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Innovative Formulation and Characterisation of Grease Made from Waste Engine Oil

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ABSTRACT

Lubricating grease is usually produced from mineral oil, making the relationship between grease and mineral oil unavoidable. Formulation of grease from waste oil can reduce the dependency of the grease industry on mineral oil as well as help to reduce the waste generation of used oil. This study aims to produce fumed silica (FS) grease from waste engine oil (WEO) and analyse the properties of the formulated grease. The method started with treating WEO to remove any contaminants in the used oil. After that, the greases are produced using a weight percentage ratio before being examined for consistency, oil separation, oil bleeding, FTIR (Fourier transform infrared spectroscopy) analysis, and corrosiveness. In terms of uniformity, oil separation, and oil bleeding, WEO percentage content had a substantial impact on the findings. The FTIR demonstrated that synthetic greases had the same spectra when evaluated between 500 cm^{-1} and 4000 cm^{-1}. The grease's corrosiveness is low, as determined by class 1 corrosiveness toward the copper strip. However, the grease properties differ when consistency, oil bleeding and oil separation test is done. Higher oil content in grease produced high oil bleeding and separation but low consistency. As a conclusion of the results, fumed silica grease with oil percentages of 83 and 82 have the most grease-like features, showing that the grease fits the traits' requirements. Based on the investigation's findings, it was established that WEO may be used as a base oil in grease formulation and that the grease's properties are satisfactory.

Keywords: Fumed Silica (FS), grease formulation, waste engine oil (WEO)

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INTRODUCTION

Lubricating grease is primarily composed of lubricating base fluid, a specific type of thickening agent, and an additive chosen based on the function of the grease. The base fluid is a lubricant, and the thickening acts as a sponge, holding the base fluid together. The base fluid used in grease is typically mineral oil, with a few instances of synthetic-base oil being utilised depending on the application. It is mostly because mineral oil may function satisfactorily as grease in industrial applications (Misozi et al., 2018). Metallic soap is the most common form of thickening used in grease production, which contains lithium, polyurea, aluminium, sodium, clay, and calcium (Daniel & Paulus, 2019). It will provide critical grease properties such as chemical and thermal stability, mechanical stability, and rheology. It also acts as a sponge, retaining the main fluid (Fan et al., 2018). Grease formulation also includes an additive addition which enhances the grease performance depending on the type of grease used. Incorporating additives into the grease improves properties such as high temperature, high pressure, water resistance, and anti-wear. Molybdenum Disulphate (MoS_2) is a common additive found in grease. According to Epshteyn and Risdon (2010), MoS_2 is a powder that ranges from dark grey to black. It is extensively employed in a wide range of non-aerospace applications. It was historically used as grease for bit lubrication and has long been a great solid lubricant (Fink, 2021).

Lubricating grease is preferred in difficult-to-reach areas of mechanically rubbing or sophisticated machinery. Grease has several properties that allow it to retain stability in water, have highly consistent shear stability, and deal with viscosity fluctuations despite changes in temperature and pressure—all of which contribute to its functionality as a storage medium for base oil and additives (Adhvaryu et al., 2005). Grease is divided into numerous varieties based on its use, such as insoluble-solid thickened greases, polymer-thickened greases, soap-thickened greases, and lithium and aluminium grease (Casserly et al., 2018).

Using waste oil as the primary oil in grease formulation is a new and emerging trend. Waste oil is petroleum-based or synthetic oil that has deteriorated due to pollution. It has lost its original properties due to pollutants (Japar, Aziz, & Razali, 2018; Japar, Aziz, Razali, & Rahman, 2018). The most frequent type of waste oil is automobile lubricant waste oil, abbreviated WEO. WEO is produced using motor oil. Motor oil waste is a high-contaminated commodity that must be treated with caution. Depletion of additives, metallic components, particle dirt and grits, and other asphaltic particles are examples of waste motor oil pollution (Tsai, 2011). Discharging waste oil into the ground or water streams, including sewage, can harm the environment. It could contaminate groundwater and soil (Hegazi et al., 2017).

Many previous studies discovered that WEO might be utilised as a base oil in grease formulation. Except for sulfation, the properties of WEO after treatment are nearly identical to those of fresh engine oil. The current study intends to produce grease from WEO and then analyse the properties of the greases based on the results. The current trend in non-soap grease manufacturing is to maximise the ability of Fumed silica (FS), known for its significantly small particle size and thickening effect, making it an excellent thickener. Because of its high surface energy, it is used in a wide range of industrial applications. Due to the flame reaction that occurs when silicon tetrachloride and oxygen come into contact, fumed silica is also known as pyrogenic silica (Ha et al., 2013). It is non-toxic and available in the form of white powder. It is commonly used as a thickening and anti-caking agent. According to Rahman et al. (2018), it is composed of amorphous silicon dioxide with silanol groups all over it (Vansant et al., 1995). Because the silanol groups are so highly reactive, they can be used to start chemical reactions. Its reactivity surface is determined by the number of silanol groups surrounding the surfaces of FS powder. Aside from that, as opposed to a large surface area, Barthel et al. (2005) claimed that the structure of FS is related to having space-filling particle characteristics. Aside from that, FS is free of swelling or chemical inertness, making it an effective thickening agent (Barthel et al., 2005). It is one of the advantages available when compared to FS. Table 2 lists the numerous trials where FS was used as a grease thickener and additive.

In other words, fumed silica (FS) is a white powder with an exceptionally low density. This thickener is non-melt and can be mixed with base oil. When FS is dispersed in oil, it has a texture similar to gel and is colourless. Furthermore, according to Whitby (2020), the network synthesis by FS in WCO increases the mixture's viscosity and thickens the structure stability. FS is resistant to oxidation, has a high-temperature tolerance, and has a significant thickening effect. Additionally, at relatively low concentrations, the FS particles can form the unique 3D network found in fumed silica (Santos et al., 2011).

It demonstrated that using fumed silica as a grease thickener is advantageous and improves the characteristics of the grease. Due to the fibrous matrix of the pore structure of FS, has tendency to entrap the base oil entirely until may turn from liquid into semi-solid state. FS grease has excellent stability with minimal oil leakage and separation. The way of formulating grease also affects the homogenization process between base oil and thickener itself. An appropriate composition of grease gives significant effects especially in term of grease consistency, oil bleeding and oil separation. The FTIR analysis is also essential since it helps in defining the type of desired or undesired impurities availability since they have a tendency in effecting the corrosiveness of the grease due to corrosive agents like sulphur and metal content. Therefore, many criteria considerations should be taken in formulating grease to ensure it meets the commercial grease standard.

MATERIALS AND METHODS

Materials

In this study, waste engine oil (WEO) with black visual colour and 0.8751 g/mL of density was acquired from a workshop in Kuantan, Pahang. Meanwhile, the fumed silica (FS) of low-density white powder with 99% purities from R&M Chemical Kuala Lumpur, Malaysia was selected to be used in this study. The FS is supposed to have a high melting point of up to 1600°C.

Grease Formulation

Pre-Treatment of WEO. The sedimentation of any large, suspended substance starts the process. The filtering operation was then started using a vacuum filter and a glass microfiber filter with a pore size of 1.2 µm. The filtration eliminated any small, suspended particles in the WEO. Evaporation was the final procedure, which helped to remove any moisture in the WEO by heating it for 1 hour at 120°C. The altered WEO was saved in a container for future usage.

Preparation of Grease Using WEO. Abdulbari et al. (2008) developed the methodical process which leads to the production of FS grease, which begins with heating WEO for a minimum of one hour at a constant temperature of 120°C. Throughout the one-hour duration, the WEO is stirred to remove all moisture. The temperature is then lowered to 80°C - 90°C in gradual phases before the gradual inclusion of FS begins. Both processes are done gradually to ensure thorough homogenisation (Abdulbari et al., 2008).

The next step is to allow homogenisation, which is accomplished by allowing the homogeniser to run at a minimum speed of 4000 rpm for three hours. When a substance similar to gel is produced, the grease production process is considered complete. The grease is then allowed to cool at room temperature. Following that, it must be kept in a closed container. Two days must elapse before testing to ensure complete cooling of the grease. It is to ensure that the test results are consistent, as the characteristics of the grease change with the presence of heat, particularly grease consistency. The grease formulation ratio employed in this study is shown in Table 1.

Table 1
Grease formulation ratio

Sample		WEO	Thickener (FS)
FS Grease	FG_{85}	85	15
	FG_{84}	84	16
	FG_{83}	83	17
	FG_{82}	82	18
	FG_{81}	81	19
	FG_{80}	80	20

Grease Analysis

Several studies were carried out on the grease produced to establish its properties. The test includes consistency, oil separation, oil bleeding, FTIR (Fourier transform infrared spectroscopy) analysis, and corrosion testing.

Consistency Test

This test was performed using an SKF Grease Test Kit TGKT 1 set. The two glass plates were placed between a fixed amount of grease, which was then pushed for 15 seconds with the weight. Then, using a calibrated measuring scale with an NLGI grade, the consistency of the grease strain was inspected and assessed. This test procedure complied with ISO 2137 (Japar et al., 2014), which details techniques for assessing the consistency of lubricating greases when only limited samples are available. Each grease's consistency level is shown by the NLGI results.

Oil Separation

Oil separation testing was done in accordance with ASTM D-1742 (Standard Test Method for Oil Separation from Lubricating Grease during Storage) to determine the propensity of oil to separate from grease during storage. The grease sample was kept at room temperature for a month in a sealed container.

Oil Bleeding

In this part, the SKF Grease Test Kit TGKT 1 was used as an alternative technique for the oil bleeding test, requiring only modest volumes of samples (Lugt, 2013). In accordance with the instructions in the SKF manual 1, a sample of fresh grease was placed on blotting paper and heated for 2 hr at 60°C using a hot plate. Using the bleed area and the percentage difference between the bleed areas of fresh grease and used grease, Equations 1 and 2 were used to determine the amount of oil stain formed on the paper. Grease that has been aged under two controlled conditions for 10 days at room temperature and at 70°C is referred to as used grease.

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

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

S_i denotes the difference between the bled areas from the fresh and used samples, DAV denotes the bleeding area's average diameter, and %Diff denotes the bled area's difference between the two samples.

FTIR (Fourier Transform Infrared Spectroscopy) Analysis

All the grease sample base oil and thickener types were determined using the Fourier transform infrared (FTIR) spectroscopy. The FTIR spectrum can reveal any changes and contamination in a grease sample by contrasting it with a fresh grease reference and a used grease sample. Grease and oil characteristics were analysed in this experiment with wave numbers ranging from 500 to 4000 cm^{-1}.

Corrosion Testing

The extent of the grease's corrosiveness toward copper strips was assessed using a corrosion test. This procedure was carried out in accordance with ASTM D4048 (Test Method for Detecting Copper Corrosion from Lubricating Grease by the Copper Strip Tarnish Test). The grease sample is placed on a copper strip prepared for this purpose and then heated in an oven or liquid bath at a predetermined temperature for a predetermined amount of time. After the test, the strips were removed from the sample, washed, and compared to the ASTM Copper Strip Corrosion Standards (ASTM D4048, 2018).

RESULT AND DISCUSSION

Grease Consistency

The most grease-like and beneficial greases have an NLGI grade 2-3 consistency or 220 - 295 mm/10 penetration (Rizvi, 2009). The grease can be dislodged from its intended location if the surface is too smooth, and it may move to the intended spot to fail. The findings of the grease consistency study are shown in Table 2. According to the research, a higher quantity of WEO used in grease formulation resulted in softer grease. It is evidenced by the zero consistency of FG85 greases and the five consistency of FG80 greases. It supports Doyle's conclusion that the quantity of thickening used directly impacts the grease's consistency (Doyle, 2015). The trend was consistent when compared to data produced by Japar et al. (2019). According to the results of Japar et al. (2019), the consistency of the grease rose as the fumed silica content increased. However, because Japar et al. (2019) utilised transformer oil for grease formation, the data differs from the findings of this study. Table 2 summarises the findings of this investigation on grease characteristics.

Table 2
Properties of formulated grease

FS grease	NLGI grade	Oil separation (%)	Oil bleeding (%)	
			At ambient	at 70°C
FG_{85}	0	5.7543	-	-
FG_{84}	1–2	4.3875	-16.4835	4.3269
FG_{83}	2	2.9645	-5.0251	1.6304
FG_{82}	3	1.0056	-3.2609	-1.6484
FG_{81}	4–5	0	2.9557	-2.4631
FG_{80}	5	0	2.8846	-2.5126

The consistency of the grease decreased as the ratio of WEO to thickener increased due to a low sponge-like structure that could hold the base oil. The oil became loosely held and easily separated from the thickening matrix system when the attraction between the thickening and the base oil decreased (Japar et al., 2019). When forced, the lower the viscosity of the grease, the more probable it is to spill oil (Abdulbari et al., 2011).

Oil Separation

When the grease was exposed to specific conditions, oil separation, also known as static oil bleeding or puddling, occurred. The low tension causes the grease to leak a small amount of oil. Oil separation can occur spontaneously in greases, and the rate is determined by the grease's composition. Oil separation is a grease that has released oil under static (storage) or typical running circumstances. Small pools of oil indicate static oil separation, especially if the grease surface is not smooth or uniform. Oil separation can happen when oil heated for a long time. This incident might occur either by keeping the grease in a warm atmosphere or by being exposed to pressure and altitude fluctuations within a warm environment storage. Grease having a poor consistency, as compared to firmer grease, is expected to have a higher proportion of oil separation (Zakani et al., 2018).

Table 2 summarises the findings of this experiment, which indicates the percentage of oil separation throughout storage duration. The oil separation values for FG81 and FG80 in Table 2 are unavailable since the grease consistency ranges between 4 and 5, necessitating a high matrix thickening force to hold the oil together. The more solid the grease becomes, the less probable it is to separate from the oil. However, due to oil separation, a thin layer of oil with a consistency of NLGI grade > 4 may be observed on top of the grease. As a consequence, the oil is gathered and weighed. Figure 1 depicts the results of an investigation into the effect of WEO content on grease oil separation. The value of oil separation increases as the WEO content in grease increases. It happened because there was no thickening matrix to keep the WEO contained inside the grease, causing the grease to flow.

According to the research, the oil separation is maximum at 85% WEO concentration, with a 5.75% oil separation. It is because of the high base oil concentration occurring during the formulation process. Since 80% WEO has the lowest base oil concentration, oil separation does not occur during the study. It occurred because the thickening in the grease held the oil together, reducing the potential of the oil to separate from the matrix thickener. A previous study by Japar et al. (2019) also demonstrated that grease with a high FS percentage did not remove the oil from the thickening matrix.

Oil Bleeding

Oil bleeding, commonly called dynamic oil bleeding, is the controlled release of basic oils and additives in response to mechanical and thermal stress. Grease's ability to bleed

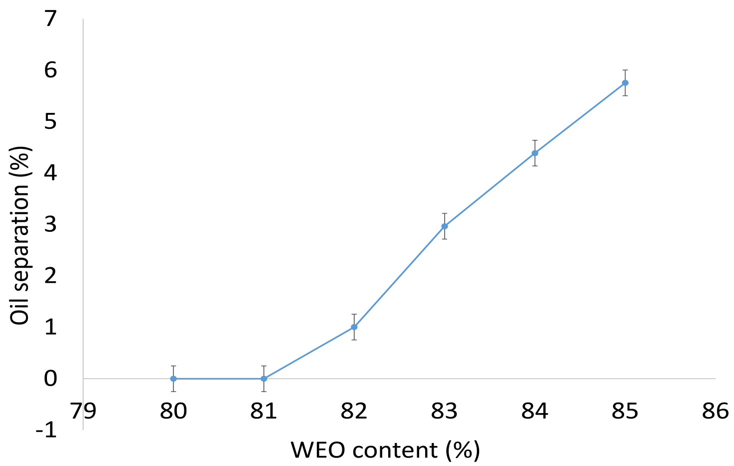


Figure 1. Effect of WEO content on FS greases' oil separation

is important since it lubricates machines at specified temperatures and conditions. On the other hand, uncontrolled oil bleeding will cause grease to leak out, causing the grease to dry out. As a result, a specific percentage of oil bleeding, ranging from -15 to +15%, is optimal (Rahman et al., 2019). A hidden meaning exists between positive and negative signs of oil bleeding values. The positive sign value of oil bleeding indicates that the grease structure is soft and prone to bleed when applied. Meanwhile, the negative sign value indicates the hardness of the grease structure, which is unlikely to bleed even at high temperatures. The oil bleeding rate is quietly related to the amount of thickener in the grease composition. The higher the amount of thickener, the lower the tendency of oil bleeding rates (Gonçalves et al., 2015). This restriction applies to both new and old grease and indicates the grease's ability to operate without re-lubrication.

Table 2 and Figure 2 show the findings of our inquiry on the link between WEO content and oil bleeding. However, due to the 0 consistency of the grease, the oil leakage percentage for FG85 is not calculated. A low NLGI grade may cause grease to leak, resulting in inaccurate figures. The results showed that when the WEO level increased, so did the proportion of FS grease oil leakage. It is because there are only a limited number of fibrous networks of thickening that can help in the retention of the base oil within the grease. Because of the limited number of fibrous networks, grease's resistance to oil flow was reduced, increasing grease permeability (Gonçalves et al., 2015).

One reason for conducting separate analyses at both temperatures (at room temperature and 70°C) is to distinguish the oil bleeding rate between normal and extreme conditions for the old grease. One of the conditions showed the grease characteristics after being used or exposed to high temperatures for a duration, while the other showed how the grease behaved after being stored for a duration of time. The grease condition and ability

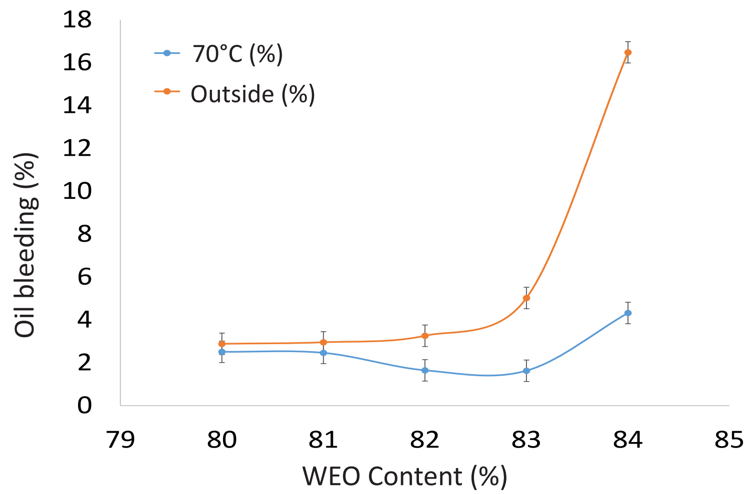


Figure 2. Effect of WEO content on FS greases' oil bleeding

to release oil after high-temperature exposure is expected to decrease compared to normal aged grease due to grease drying out and oil leakage during high-temperature exposure.

Japar et al. (2019) discovered that when heated at 70°C and ambient temperature, the oil leakage of the grease is larger for grease with higher FS concentration. However, this study found the contrary. The results indicated that at 80% WEO concentration, the oil bleeding is 2.5% at 70°C and 2.88% at ambient temperature, while at 84% WEO content, the oil bleeding is 4.3% at 70°C and 16.48% at ambient temperature. It could be due to the oil used, which imparts different qualities to the formed grease. Japar et al. (2019) utilised waste transformer oil as the base fluid in their investigation, which has different qualities from the WEO used in this study.

FTIR Analysis

A useful tool for understanding chemical component availability in certain materials or goods is the Fourier Transform Infrared (FTIR) Spectroscopy. This equipment can also perform quantitative and qualitative organic and inorganic substance analyses. Several chemical compound groupings can be found in the oil, including additives, contaminants, and chemical modifications. The FTIR analysis of a grease sample is used to evaluate its content, such as additives, impurities, product degradation, and components. Figure 3 depicts the FTIR spectrum of the tested FS grease. The FS grease spectrum revealed similarities and differences between the greases. It is because the components used in the grease composition are comparable. The only distinction between the two is the proportion of each constituent.

The spectra of FS greases indicated a strong peak in the 2800 – 3000 cm^{-1} region, showing that C-H stretched (Nabi et al., 2013). The peaks continued in the 1450 – 1460 cm^{-1} spectrum area, exhibiting CN stretch, and the 1350 – 1380 cm^{-1} spectrum area, exhibiting C-H symmetrical bend (Cyriac et al., 2016). The peak on the spectrum 1080 – 2000 cm^{-1} showed the presence of a Si-O-Si stretch inside the FS greases, and the peak on the spectrum 800 – 810 cm^{-1} indicated the presence of an S–S stretch which indicates the presence of diesel that usually exist in used oil as a contaminant as well as thickener inside the grease, and the peak on the spectrum 450 – 460 cm^{-1} indicated the presence of halogen within the grease (Mudalip et al., 2012). However, the grease formulation is expected to have the minimum amount of impurities that have a lower effect on the grease properties.

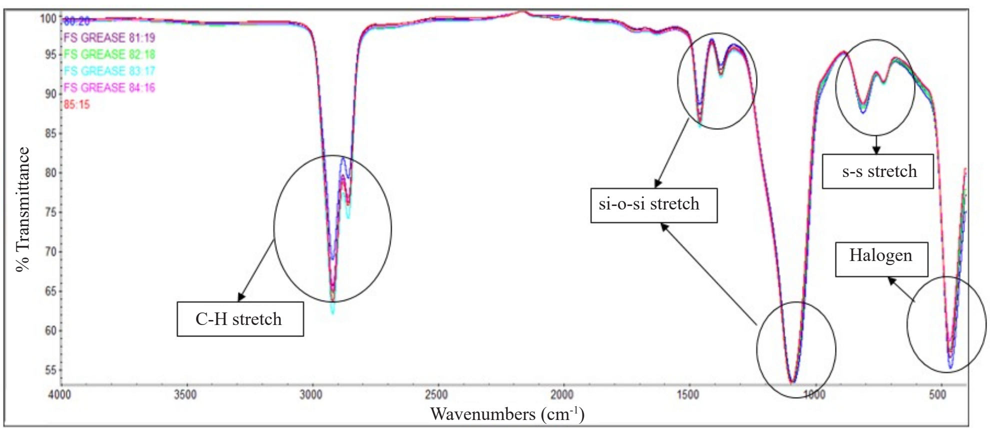


Figure 3. FTIR spectrum of FS greases

Corrosion

A corrosion test can be done to predict the risk of an attack on lubricated goods containing copper or copper alloy. The ASTM D4048 standard determines how corrosive the grease is to the copper strip. The results of corrosion experiments on FS grease are shown in Table 3.

Table 3

FS grease corrosiveness level towards the copper strip

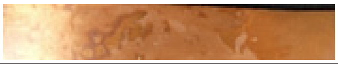
Sample	Results	Strip appearances
Freshly-polished strip		
FG_{85}	1b – slight tarnish (dark orange)	
FG_{84}	1b – slight tarnish (dark orange)	

Table 3 *(Continue)*

Sample	Results	Strip appearances
FG_{83}	1b – slight tarnish (dark orange)	
FG_{82}	1b – slight tarnish (dark orange)	
FG_{81}	1b – slight tarnish (dark orange)	
FG_{80}	1b – slight tarnish (dark orange)	

Table 3 demonstrates that the formulated grease is not corrosive to the copper strip. All copper strips were corrosive in class 1 or above, specifically 1b, where only a small tarnish occurred on the tested copper strip and the strip's colour is dark orange.

CONCLUSION

In terms of WEO content, the grease produced by WEO was also high quality. WEO concentration has been proven to be related to oil bleeding and separation but is inversely proportional to grease consistency. According to the study findings, the FTIR analysis of the grease revealed no contaminant. It proved that the WEO treatment technique could eliminate contaminants found in WEO. Despite achieving only class 1 was achieved throughout this experiment, the formulated grease demonstrated decreased corrosiveness on the copper strip. According to the data acquired throughout the trial, FG83 and FG82 have the most grease-like features, showing that the grease meets the standards for the traits. More study is also required to learn to discover more about the properties of grease.

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