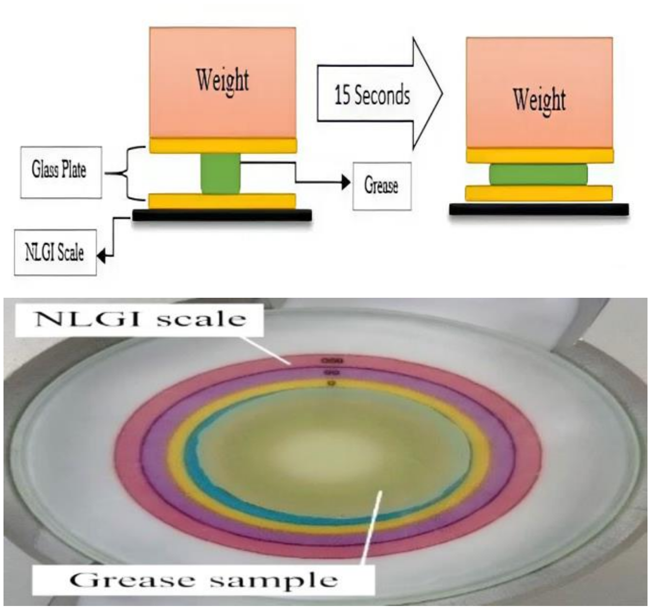


Figure 2: Way of using the skf grease test kit and nlgi scale of grease sample

2.3.2 Oil Separation Test

The oil separation test was done to determine if grease is likely to lose oil during storage. This test was conducted at 25°C by storing the grease for 2 months in a closed container. Then, the mass of separated oil was collected and measured as appeared on the grease surface. Next, ~~an~~ equation 1 was applied to find the percent of oil separation value. However, the acceptable of oil separation limit is < 4% [23]. If it goes over 4%, the grease is not acceptable [25]. Figure 3 depicts the systematic diagram for oil separation process on grease.

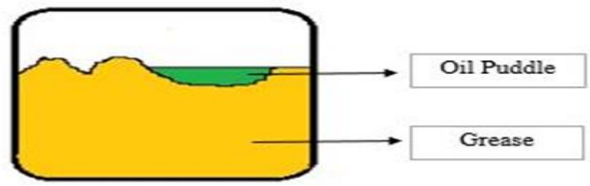


Figure 3 Systematic diagram for oil separation process on grease

$$\%Oil\ Separation = 100 \times \frac{Mass_{Oil\ Seperated}}{Mass_{Grease\ Sample}} \quad (1)$$

2.3.3 Oil Bleeding Test

The oil bleeding test was done with SKF's grease bleeding kit. The percentage of oil bleed was measured by following SKF's test kit manual from 2009. This method used a small sample volume and placed the sample on blotter paper. The paper was then heated for 2 hours on a hot plate set at 60°C, following SKF's manual for fresh grease. The amount of oil bleeding on the paper was identified by measuring the bleed area. The percentage difference in bleed area between fresh and used samples was calculated using equations (2) and (3). "Used greases" refers to greases that underwent controlled aging for 10 days at room temperature and at an extreme temperature of 70°C, according to Gonçalves, Marques [26]. The most acceptable result falls between -15% and 15% of oil bleeding values [23]. Figure 4 and Figure 5 depict the different types of oil bleeding analysis conditions and the schematic diagram of grease's oil bleeding process.

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 4: Oil bleeding analysis at three different types of conditions

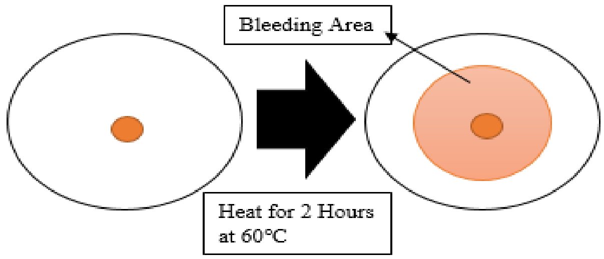


Figure 5 Schematic diagram of oil bleeding process

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

$$\%Diff = 100 \times \frac{(S_{used} - S_{Fresh})}{S_{Fresh}} \quad (3)$$

In this situation, "S_i" represents the combined bleeding area from fresh and used samples. "DAV" is the average diameter of the bleeding area, and "%Diff" indicates the difference in bleeding area between fresh and used samples.

2.3.4 Dropping Point Test

The analysis of the dropping point was conducted by following the guidelines in the American Standard Testing Method (ASTM) D2265, a standard method for determining the dropping point of lubricating grease across various temperatures [27]. In this experiment, a 5g grease sample was placed in a test cup inside a test tube. The tube with the sample was placed in an aluminum oven heated to a specific temperature. A thermometer in the tube measured the rising temperature. When a specific temperature was reached, a droplet fell from the cup to the tube base. The temperature was recorded as the observed falling point and the oven temperature measurement. Furthermore, the higher the dropping point value the better the grease will be [23]. Figure 6 depicts the equipment used for this test and the drop point value was then determined using equation 4 below.

Figure 6 HK-3498 high temperature dropping point equipment (ASTM D2265)

$$DP = ODP + \frac{[(BT - ODP)]}{3} \quad (4)$$

In this context, "DP" stands for dropping point, "ODP" is the thermometer reading when the first drop reaches the test tube bottom, and "BT" is the block oven temperature when the drop falls.

2.3.5 Corrosion Test

A corrosion test was done to analyze the ability of formulated greases to react with the copper strips. The test followed the ASTM D-4048 guidelines, which outline a method for detecting copper corrosion from lubricating grease using Copper Strips. In this test, the copper strips were immersed in grease and heated in an oven at a set temperature of 100℃ for 24 hours. After the test, the strips were removed, cleaned (using ethanol as solvent), and compared to ASTM Copper Strip Corrosion Standards. The best grease fell under the slightly tarnished group category [23]. However, figure 7 and Figure 8 below show the immersion of copper strips into test tubes of grease before being heated in an oven and the observed categorization of corrosiveness levels for the tested copper strips.

Figure 7 The immersion of copper strips into test tubes of greases before being heated into hot water and oven

Figure 8 Classification of Copper Strip Corrosion Standards According to ASTM D4048

3.0 RESULTS AND DISCUSSION

This section talks about the study's discoveries. Table 2 below shows the results of tests on grease properties, including consistency, oil bleeding, oil separation, dropping point, and corrosion tests.

Table 2 Overall Results for The Formulated Greases After Testing

Code Greases	NLGI Consistency Values	Oil Bleeding		Oil Separation Values	Dropping Point Values	Corrosion Test Results
		abt	70°C			
$F.G_{94}$	000	-	-	-	-	1a – slight tarnish (light orange)
$F.G_{91}$	1-2	20.3	13.2	3.5	>350	1b – slight tarnish (dark orange)
$F.G_{90}$	2-3	8.6	1.3	0	>350	1b – slight tarnish (dark orange)
$F.G_{89}$	3	-8.9	-15	0	>350	1b – slight tarnish (dark orange)
$F.G_{87}$	4-5	-18	-24	0	>350	1b – slight tarnish (dark orange)

3.1 Consistency Values of Fumed Silica Greases

The consistency of grease is mostly decided by its NLGI value, which indicates the hardness of grease either at NLGI 6 (hard) or NLGI 000 (soft). This characteristic is closely connected to how easily the grease flows and dispenses. When the consistency of greases increase, like NLGI 6, they are usually used in high-speed and low-load situations [28]. Greases with higher NLGI numbers are suitable for bearings with low speeds, high temperatures, and high water resistance. Conversely, lower NLGI numbers are for slower rolling bearings at lower temperatures [28].

Figure 9 below displays the outcomes of the consistency test in this study.

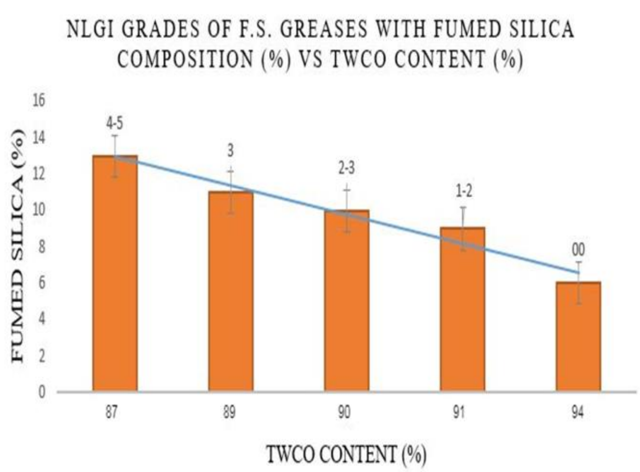


Figure 9 NLGI grade of F.S. Greases with Fumed Silica Composition and TWCO Content

All the greases made with F.S. exhibit a consistent trend. As the TWCO content varies, the NLGI value changes inversely. Greases with more TWCO tend to have a softer structure than to those with lower TWCO. For example, $F.G_{94}$, with an NLGI value of 000, has a structure similar to ketchup. In contrast, $F.G_{87}$ has NLGI values of 4-5. Additionally, $F.G_{91}$, $F.G_{90}$, and $F.G_{89}$ have NLGI values of 1-2, 2-3, and 3, respectively. These findings align with the assertion made by Kaperick [29] that grease consistency depends on the amount of thickener presents in the grease composition.

However, more thickener in grease leads to a higher consistency value. This happens because the thickener traps more base oil, forming a thick layer that affects grease hardness. Greases with high NLGI values, like $F.G_{87}$ in Table 2, are considered hardest due to the high amount of thickener with high NLGI value.

The thickness of grease seems to be linked to the proportion of base oil and thickener. When there's more base oil than thickener, the thickener struggles to completely trap the base oil because of the limited pore structure in it. The pore structure of the thickener acts as a reservoir of grease's base oil. This can be seen from the evidence in Table 2, where $F.G_{94}$ remains in a liquid form with only a small amount of semi-solid grease state. However, this issue is likely due to the incomplete entrapment process which leads to the weak attraction between the base oil and thickener, causing the base oil to be loosely held and easily separated from the thickener matrix system. As a result, the grease gets swept away easily. The lower the grease consistency, the more likely it is to release base oil when the force is applied. Grease has viscoelastic properties, which display both elastic and viscous behaviour under shear stress. Interestingly, it can maintain these properties even under various forces during use. Therefore, the consistency test aims to assess the grease's ability to retain its structure without excessive oil loss and ensure it performs well when subjected to force.

3.2 Oil Bleeding and Oil Separation Values of Fumed Silica Greases

The oil bleeding is important in grease to make sure it lubricates effectively. Grease needs to bleed because the thickener in it isn't a lubricant. The right amount of oil bleeding is necessary to provide good lubrication, reduce friction, and prevent wear between surfaces in different temperatures. The oil bleeding values at ambient or extreme (70°C) temperatures, are determined by the rate at which the grease releases oil. The results of the study, shown in Table 2, explore the connection between TWCO content and oil bleeding values for FG_{91}, FG_{90}, FG_{89}, and FG_{87} at the both ambient and extreme temperature (70°C). The oil bleeding values for these greases at ambient and extreme temperature (70°C) are FG_{91} (20.273% & 13.2762%), FG_{90} (8.5558% &

1.2509%), FG_{89} (-8.8514% and -15 %), and FG_{87} (-18.633% & -24.046%). At the same time, the oil separation values of FG_{91}, FG_{90}, FG_{89}, and FG_{87} are 3.5087, 0, 0, and 0 respectively, indicating that all the greases are acceptable because their oil separation value is below 4% [25]. Unfortunately, FG_{94} wasn't part of this study because it has a lower NLGI value and tends to get swept easily.

According to Table 2, the amount of oil bleeding increases significantly as the TWCO content goes from low to high. This is due to insufficient amount of fibrous thickener to retain the base oil, leading to the oil bleeding process. It's observed that the percentage of oil bleeding in greases is higher at ambient temperature compared to 70°C. This is attributed to prolonged exposure to high temperatures, causing oxidation in the grease. Oxidation-induced changes in oil bleeding values result in microstructural changes in the grease matrix, causing it to dry out. When base oil bleeds out, the soap's volume fraction increases, making the soap structure denser and reducing grease permeability. At 70°C, the lower oil bleeding value is due to thermal stress forcing rapid oil bleeding, leading to the evaporation of base oil. This affects the thickener's fibrous matrix layer, making it denser and thicker, resulting in stiffer grease with limited oil flow. The lower oil bleeding rate at 70°C is due to this rapid process. Additionally, heat alters the thickness of the thickener's fibrous layer, confirming the theoretical expectations.

The study found that at 70°C and room temperature, the oil bleeding rate indicates a direct relationship with the amount of base oil but an inverse relationship with the thickener amount. Grease at 70°C showed less oil bleeding than at room temperature due to changes in the thickener's microstructure caused by high temperature and grease oxidation. Despite a reduction, the oil bleeding percentages at 70°C, as shown in Table 2, are still within -15% until +15%. Fortunately, most tested greases had an acceptable oil bleeding range, except for $F.G_{87}$, suggesting that the choice of thickener type and amount significantly affects grease oil bleeding. The thermal stress strongly impacts the grease's microstructure, influencing its oil-bleeding ability.

Unfortunately, excessive oil bleeding can cause grease to lose lubricating properties as it dries out. According to Japar *et al*. [30], the ideal oil bleeding rate for grease is between -15% and 15%. A positive value indicates more bleeding in used grease than fresh grease, while a negative value suggests less bleeding. The relationship between oil bleeding rate and grease structure helps identify the oil bleeding ability in fresh and used grease. If used grease bleeds more than fresh grease (positive value), it becomes softer and tends to loss its original form [30]. On the other hand, if used grease bleeds less than fresh grease (negative value), it may dry out meaning cannot provide better lubrication. However, determining the suitable grease composition with an appropriate oil bleeding rate is crucial for specific machine applications, as it involves balancing the base oil and thickener needed.

3.3 Dropping Point Values of Fumed Silica Greases

The dropping point in grease formulation refers to the temperature when the grease changes from a semi-solid to a liquid state. It helps assess the bond between the base oil and thickener, considering via their van der wall attraction during the formulation process [31]. Table 2 shows the dropping point values for all formulated F.S. greases, which are consistently above 350 ℃, indicating the greases can handle high-temperature operations effectively.

A high dropping point temperature range is considered desirable for all types of formulated grease. Unfortunately, not all greases have good temperature resistance because the dropping point value depends on the type of thickener used. F.S. greases, in particular, have a dropping point value above 350℃, indicating their suitability for high-temperature machines. The high melting point of F.S. is due to its high glass temperature properties, making it more resistant to melting than other optical glasses. Studies by Japar *et al*. [30] and Abdulbari *et al*. [20] also support the idea that F.S. grease maintains a dropping point value above 350℃. However, it's important to note that F.S. greases may decompose and cannot be reused after testing. The study aims to confirm that F.S. grease can consistently withstand dropping point values over 350°C and resist burning off easily during use.

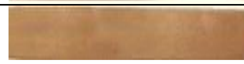
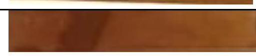
As mentioned earlier, this analysis helps in checking the ability of the base oil and thickener in grease to mix together by comparing greases with similar types but different base oil amounts [30]. To make sure the thickener structure forms completely and works in a broad temperature range, dropping point tests are done for quality assurance. Choosing the right base oil and thickener is crucial for grease performance. If the base oil has a low VI, it can lead to increased oxidation and less base oil, causing minimum oil bleeding occurrence. The type of thickener used also affects the dropping point value of the grease. Some non-soap thickeners, such as F.S. and bentonite, can handle a wide temperature range, while others like sodium stearate and lithium 12-hydroxystearate cannot [21, 31].

In short, the kind of thickener used in grease affects its dropping point. The TWCO content, related to thickener in grease, influences the dropping point value. The VI of the base oil also plays a vital role in enhancing grease thermal stability during oxidation as well as helping it in maintaining the functionality [32]. TWCO has a high VI value, preventing easy oxidation of the base oil and preserving grease lubricity [32]. The dropping point value depends not only on the thickener type but also on the base oil [32]. More thickener usually means a higher dropping point, but excessive thickener (too little TWCO) can leads to inadequate lubrication. Therefore, a moderate amount of thickener and good base oil characteristics are crucial for achieving a balanced grease performance.

3.4 Corrosiveness of Copper Strips against Fumed Silica Greases

The corrosion test can be assessed according to the copper strip appearance when immersed into the formulated greases. Table 3 shows the results of corrosion tests on F.S. greases, and Figure 8 categorizes the corrosiveness level seen on the tested copper strips.

Table 3 Outcomes of Corrosion Studies Conducted on F.S. greases

Type of Grease	Appearance of Copper Strips
Fresh State of Copper Strip	
$F.G_{94}$	 (1a)
$F.G_{91}$	 (1b)
$F.G_{90}$	 (1b)
$F.G_{89}$	 (1b)
$F.G_{87}$	 (1b)

The grease in Table 3 did not corrode the copper strips significantly. The strips showed a slight tarnish, with ratings of 1a (light orange) and 1b (dark orange). Despite this, the corrosiveness levels of all the greases are considered acceptable due to meet the class 1 criteria set by ASTM, as recommended by industry standards [30].

TWCO doesn't cause corrosion in metals with copper. Moisture levels in the previous study don't significantly affect the corrosion of copper strips. Because TWCO is considered non-corrosive, it's recommended for use as base oil in grease to minimize the chance of corrosion on copper-containing metals. In Table 2 and 3, results for all the formulated greases show a non-corrosive effect on copper strips, with only slight tarnishing (class 1a - light orange and 1b - dark orange). Although the greases are accepted as somewhat corrosive (class 1) based on ASTM rating, it's considered acceptable according to A. Japar *et al*. [30].

4.0 CONCLUSION

The eligibility of TWCO can be seen clearly to form into grease product by turning from liquid state into semi-solid state due to the complete emulsification by the appropriate thickener. Next, the effect of F.S. application leads to the thermal stability of grease against the dropping point value. Plus, the treatment process is also necessary to minimize the moisture content value (heating) and undesired impurities (filtration) in the TWCO, which affects the positive impact on the level of grease corrosion. Water has metal elements that may lead to the corrosion of the copper strips. So, heating can minimize the amount of metal content in the WCO. Even though all F.S. greases provide a pleasant performance of data analysis except for $F.G_{94}$, $F.G_{91}$, and $F.G_{87}$, the best-selected grease falls on $F.G_{90}$ due to meet the best criteria of grease performances in terms of consistency, oil bleeding, oil separation, dropping point and corrosiveness level. However, according to the data obtained for all the formulated greases, $F.G_{90}$ is more suited for bearing application due to having 2-3 NLGI values and considerable oil bleeding values which is important for lubricity purpose.

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Conflict of Interest

Regarding the publishing of this work, the authors state that there is no conflict of interest.

References

[1] Hairunnaja, M. A., Abd Aziz, M. A., Bashari, N. A. F., Arifin, M. A., Nedumaran, N., Md Isa, K., & Md Ali, U. F. 2023. The Grease Formulation Using Waste Substances from Palm Oil Refinery and Other Industrial Wastes: A Review. *Pertanika Journal of Science & Technology*. 31(5): 2505–2529. https://doi.org/10.47836/pjst.31.5.25.

[2] Chandraseagar, S., Abdulrazik, A. H., Abdulrahman, S. N., & Abdaziz, M. A. 2019. Aspen Plus simulation and Optimization of Industrial Spent Caustic Wastewater Treatment by Wet Oxidation Method. *IOP Conference Series: Materials Science and Engineering*. 702(1): 012011.https://doi.org/10.1088/1757-899X/702/1/012011.

[3] Abd Aziz, M. A., Md Isa, K., & Rashid, R. A. 2017. Pneumatic Jigging: Influence of Operating Parameters on Separation Efficiency of Solid Waste Materials. *Waste Management and Research*. 35(6): 647–655. https://doi.org/10.1177/0734242X17697815.

[4] Nur Amira Fatihah Bashari, Mohd Aizudin Abd Aziz, Muhammad Auni Hairunnaja, & Mohd Azmir Arifin. 2024. Formulation of Lubricating Grease from Waste Oil: A Review. *Pertanika Journal of Science and Technology*. 32(5). http://dx.doi.org/10.47836/pjst.32.5.15.

[5] Razali, M. N., Mohd Isa, S. N. E., Md Salehan, N. A., Musa, M., Abd Aziz, M. A., Nour, A. H., & Yunus, R. M. 2020. Formulation of Emulsified Modification Bitumen from Industrial Wastes. *Indonesian Journal of Chemistry*. 20(1): 96–104. https://doi.org/10.22146/ijc.40888.

[6] Aziz, M. A. A., Isa, K. M., Miles, N. J., & Rashid, R. A. 2019. Pneumatic Jig: Effect of Airflow, Time and Pulse Rates on Solid Particle Separation. *International Journal of*

Environmental Science and Technology. 16(1): 11–22. https://doi.org/10.1007/s13762-018-1648-4.

[7] Ganesan, K., Sukalingam, K., & Xu, B. 2019. Impact of Consumption of Repeatedly Heated Cooking Oils on the Incidence of Various Cancers-A Critical Review. *Critical Review Food Science Nutrition*. 59(3): 488–505. https://doi.org/10.1080/10408398.2017.1379470.

[8] Garcia-Jares, C., Barro, R., & Llompart, M. 2012. 1.08 - Indoor Air Sampling. In J. Pawliszyn (Ed.). Comprehensive Sampling and Sample Preparation (pp. 125–161). Academic Press. https://doi.org/https://doi.org/10.1016/B978-0-12-381373-2.00008-9.

[9] Ifegwu, O. C., & Anyakora, C. 2015. Chapter Six - Polycyclic Aromatic Hydrocarbons: Part I. Exposure. In G. S. Makowski (Ed.). *Advances in Clinical Chemistry*. 72: 277–304. Elsevier. https://doi.org/https://doi.org/10.1016/bs.acc.2015.08.001.

[10] Orsavova, J., Misurcova, L., Ambrozova, J. V., Vicha, R., & Mlcek, J. 2015. Fatty Acids Composition of Vegetable Oils and Its Contribution to Dietary Energy Intake and Dependence of Cardiovascular Mortality on Dietary Intake of Fatty Acids. *International Journal of Molecular Sciences.* 16(6): 12871–12890. https://doi.org/10.3390/ijms160612871.

[11] Dahawi, Y. A., Abdulrazik, A., Abu Seman, M. N., Abd Aziz, M. A., & Mohd Yunus, M. Y. 2019. Aspen Plus Simulation of Bio-Char Production from a Biomass-Based Slow Pyrolysis Process. *In Key Engineering Materials.* 797: 336–341. http://dx.doi.org/10.4028/www.scientific.net/KEM.797.336.

[12] Hu, W., Fitzgerald, M., Topp, B., Alam, M., & O'Hare, T. J. 2019. A Review of Biological Functions, Health Benefits, and Possible De Novo Biosynthetic Pathway of Palmitoleic Acid in Macadamia Nuts. *Journal of Functional Foods*. 62: 103520. https://doi.org/https://doi.org/10.1016/j.jff.2019.103520.

[13] Dave, D., & Routray, W. 2018. Current Scenario of Canadian Fishery and Corresponding Underutilized Species and Fishery Byproducts: A Potential Source of Omega-3 Fatty Acids. *Journal of Cleaner Production*. 180: 617–641. https://doi.org/https://doi.org/10.1016/j.jclepro.2018.01.091.

[14] Hairunnaja, M. A., Aziz, M. A. A., & Hamid, N. B. 2023. Fundamental Study on the Raw Material Selection for the Formulation of Novel Dolomite A+ Concentrated Solution. *American Institute of Physics Conference Series*. http://dx.doi.org/10.1063/5.0133354.

[15] Nogoy, K. M. C., Kim, H. J., Lee, Y., Zhang, Y., Yu, J., Lee, D. H., . . . Choi, S. H. 2020. High Dietary Oleic Acid in Olive Oil-supplemented Diet Enhanced Omega-3 Fatty Acid in Blood Plasma of Rats. *Food Science & Nutrition*. 8(7): 3617–3625. https://doi.org/10.1002/fsn3.1644.

[16] Rohazriny Rohim, Khairuddin Md Isa, Umi Fazara Md Ali, Mohd. Aizudin Abd Aziz, Naimah Ibrahim, Muhammad Auni Hairunnaja, Nur Amira Fatihah Bashari. 2024. Hydrothermal Liquefaction of Jatropha Curcas (J.Curcas) Under Subcritical Water Conditions: Water and Palm Oil Mill Effluent as Solvents. *Social Science Research Network (SSRN)*. http://dx.doi.org/10.2139/ssrn.4913966.

[17] Sia Yiik Swan, Muhammad Auni Hairunnaja, Nurhusna Samsuddin, Syed Mahmood, Mohd Aizudin Abd Aziz, & Mohd Azmir Arifin. 2024. A Review on the Development of Microcarriers for Cell Culture Applications. *Pertanika Journal of Science & Technology*. 32(5). http://dx.doi.org/10.47836/pjst.32.5.01.

[18] Wardana, I. N. G., Widodo, A., & Wijayanti, W. 2018. Improving Vegetable Oil Properties by Transforming Fatty Acid Chain Length in Jatropha Oil and Coconut Oil Blends. *Energies*, 11(2): 394. https://doi.org/10.3390/en11020394.

[19] Herman, I., Md Isa, K., Ibrahim, N., Kasim, F., & Abd Aziz, M. A. 2021. A Single Step Transesterification Process to Produce Biodiesel from the Spent Cooking Oil. *IOP Conference Series: Earth and Environmental Science*. 765(1): 012077. https://doi.org/10.1088/1755-1315/765/1/012077.

[20] Abdulbari, H. A., Zuhan, N., & Mahmood, W. K. 2017. Biodegradable Grease from Palm Oil Industry Wastes. *Journal of Advanced Research in Fluid Flow*. 1–10.

[21] Sofi, S., Abd Aziz, M. A., a Japar, N. S., Abdu Rahman, N., Abdulhalim, A., & Mohd Yunus, M. Y. 2019. Preparation and Characterization of Grease Formulated from Waste Transformer Oil. *IOP Conference Series: Materials Science and Engineering*, 702(1): 012034. https://doi.org/10.1088/1757-899X/702/1/012034.

[22] Japar, N. S. A., Aziz, M. A. A., Razali, M. N., Zakaria, N. A., & Rahman, N. W. A. 2019. Preparation of Grease using Organic Thickener. *Materials Today: Proceedings*. 19: 1303–1308. https://doi.org/10.1016/j.matpr.2019.11.141.

[23] Hairunnaja, M. A., Abd Aziz, M. A., Abdu Rahman, N. W., Arifin, M. A., Md Isa, K., & Md Ali, U. F. 2023. Innovative Formulation and Characterisation of Grease Made from Waste Engine Oil. *Pertanika Journal of Science and Technology*. 31(5): 2375–2387. http://dx.doi.org/10.47836/pjst.31.5.17.

[24] Indah Thuraya Herman, Khairuddin Md Isa, Naimah Ibrahim, Saiful Azhar Saad, Tuan Amran Tuan Abdullah, Mohd Aizudin Abd Aziz, & Muhammad Auni Hairunnaja. 2024. Performance of Waste Cooking Oil Esterification for Biodiesel Production Using Various Catalysts. *Pertanika Journal of Science & Technology*. 32(2): 669–684. https://doi.org/10.47836/pjst.32.2.10.

[25] Japar, N. S. A., Aziz, M. A. A., & Hamid, N. 2020. Synthesis and Properties Evaluation of Sodium Grease Formulated from Used Transformer Oil as Base Oil. *IOP Conference Series: Materials Science and Engineering*. 863(1): 012058. https://doi.org/10.1088/1757-899X/863/1/012058.

[26] Gonçalves, D., Marques, R., Graça, B., Campos, A., Seabra, J., Leckner, J., & Westbroek, R. 2015. Formulation, Rheology and Thermal Aging of Polymer Greases—Part II: Influence of the Co-thickener Content. *Tribology International*. 87. https://doi.org/10.1016/j.triboint.2015.01.012.

[27] Nagendramma, P., & Kumar, P. 2015. Eco-Friendly Multipurpose Lubricating Greases from Vegetable Residual Oils. *Lubricants*. 3(4): 628–636. http://dx.doi.org/10.3390/lubricants3040628.

[28] Mortier, R. M., Fox, M. F., & Orszulik, S. 2010. *Chemistry and Technology of Lubricants.* Third edition. https://doi.org/10.1007/978-1-4020-8662-5.

[29] Kaperick, J. P. 2013. Grease Thickeners. In Q. J. Wang & Y.-W. Chung (Eds.). *Encyclopedia of Tribology* (pp.1562-1567). Springer US. https://doi.org/10.1007/978-0-387-92897-5_928.

[30] A. Japar, N. S., A. Aziz, M. A., & Razali, M. N. 2019. Formulation of Fumed Silica Grease from Waste Transformer Oil as Base Oil. *Egyptian Journal of Petroleum*, 28(1): 91–96. https://doi.org/https://doi.org/10.1016/j.ejpe.2018.12.001.

[31] Abd Aziz, M. A., Bashari, N. A. F., Hairunnaja, M. A., Arifin, M. A., & Isa, K. M. 2024. The Application of Continuous Pneumatic Jig for Solid Waste Separation. *International Journal of Environmental Science and Technology*. https://doi.org/10.1007/s13762-024-05691-9

[32] Palub. 2020. Understanding the Viscosity Index of a lubricant. https://www.q8oils.com/energy/viscosity-index/.