

Biodegradable Grease from Palm Oil Industry Wastes

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Abstract

This paper presents an experimental grease formulation made from spent bleaching earth (SBE) as the thickener, waste cooking oil (WCO) as the base oil and fumed silica as the additive. The properties of the different grease formulations, such as the thermal stability, the decomposition temperature, the penetration, the corrosivity on a copper strip, the drop point, and the friction-coefficient, were evaluated by standard methods and the grease formulations with and without the additive were compared. The results show that the grease without fumed silica required a high percentage of SBE (up to 80% w/w) while the addition of the fumed silica reduced the amount of SBE and increased the amount of the base oil. Fumed silica increased the penetration number of the grease by 1 standard (NLGI standard), made the grease slightly corrosive, eliminated the drop point, and increased the decomposition temperature. The friction coefficient of the formulated grease had an average value of 0.095 without fumed silica and 0.11 with fumed silica. The overall results show that SBE and WCO can be used to formulate grease and that fumed silica can increase the performance of the formulated grease.

Keywords: Grease; Tribology; Vegetable oil; Thermal stability; Corrosion

1. Introduction

Malaysia is known as one of the major palm oil producers in the world [1]. Bleaching earth is massively used in this industry especially in the Pre-treatment of crude palm oil during. Bleaching earth is usually used to remove unwanted odors and dark colour matters due to its high absorption properties [2]. Every year, an estimated 600,000 metric tons of spent bleaching earth (SBE) is produced worldwide and discharged into the environment [3,4]. An estimated 195,000 tons of SBE is generated in Malaysia and is generally disposed of in landfills [5]. The accumulated amounts of SBE is considered as a serious potential threat to the environment due to the complex nature of the components it carries after crude palm oil refining process and the high cost of recycling. On the other hand, vegetable oils are known to provide excellent lubricity due to their ester functionality. The polar heads of the fatty acid chains attach to metal surfaces by a chemical process that allows a monolayer film formation with the non-polar end of fatty acids sticking away from the metal surface. The fatty acid -CH₂- chain offers a sliding surface that prevents the participating metal from making direct contact with each other. When a film is not formed, contact may result in rising temperature at the contact zones of moving parts causing adhesion, scuffing or even metal-to-metal welding. An estimated 0.5 million tons of waste cooking oil is produced in Malaysia annually from food-related industries and household use [6]. Lubricant's (greases) primary function is to control friction and wear [7,8]. Grease is usually consisted of at least two basic ingredients, which are base oil and thickening agent (thickener) [9-11]. Normally, additives are added to improve the performance of the grease [12]. Most of the greases these days are derived from petroleum sources such as mineral oil and synthetic fluids as base oil [9-11]. Generally, several types of greases exist in the market which are soap greases, non-soap grease, synthetic grease (organic ester, polyglycol, and silicones), and bio-based greases. However, mineral oils thickened with metallic soaps, silica, and bentonite or poly urea derivatives [13] are the main ingredients for greases used in modern industrial applications. The use of petroleum base lubricant has created an argument over the impact and feasibility of petroleum based source towards the environment and economy as petroleum is toxic to environment and a major cause of pollution. The non-biodegradable properties of mineral oil could be deleterious to nature since it can devastatingly pollute water and environment (Adhvaryu and Erhan, 2002). Thus, vegetable oil seems to be a promising candidate to replace petroleum oil or mineral oil as base oil and is a cheaper option compared to synthetic oil. Utilization of industrial wastes into added value products would eliminate the potential hazardous of these wastes. At the same time, the biodegradable lubricating grease produced from industrial wastes offers a cheap alternative that is likely to meet the

standard of commercial industry lubricant standard and to ensure the sustainable of local environment from the effect of non-biodegradable lubricating grease. Multipurpose grease is a type of grease designed to fit into various operating conditions. In comparison with synthetic or specialty grease, multipurpose grease is designed to be able to operate at extreme conditions such as high temperature or cold temperature climate, extreme pressure, or in niche market such as medical facilities and air-craft. Multipurpose grease normally would suit a working condition of most of the application where the need of grease is necessary.

Despite the wide studies on the performance of bio-grease, there was no researchers brings the innovative nor innovation in converting the waste into the products is reported, especially in the lubricating greases. In this present work, waste cooking oil (WCO) and spent bleaching earth (SBE) from vegetable oil refinery mills were utilized and mixed to formulate a lubricating grease and its performance was evaluated in a multi-purpose grease formulation. WCO is a good substitute for mineral oils and it can be used without any additional processing steps resulting in a cost reduction of cost. Bleaching earth is derived from natural clays, most of which are from bentonite type clays. Bentonite clays have excellent rheological and absorbent properties with excellent plasticity and lubricity, high dry bonding strength, high shear and compressive strengths, good impermeability and low compressibility [15]. Both these raw materials are renewable resource sources that can be found locally and they signify an alternative to fossil fuels for grease formulation. Fumed silica as an additive was added to the grease formulation as stabilizing agent and its effect was also examined. The grease formulations were also characterized using, thermal analysis, penetration test, copper corrosion test, drop point test, and the friction-coefficient using standard method.

2. Methods

Spent bleaching earth (SBE) was collected from a local palm oil refinery (FELDA Vegetable Oil Sdn Bhd), and waste cooking oil was collected from various sources on the University Malaysia Pahang campus. The SBE was used in its original condition, and the WCO collected was analyzed to determine the properties of the oils. Prior to analysis, the oil was filtered to remove solid particles and was kept in an air-tight container to avoid further oxidation and moisture. The oil analyses that were carried out included fatty acid composition, percentage of free fatty acid (FFA), acidity, density, thermal stability, and viscosity. The properties and methods are described in the table below.

2.1 Waste analysis

Both WCO and SBE were analyzed prior to use to determine their properties and characteristics. The results of the analysis are presented in tables 3 and 4, and figures 1, 2, and 3.

2.1.1 WCO analysis

For WCO, the analyses included fatty acid composition, density, melting point by DSC, decomposition temperature by TGA, moisture content, acidity as free fatty acid value, kinematic viscosity and viscosity index.

2.1.1.1 WCO fatty acid composition

The fatty acid composition of the methylated reaction products was analyzed using GC/MS (Agilent 5975 Inert XL MSD coupled to a 7890 A gas chromatograph). The analysis was performed with a DB-1MS capillary column (L, 30 m; i.d., 0.25 mm; thickness of film, 0.25 μm). The column temperature was programmed to increase from 185°C to 210°C at 2°C.min⁻¹, to hold at 190°C for 7 min, and then to increase again to 230°C at 3°C.min⁻¹ and hold for 6 min with helium as the carrier gas. The fatty acid composition of the products used in this study is shown in table 3.

Table 1 Fatty acid composition of WCO

Fatty acids	Percentage (%)
C12:0	-
C14:0	0.1
C16:0	13.7
C16:1	-
C18:0	-
C18:1	24.26
C18:2	49.82
C18:3	3.81
C20:0	0.45
C20:1	0.47
C22:0	-

2.1.1.2 Density

The density was measured using a computer controlled gas pycnometer (AccuPyc 1340 Gas Displacement Density Analyzer, Micromeritics, USA) operating at 25°C. A supply of high purity helium gas was used as the medium for the density measurement. The density measurements were performed with a standard 20 cm³ specimen chamber using 10 purges for a chamber followed by 3 measurement cycles. The density measured for the WCO was 0.888 g/cm³.

2.1.1.3 Heat flow analysis by Differential Scanning Calorimetry (DSC)

The starting materials were measured using Differential Scanning Calorimetry (DSC) (TA instruments, TA-Q1000) under nitrogen gas flow. The cooling rate was 10°C/min for samples in sealed hermetic aluminum pans for a temperature range of 50°C to -40°C. Liquid specimens weighing approximately ±5 mg were placed in an aluminum sample pan under nitrogen at a flow rate of 100 mL/min. DSC was used to determine the temperature at which the waste cooking oil starts to freeze and melt by observing the heat flow. Figure 1 shows the result of the oil analysis by DCS. The figure shows the oil start to freeze at a temperature of 0.73°C, putting the lower limit of operation at about 0°C for using waste cooking oil as the base oil.

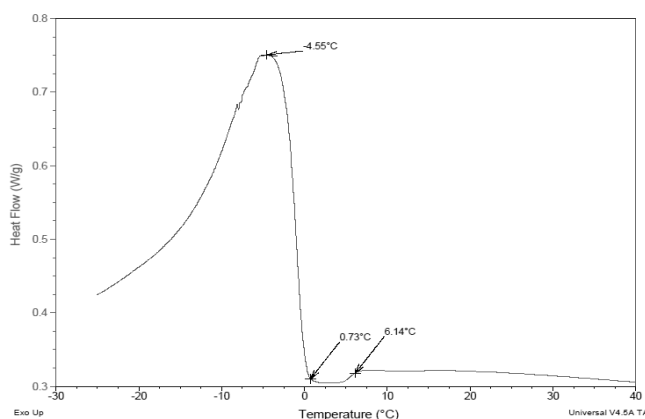


Figure 1 Exothermic DSC plots of used WCO

2.1.1.4 Thermal analysis by Thermogravimetric Analyzer (TGA)

TGA measurements were carried out using TA instruments, TGA Q500 Thermogravimetric Analyzer. Each sample weighed about 10 mg (±5 mg) at a scanning temperature range of 25°C-900°C and a heating rate of 10°C/min. TGA was conducted by placing the compounds in platinum crucible under nitrogen atmosphere at a flow rate of 100 ml/min to avoid unwanted oxidation.

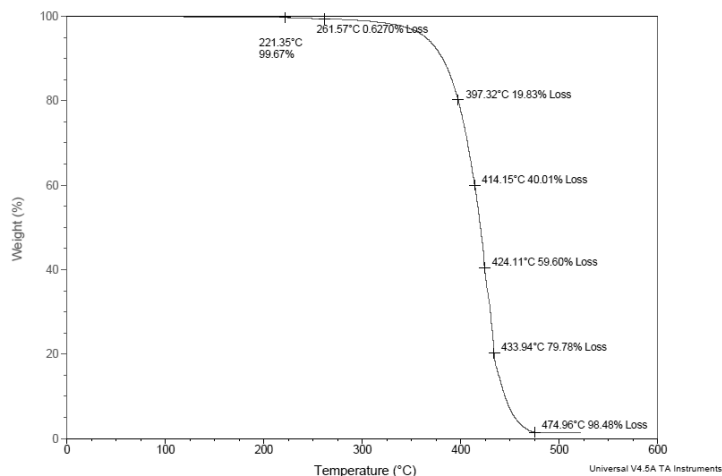


Figure 2 Thermal stability of WCO.

Figure 2 shows the thermal stability of the WCO. Waste cooking oil started to decompose at temperatures above 300°C. The decomposition accelerated when the temperature exceeded 350°C. The WCO completely decomposed at a temperature of 470°C. This result shows that most of the volatile components of the WCO vaporized at temperatures above 350°C and almost all the oil had vaporized at 500°C. Thus, WCO can be used at operating temperatures up to 300°C without undergoing significant vaporization.

2.2.2 SBE analysis

2.2.2.1 Particle size analysis

The SBE particles' size was measured using a computer controlled Malvern Mastersizer 2000 (Scirocco 2000) using the dry method. The measurements determined the particles' size uniformity and the distribution of the SBE particles. The particles' size distribution was determined by entering specific properties of the particles. The diffraction index used was 1.533, and the measurement time was set to 20 seconds by laser diffraction. Approximately 1 g of the SBE sample was used for this measurement. Laser diffraction reported the results as a volume equivalent spherical diameter. The SBE particles' size was estimated to range from 5 µm to 20 µm.

2.2.2.2 Density

The density of the SBE was measured using a similar procedure as described for measuring the density of WCO. The density of the SBE measured at 25°C was 0.8899 g/cm³.

2.2.2.3 Thermal stability of SBE by TGA

TGA measurements were carried out using TA instruments, TGA Q500 Thermogravimetric Analyzer. SBE samples (~10 mg) were measured at a scanning temperature range of 25°C-950°C and a heating rate of 10°C/min. TGA was conducted with the compounds placed in a platinum crucible under nitrogen atmosphere at a flow rate of 100 ml/min. Figure 3 shows the result of the thermal stability of SBE for low and high temperature conditions, which will set the operation limit of the formulated grease because the base oil determines the major function of the grease. Using SBE as a thickener has advantages because of its high temperature stability.

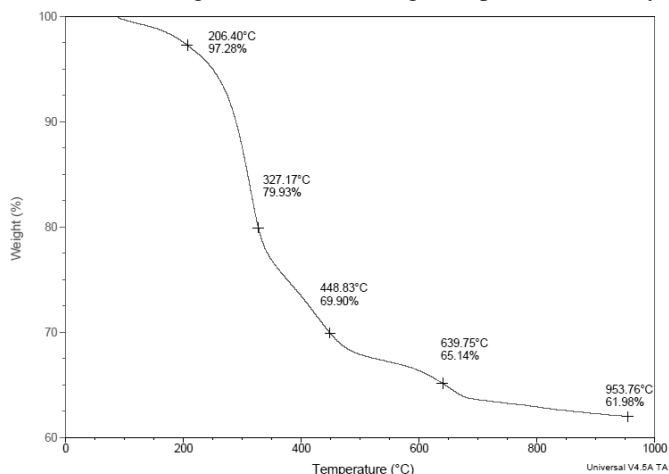


Figure 3 The decomposition and thermal stability of SBE

2.2 Fumed silica

Fumed silica (SiO₂) (Sigma-Aldrich) was used as a rheology modifier and stabilizing agent to improve the dispersion of the SBE particles. Fumed silica (FS) had a surface area of 200±25 m²/g, a mean particle size of 0.014 µm, a melting point above 1600°C and a density of 2.3 lb/ft³ (0.0368 g/cm³) at 25°C.

2.3 Grease formulations

This experimental grease formulation used WCO and SBE as its main components. The influence of additives on the base grease was assessed. The lubricating grease from WCO and SBE was formed by mixing the thickener slowly into the base oil until it became viscous and non-Newtonian. The SBE was mixed and dispersed in an adequate and proportional manner into the WCO. The mixture was vigorously mixed for several hours to homogenize the formulated grease. WCO (fixed weight for all formulations) was poured into a 2000 ml steel container and was placed under an overhead stirrer equipped with a steel propeller type impeller. SBE that had been sieved and properly weighed was then added into the container gradually under slow agitation (200 rpm) before ramping up the mixing speed to between 1000 rpm and 1500 rpm to facilitate and speed up the mixing and homogenizing of the components. The base grease was composed of 20-30% base oil and 70-80% thickening agent (w/w), which acted as the foundation for a basic grease. The mixing procedure was carried out at room temperature (25°) until the SBE and WCO were fully mixed (approximately 2-3 hours). The base grease was then collected and underwent the penetration test to assess the rigidity of the formulation. The fully mixed base grease was then mixed with a small percentage of fumed silica (variable weight depending on the formulation) until all of the fumed silica was homogenized into the base grease. However, subsequent grease formulations could have all three components mixed (base oil, thickener and the additives) simultaneously once the penetration and the final formulation is known for use in the other analyses. The properties and performance of the various grease formulations were evaluated by various methods in the chemical engineering lab at UMP and at the Lanzhou Institute of Solid Lubricant lab at the Chinese Academy of Sciences, Lanzhou, China.

2.4 Lubricating grease analysis

Analytical testing determined the grease's properties and the conditions for which it was suited. Determining the final properties of the formulated grease is crucial to predict the grease formulations' functions in different applications.

2.4.1 Penetration test

A penetrometer was used to evaluate the hardness of the formulated greases as described by ASTM D-217. A stainless steel tip brass cone (45° cone, weight = 102.5 g) was allowed to penetrate the grease for 5 sec and was repeated 3 times. The average results obtained were compared with the National Lubricating Grease Institute (NLGI) standards. The NLGI grade corresponding to the cone penetration value for the formulated grease was subsequently used in the Results and Discussion. The base hardness for the evaluated grease should be within standards 1, 2 or 3. The selection of grease was made by mixing the appropriate percentages of SBE and WCO until the desired hardness was obtained.

2.4.2 Copper Corrosion Test for WCO (ASTM D-130) and formulated grease (ASTM D-4048).

The oil samples were heated for 1 h at temperatures between 100°C and 120°C prior to using them in the test to remove most of the moisture that could influence the corrosiveness of the oils. The copper strip test was performed to measure the corrosiveness of waste cooking oil. The test conditions followed the guidelines in ASTM D-130 for lubricating oil. The copper strip was immersed in the WCO at 100°C for 3 hours. The color change of the copper was then compared with the standard. For formulated grease, the guidelines in ASTM D-048 (Standard Test Method for Detection of Copper Corrosion from Lubricating Grease) were used to assess the corrosivity of the lubricating grease formulations. A prepared copper strip was completely immersed in the test grease and was heated at the specified conditions, usually 100°C (210°F) for 24 h. At the end of the test period, the strip was removed, washed, and compared with the ASTM Copper Strip Corrosion Standards.

2.4.3 Tribology test

The tribology properties of the grease were evaluated with help from Lanzhou Institute of Solid Lubricants, China. The tribology properties of the lubricating grease significant contribute to the performance of the grease during operation. The tribology studied here only focuses on the friction coefficient and the effect of fumed silica on the grease tribology. The tribology test was performed as described by ASTM D-2596 by conducting wear tests at a rotating speed of 1450 rpm (linear speed of 33.38 m min⁻¹) and a load of 392 N for 30 min. The ambient

temperature was about 15°C. The friction coefficient of the formulated grease was recorded automatically using a strain gauge equipped with the four-ball tester by the SRV test machines used.

2.4.4 Drop point test

The drop point of the grease was evaluated to further investigate its stability at high temperatures and was performed as described by ASTM D-2265 (Standard Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range).

2.4.5 Thermal analysis (TGA)

The grease was assessed by thermal analysis using a TGA Q500 Thermogravimetric Analyzer under a 100 ml/min nitrogen gas flow with an open platinum pan. The temperature ramped up at 10°C/min until it reached 950°C.

3. Results and discussion

3.1 Grease formulations

A lower percentage of SBE (50% to 60%) required a higher percentage of fumed silica (3% to 5% of fumed silica). From the formulation, the addition of fumed silica was made by adding from 1% of fumed silica for every 15% SBE (w/w). The addition of fumed silica significantly changed the grease hardness because of its low density and very small particle size. The same weight of fumed silica takes up a larger volume than SBE because its density is much lower than that of SBE. Using fumed silica reduced the SBE amount based on weight but significantly increased the amount of base oil used. In this study, it was possible to increase the amount of base oil up to 70% (w/w) of the total amount of the grease by adding fumed silica to yield the same penetration grade as a 25%-30% base oil with 75%-70% thickener (w/w) (table 5). The penetration test was used to evaluate the hardness of the grease, which is important because the lubricating grease should behave as a non-Newtonian material [16], but the semi-solid structure of the grease has a tendency to flow. Of the formulated greases, those that fell within the hardness NLGI standards 2 [9,16] and 3 [16] exhibited the most grease-like behaviors and functions. These basic behaviors are also important to ensure that the formulations act as a semi-solid grease rather than a hard grease, which would indicate a lack of base oil and would behave similar to a solid lubricant. However, solid lubricants are not the focus of this study. Conversely, if the grease was too soft, the oil would have an overarching tendency to separate from the thickener phase and to drip. Most grease is semi-solid. The penetration level is measured by the depth that the cone digs into the grease. The level of dispersion of the SBE into the WCO is crucial in determining the penetration level. The homogeneous mixture of these materials helps preserve the mechanical stability and consistency of the grease. Thus, the SBE and WCO need to be completely homogenized to avoid oil separation and sedimentation during storage. This phenomenon can be avoided by adding fumed silica as a modifier to support the strength of the grease during operation and improving its stability. Greases need to be thermally stable, resistant to oxidation and mechanically stable during operation.

Table 3 Percentage of thickener and base oil, oil separation and NLGI standard

Thickener (%)	Base Oil (%)	NLGI Standard
Below 60	More than 40	Too soft, immeasurable
60	40	0 to 1
65 to 70	35 to 30	1 to 2
70 to 75	30 to 25	2 to 3
75-80	25-20	2 to 3
More than 80	<20	3 to 4

Table 6 shows the formulations of the greases and the percentage of the base grease and fumed silica and the resulting NLGI standard classification from the penetration test. A minimum of 60% thickener was required to form a grease with a firm structure, although 65% thickener was required to obtain a reasonable hardness in the

mixture structure. Too soft of a structure would cause the entire grease to flow [17] and behave as a Newtonian liquid.

Table 4 Penetration of formulated greases

Formulation			Penetration
Base Oil	Thickener	Additives	
Waste Cooking Oil	Spent Bleaching Earth	Fumed Silica	NLGI standard
20	80	0	2 – 3
23.5	75	1.5	2
23.6	75.6	0.8	2
24	76	0	2
25	75	0	1-2
29	71	0	1
30	70	0	0
40.85	55.45	3.70	3
65.5	17.25	17.25	4
66	17	17	4

The formulation of grease based on the percentage (w/w) of these three ingredients was crucial in determine the rigidity and NLGI standard of the grease. Adding fumed silica at various concentrations increased the grease’s rigidity, which showed that fumed silica provided strength to the grease structure.

Table 5 Drop point for the formulated greases

Formulation ([base oil]/[thickener/fumed silica])(w/w (%))	Drop point temperature
20/80	> 350 °C
25/75	> 350 °C
30/70	> 350 °C
31/69	> 350 °C
30/69/1	> 350 °C
35/63/2	> 350 °C
70/15/15	> 350 °C

Table 7 shows the results of the drop point test for the formulated greases. None of the formulated greases had a drop point, even at temperatures exceeding 350°C, which indicates that no drop point exists. This observation is due to the clay, which is a non-melting type of substance. This result is in agreement with the observations of Verdura et al. [9] and Boehringer [17], which showed that grease made of clay is a non-melting type grease that tends to decompose before reaching a drop point. The drop point indicates the optimal temperature at which grease is suitable for an operation. Above its drop point temperature, the lubricating grease loses its firmness and rigidity and may leak into other parts of the machinery. Thus, it is crucial that the lubricating grease not soften or flow within normal operating temperatures. The drop point property is useful for determining the suitability of the grease for specific applications. The SBE possesses thermal stability because it did not melt easily. However, the fumed silica used in this formulation had a melting point that exceeded 1600°C (per Sigma-Aldrich); therefore, the lack of drop point for this formulated grease proves that this grease is thermally stable over a wide range of temperatures. Stachowiak and Batchelor [7] observed that a grease’s service lifetime is often determined by the loss of its consistency due to the semi-solid structure of grease turning into a hard deposit or a liquid state during operation. The grease’s inherent properties are important for its real-life application because the grease must maintain its

structure as a non-Newtonian lubricant despite high service temperatures. The failure of the grease to remain semi-solid causes it to liquefy and increases lubrication failure.

3.2 Tribology properties

Figure 4 shows the friction coefficient versus time for different grease formulations. The friction coefficient for grease consisting only of SBE and WCO fluctuated slightly before stabilizing. The greases with fumed silica had higher friction coefficients and fluctuations. The fluctuation trend of the friction coefficient strongly correlates with contact while friction forces subdued the variation. This phenomenon is explained by the friction behavior of the formulated grease, which is defined as the instantaneous friction force/instantaneous contact load [7]. The average values of the friction coefficient for both samples SBEWCO were 0.095 and 0.11 for greases formulated from WCO, SBE and FS.

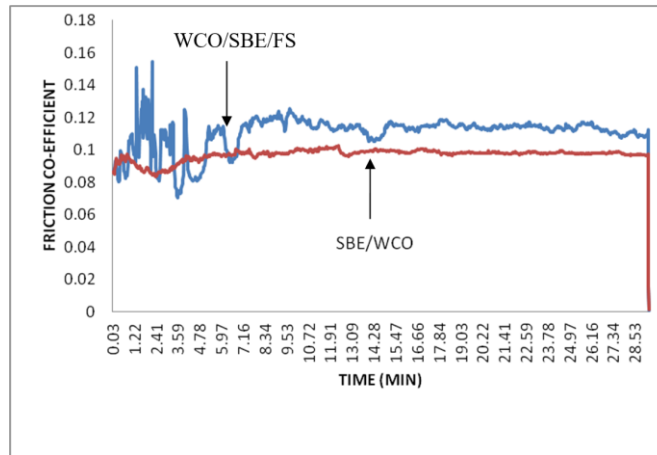


Figure 4 Friction coefficient vs. time for greases made of SBE and WCO

The study of friction coefficients is decisive in lubrication as part of the tribology study. The friction coefficient (kinetic) is defined by ASTM 996 as “the ratio of force required to move one body over another, to the total force applied normal to those surface, once the motion is in progress”. Therefore, a higher friction coefficient requires more energy to move one body over another and vice versa. The trend of grease formulated from SBE and WCO shows a lower friction coefficient and shows only a small change of friction coefficient during static conditions (static friction coefficient) compared with kinematic conditions (kinematic friction coefficient) in comparison with formulated grease with fumed silica. Grease mixed with fume silica is more viscous and thus requires more energy to move from the static condition to kinematic condition. A low friction coefficient is desirable because a high value of the friction coefficient in an engineering application can lead to an intolerably high friction force and large frictional energy losses [20]. The coefficient of friction is regularly used to determine the transition between boundary lubrication and elastohydrodynamic (EHD) lubrication [21].

3.3 Thermal stability

Figure 5 shows the thermal stability of the different grease formulations as well as the differences between greases formulated with and without fumed silica. The grease without fumed silica remained stable without any considerable degradation until the temperature reach 200°C. The addition of fumed silica improved the temperature resistance of the grease, demonstrating that the fumed silica can help the grease reach an operation temperature of 150°C or higher without significant degradation.

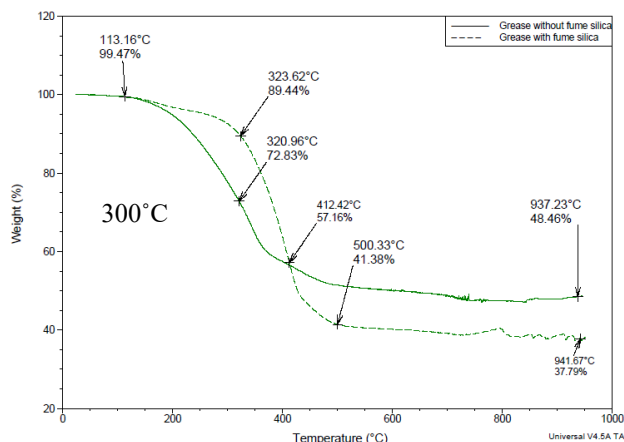


Figure 5 The thermal stability of the formulated greases

3.4 Corrosivity

Corrosivity is a crucial property of lubricating greases. Oxidation and degradation of the base oil can form acidic substances that will lead to corrosion and cause failure of the components. The evaluation the formulated greases suggested that the acidity of the oil and the SBE have the propensity to cause corrosion. Table 8 shows the result of corrosivity of the base oil made of WCO and new unused palm cooking oil (Vesawit, Malaysia). Table 9 shows the corrosivity of the formulated grease with and without fumed silica. The corrosivity for both base oils (WCO and new cooking oil) was similar. Both of the oils were slightly corrosive according to the result (1b as slight tarnish). Greases formulated without fumed silica had similar corrosivity as those with fumed silica, indicating that fumed silica is not responsible for the grease's corrosivity.

350°C

Table 6 Base oil corrosiveness test (100°C, 3 h)

Sample	Result
Waste cooking oil	1b – Slight tarnish (dark orange)
New cooking oil	1b – Slight tarnish (dark orange)

1b = Slight tarnish with dark orange

Table 7 Grease corrosiveness study (100°C, 24 h)

Sample	Result
SBE +WCO+FS	1b – Slight tarnish (dark orange)
SBE + WCO	1b – Slight tarnish (dark orange)

Acidity can be corrosive toward metal. Vegetable oil consists of fatty acids, which are also acidic. The acidity of waste cooking oil is higher because of the presence of free fatty acids after cooking, which may degrade the oil. This study found that the waste cooking oils were only slightly corrosive toward metal (copper) and were comparably corrosive to the new unused cooking oil.

4. Conclusion

Making lubricants from WCO and SBE is a new concept. WCO is derived from abundant sources of cooking oil, such as frying and other food applications, which find their way into drains. WCO and SBE are a unique source for making lubricants that can repurpose waste into grease formulations to create an alternative and environmentally friendly product. This lubricant grease could be applied to the automotive and food industries as well as to other general industries because of their special characteristics, lubrication properties and high temperature resistance. The performance of these grease formulations made from the WCO and SBE shows promise for future applications. Further optimization with additional additives is required to tailor the performance of these formulated greases for commercial use to compete with existing lubricating greases on the market.

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