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Preparation and characterization of grease formulated from waste transformer oil

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Abstract. Grease is known as a lubricating material in a form of solid to semisolid mixture which is produced with the combination of base oil (80% - 95%), thickener (2% - 15%) and additive (0% - 10%). The limitation of soap grease performances as well as the volatile oil prices worldwide has led the interest in seeking alternative grease formulation. This research is focusing on the formulation of non-soap based grease from waste transformer oil (WTO) by two types of non-soap based thickeners, i.e. fumed silica and bentonite. The grease is prepared using different ratios of WTO and non-soap thickener ranging from 90:10 to 65:35. The physical and chemical properties of the formulated grease were evaluated using the ASTM International grease testing standard – for consistency and oil separation, and using the thermalgravimetric analyzer (TGA) and Fourier Transform Infrared Spectroscopy (FTIR) – for thermal stability and composition identification. It was found that the grease consistencies were directly proportional to the amount of thickener and the oil separated from the grease will affect the grease stability. Greases were found thermally stable at temperature as high as 150 °C. From the overall test, the best formulation was the grease formulated using bentonite with the ratio of 80:20 with NLGI grade 2 consistency.

1. Introduction

Grease is a solid to semisolid mixture which is produced with the combination of base oil, thickener, and additive with the range ratio of 80% to 95%, 2% to 15% and 0% to 10% respectively [1]. Grease also is one of the oldest forms of lubricating material and in early 2000, grease was made from environmentally friendly and biodegradable materials. The formulated grease provides satisfactory performance in most of the industrial applications. The important factors that affecting the properties and characteristics of grease are the amount and type of thickener used, the viscosity and physical characteristics of the oil, and the additives. The grease is fundamentally designed to separate moving parts, decreasing the friction between two moving parts to minimize wear and also reducing the heat generation [2]. Variation ratio of base oil, thickener and formulation process, producing different grease properties and specialty. The Association of Southeast Asian Nations (ASEAN) countries, is further contributing to the growth of the lubricant consumption as a whole and grease in particular, in the region [3]. Over the next two years, the compound annual growth rate for industrial grease demand will be a measly 0.3 percent, bringing global consumption to 691,000 tons in 2019 [4]. There are several reasons to get involved in this field of formulating the non-soap based grease from waste transformer oil (WTO).

The first reason is characteristic of soap-based, secondly is economy and availability of raw material which is petroleum and lastly is environment.

Non-soap grease usually reserved for specialist application and it is costly than soap greases [5] thus, this type of grease is not commonly used compared to soap greases. However, it has distinct properties that most soap greases do not have, unless with the presence of selected grease additive. For example, non-soap greases are thermally stable thus, it is able to withstand wider temperature range. Some of the non-soap greases also have excellent water resistance as some of the non-soap thickeners is water insensitive and corrosion resistance. Normally, such properties are apparent in most soap greases only by the help of grease additive for enhancing the grease properties or adding the desired properties. In general, non-soap thickener is known for its key characteristics which are excellent performances over high temperature, water resistance, and good thermal and oxidative stability [6].

The petroleum is classified as one of the non-renewable resources and the oil prices are currently unstable worldwide. This study is providing information on the possibility of formulating alternative grease from waste oil generated by the power industry (waste transformer oil). This could possibly help in reducing the pollution caused by the waste oil – due to illegal disposal [7], as well as in reducing the overall cost of grease production [8].

Due to these reasons, the distinctive non-soap thickener properties, waste oil-related pollution, and the volatile oil prices, this study aimed to investigate the potential of converting the waste transformer oil (WTO) into valuable end products which are non-soap based grease. In order to achieve this, different ratios of WTO and thickener were being tested and analyzed using chemical and physical analysis to ensure the suitability of the grease produced.

2. Materials and methodology

All materials used, the process of preparing the grease, and the characterization of grease were discussed in the following subchapters.

2.1. Materials

The formulation of grease was based on the varies ratio of three main components which are base oil, thickener, and additives. In this study, waste transformer oil (WTO) was used as base oil. Initially the oil was pre-treated in order to eliminate the impurities. For the thickener, two different types of non-soap based thickener were used (bentonite and fumed silica). Thickeners are molecules, polymers or particles that are partially soluble in lubricating fluid. Different types of thickener will affect the grease performance mainly in dropping point, water-resistance and grease consistency.

2.2. Pre-treatment of waste transformer oil

This process is one of the important steps in this study to ensure that the waste transformer oil was free from any impurities and in a good condition before went through the grease formulation process. Two major steps involved in this process were filtering and heating. The filtering process was done by applying the vacuum filter to ensure the waste oil was free from any impurities or particulates such as solids and inorganic material [9]. After that, the waste oil went through the second pre-treatment process which was heating or dewatering process. Waste oil was heated at high temperatures up to 100 °C with continuous stirring for 2 hours to eliminate the water content.

2.3. Preparation of grease

Table 1 and Table 2 show the ratio of the non-soap based grease formulation for two different types of thickener namely, bentonite and fumed silica. The formulation consists of 4 major steps. Initially, the waste transformer oil was pre-treated process. After that, waste transformer oil and thickener are weighted according to the ratios. The waste oil will be heated at temperature 120 °C while stirring using the homogenizer to ensure the oil is free from water. Then, thickener will be added slowly into the solution and the speed of homogenizer need to be adjusted for the homogenizing process for at least 1 hour.

Table 1. Bentonite grease formula.

WTO (%)	Bentonite (%)
90	10
85	15
80	20
75	25
70	30
65	35

Table 2. Fumed silica grease formula.

WTO (%)	Fumed silica (%)
94	6
92	8
90	10

2.4. Characterization of grease
The formulated grease will undergo several physical testing such as consistency test, oil separation test, thermogravimetric analysis and also FTIR analysis in order to identify the characteristic of grease itself.

2.4.1. Consistency. Consistency test (ASTM D-217) is defined as the degree to which a plastic material resists deformation under the application of force. In the case of lubricating greases, this is a measure of the relative hardness or softness and has some relation to flow and dispensing properties. Harder and softer grease will be depending on the grease consistency number or NLGI (National Lubricating Grease Institute) class and also will produce different characteristics of grease itself. Grease will release some of lubricating oil when the mechanical stress had been applied. This oil is squeezed out of the thickener matrix and will be delivered to the lubrication points. The point of the oil release is to provide lubrication, film formation and as well as wear prevention that will make sure the optimum performance of the equipment. The higher the penetration number, the softer the texture of grease.

2.4.2. Oil separation. Oil separation test (ASTM D-1742) also known as stability test upon storage which basically to determine the ability of a grease to separate oil during storage and to also the stability of the grease formulated. An oil separation test monitors the separation of oil from grease at elevated temperatures for a defined period of time. The container is then left at room temperature for one month. The tendency of oil to separate during storage can be an important characteristic. When the base oil begins to separate from other raw materials in grease, the remaining material may change in consistency and potentially affect the ability of the grease to function as designed. In general, the lesser the amount of oil separated, the better the stability and the longer the lifetime of the formulated grease.

2.4.3. Thermogravimetric analysis. Thermogravimetric analysis (TGA) is categorized in the family of thermal analysis techniques used to characterize a wide variety of materials [10]. TGA measures the amount and rate which is the velocity of change in the mass of a sample as a function of temperature or time in a controlled atmosphere [11]. The results are used usually in order to determine the thermal or oxidative stabilities of the materials as well as their compositional properties. This analysis has the capability to predict grease high-temperature life and designed to be a comprehensive method of oil bleeding. Currently, many standard analysis methods are available to identify the thermal stability of grease, but none of them provides comprehensive data related to the oil separation and evaporation of greases within a short time [11]. Next, this analysis also able to applied to select materials for certain end-use applications, predict product performance and improve product quality. Details that can be gained from this analysis such as thermal stabilities, oxidative stabilities, estimation product lifetime and moisture and volatiles content [12]. The analysis can be carried out in an inert atmosphere such as nitrogen or a reactive atmosphere such as oxygen. In general, a few milligrams of the sample were weighed and heated under controlled conditions with a heating rate. The higher the temperature for decomposition of waste oil, the higher the thermal stability of formulated grease.

2.4.4. FTIR analysis. Fourier Transform Infrared Spectroscopy (FTIR) is the analysis to analyze the functional group that exists inside the grease and oil. The FTIR will provide specific information on the molecular structure and chemical bonding of the grease that it is useful to identify the organic and inorganic material involve in the substance. The principle of FTIR is based on there being molecules present in a lubricant that absorb infrared light to different degrees because of their chemical structure. The sample of grease is applied to an attenuated total reflectance (ATR) cell. In the contact zone, the grease will expose to the infrared light and the infrared spectrum will show the corresponding wavenumber based on the absorption of infrared light.

3. Result and discussion

3.1. Grease consistency

The consistency of the grease is basically depending on the result according to the National Lubricating Grease Institute (NLGI) class. This is a measure of the relative hardness (NLGI class:6) or softness (NLGI class:000) and has some relation to flow and dispensing properties. whether the grease is soft or hard, it does not mean that the grease cannot be used. The stiff greases usually used in very high speed and low load applications [13]. The higher the NLGI numbers, the higher the consistency and the grease is suitable for the application of journal bearings with low speed, high operating temperature and to avoid any water washout. On the other hand, the lower the NLGI numbers, the lower the consistency and the grease is usually used for low speed rolling element bearings and cold temperature operation [13].

Tables 3 and 4 show the results gained on the consistency class of each formulated grease. Greases that categorized under NLGI grades of 0 to 1 were bentonite grease with ratio 85:15 and fumed silica grease with ratio 94:6. This type of grease has a very soft to soft texture and suitable for the application of centralized lubrication systems with low temperatures. The grease which is categorized under NLGI class of 2 to 3 is bentonite with ratio 80:20, 75:25, 70:30 and fumed silica grease with ratio 92:8. This type of grease has medium soft to medium texture. Lastly, grease with NLGI class of 4 are bentonite grease with ratio 65:35 and also fumed silica grease with ratio 90:10.

Table 3. Bentonite grease consistency and oil separation.

WTO (%)	Bentonite (%)	Consistency (NLGI)	Oil separation (%)
90	10	-	0
85	15	0 – 1	0
80	20	2	0
75	25	3	0
70	30	3	0
65	35	3 – 4	0

Table 4. Fumed silica grease consistency and oil separation.

WTO (%)	Fumed silica (%)	Consistency (NLGI)	Oil separation (%)
94	6	-	0
92	8	3	0
90	10	3 – 4	0

The commercial greases available nowadays have a consistency ranging from NLGI grade 1 – 4. Most commercial greases are commonly formulated with NLGI grade 2 consistency because, at this consistency, the grease has the most grease-like semi-solid appearances. The present study found that only bentonite grease with ratio of 80:20 is having such consistency. Thus, bentonite grease with ratio of 80:20 is selected as the best grease formulation.

3.2. Separation of oil from grease

The oil separation test was carried in order to identify the tendency of oil to be separated from grease during the storage in accordance with ASTM D-1742 [14]. An oil separation test will monitor the separation of oil from grease at elevated temperatures for a defined period of time. The rate of oil released from the grease will increase with time and vary based on the temperature at which it is stored [15]. When the base oil starts to separate from other raw materials in grease, the remaining material may change in consistency and potentially affect the ability of the grease to function as designed. The sample of formulated grease is stored in an encloser is then leave at room temperature for a month [9].

Tables 3 and 4 show that all formulated greases have no oil separation. Both types of thickeners used which categorized as organo-clay and standard silica have an oil keeping ability and outstanding heat resistance which needs a longer time to do the oil separation process [16].

For bentonite grease with a ratio of 90:10, the inexistence of separated oil was due to the liquid-like appearance of the grease for which indicated that no grease structure is formed. At this ratio, the thickener is dispersed and dissolved in the WTO. This finding is due to the insufficient thickener to form 3D thickener networks to absorb the WTO into the thickener structure [17]. However, the grease structure is forming with the presence of sufficient thickener and able to hold the oil thus resulting in no oil separation over time.

3.3. TGA of grease

According to figure 1, the sample of formulated grease has high thermal stability as the grease is able to operate at higher temperatures. The TG curve presented two weight loss steps, which are at temperature 194.08 °C and 380.44 °C respectively. The WTO started to decompose at temperatures above 150 °C and the quality loss of WTO occurs when the temperature is raised to 150 °C under normal pressure. The loss rate of WTO is gradually increased accompanied by temperature rise. The decomposition accelerated when the temperature keeps increasing until more than 200 °C. Generally, the first decomposition happened at temperature range 190 °C – 360 °C due to the elimination of low molecular weight of sample. The WTO completely decomposed at a temperature 380 °C. This result shows that most of the volatile components in WTO will be vaporized at a temperature above 200 °C and almost all of the oil had vaporized at 490 °C. Thus, WTO can be used at operating temperatures up to 150 °C without undergoing significant vaporization. Other than that, the grease formulated is categorized under medium volatile as the product able to withstand at more than 150 °C and does not evaporate.

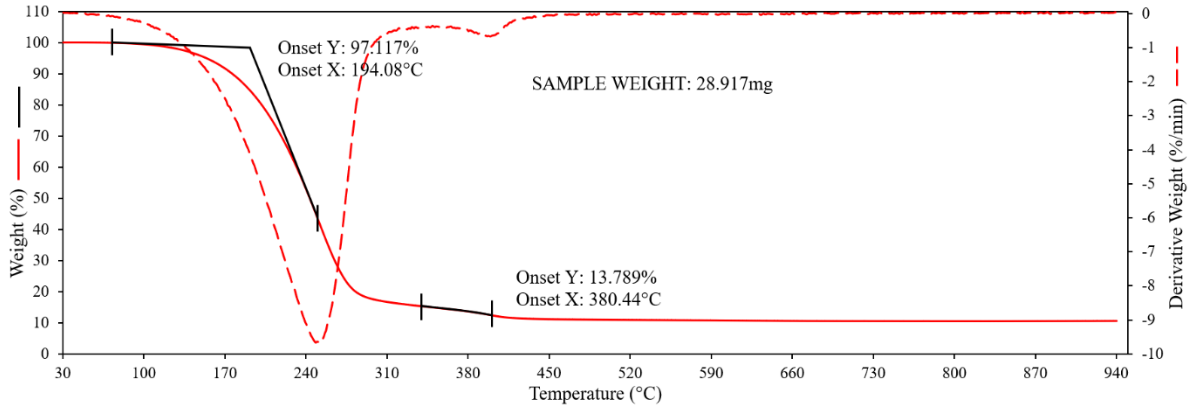

Figure 1. TGA of bentonite grease with 80% WTO.

3.4. FTIR characterization

Figures 2 and 3 show the FTIR peaks of bentonite and fumed silica greases, respectively. Both greases have peaks at wavenumber between 400 cm^{-1} to 600 cm^{-1} and 900 cm^{-1} to 1500 cm^{-1} which represent a C-O functional group, and at 2700 cm^{-1} to 3000 cm^{-1} indicating the C-H functional group.

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According to the analysis, the intensity of absorbance peaks at 2922 cm^{-1} and 2854 cm^{-1} increase with aging due to the adsorption process of mineral oil to the paper surface during aging. The major degradation of the aging mechanism in mineral oil is oxidation. Oxidation will decompose the hydrocarbon molecules into other substances such as hydroperoxide, alcohol, aldehyde, ketones and also esters.

The oxidation reaction of mineral oils also will produce carboxylic acids (R-COOH) that bind with alcohol in order to generate esters. During thermal aging process, the carboxylic acids generated from the oxidation process of mineral oil can react with hydroxyl groups on the cellulose forming a cellulose graft polymer via a condensation reaction. Other than that, Bentonite which is acts as a thickener were found at the wavenumber 1034 cm^{-1} in figure 2 [18].

From figure 3, the wavenumber produced at 3000 cm^{-1} represents the fumed silica thickener content in the grease sample [19]. The other objective of this analysis is to determine the water content in the sample and the wavenumber for water is at 3600 cm^{-1}. Both of the sample have lesser water content which proved that the grease is a good thermal ability product.

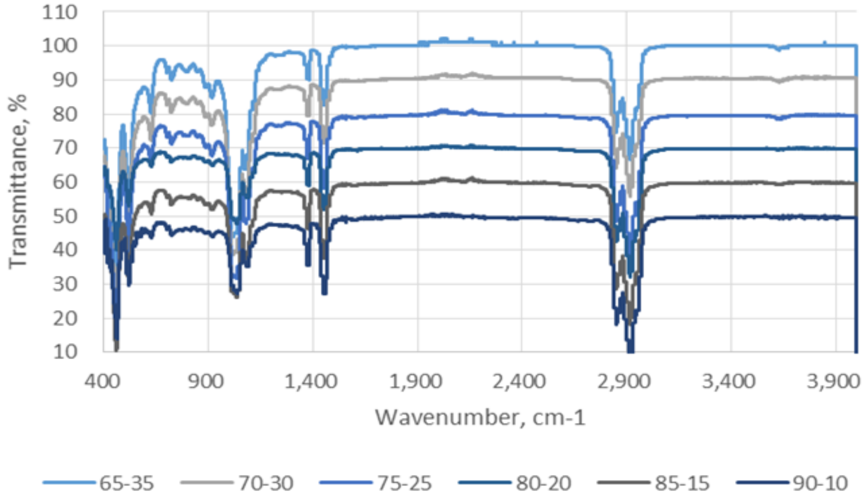

Figure 2. FTIR spectrums of bentonite greases.

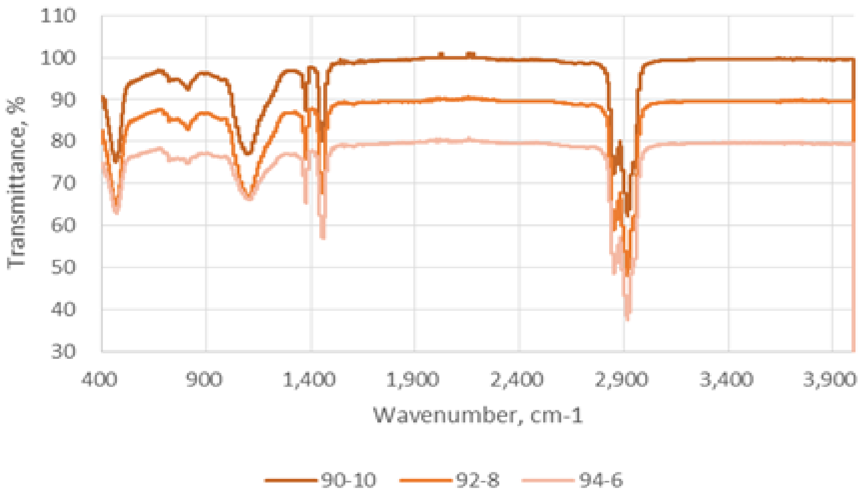

Figure 3. FTIR spectrums of fumed silica greases.

4. Conclusion

In this research, grease from WTO as the base oil was formulated successfully by using two different types non-soap thickener which is bentonite and fumed silica. The best grease formulation is bentonite grease with ratio of 80:20 (WTO: bentonite) because it has the consistency of NLGI grade 2 that is comparable to most commercial greases. The other formulated greases are classified as NLGI grade 3 – 4 grease with no oil separation during storage. The WTO and the greases can be exposed to operating conditions as high as 150°C or above, respectively, without substantial evaporation or degradation. It is proven that the WTO could be used as alternative base oil in grease formulation and at the same time, reducing the dependency of lubricant towards conventional mineral oil.

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