Conversion of Waste Transformer Oil into Grease

N. Suhaila A. Japar, Mohd Aizudin Abd Aziz, and N. W. Abdu Rahman

Abstract This work is aimed to study the viability of waste transformer oil (WTO) as grease's base oil. The shift of lubricant formulation towards high-performance materials and green formulation has led to the development of various lubricant formulations, including grease. Waste reduction is one of the formulation research trends where waste-based material, i.e. waste oil generated from automotive industries, is used as one of the grease constituents, and the grease's characteristics and performances are evaluated and compared to the conventional grease. Variability of waste oil composition, a mixture of conventional and synthetic oil, has led to inconsistent grease properties and performances. This issue, however, is uncommon in power industry-generated waste oil and becomes a potential alternative to replace the waste oil from automotive industries, thus creating this opportunity. It was found that, after conducting WTO analysis, incorporating WTO in grease formulation and evaluating the WTO-based grease characteristic, the WTO is viable to be used as grease base oil.

Keywords Waste transformer oil · Grease formulation · Sodium grease · Fumed silica grease · Oil and grease analysis

1 Introduction

Grease is a semisolid lubricant where a thickening agent is dispersed in a liquid lubricant. It is made up of three elements of base oil, thickener and additives. Mineral oil is the most commonly used base oil, but a wide range of other base oils types, such as synthetic oil, silicone and vegetable oil, also available to be used. Grease thickener, either soap or non-soap thickener, is used to trap the oil in its structure until it is ready to be utilized in lubrication.

Grease has been used for plenty of applications, involving machinery and moving parts, acts as a seal and provides protection against corrosion and at the same

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time being able to reduce noise and shock. Grease, similar to lubricating oil, serves primarily to keep moving part apart, reduces friction, transfers heat, carries away contaminants, transmits power, and wear and corrosion protection (Donley 2012). Historically, lubricant was used since Ancient Egypt, where back then—water is used to help in reducing friction between the sled and the sand to ease the movement of a statue. The grease-like substance was first identified back in 1400 BC, where animal fats were used to lubricate the axle of chariots.

1.1 Current Trends in Grease

The industrial development has come a long way, and the lubricant has gained its importance in the industry. The development and advancement of grease were based on the grease thickener since the mid-1880s. Up to this date, there is no significant development in terms of grease manufacturing, but the trends are changing based on rapid industrialization and urbanization. According to Gresham (2018), future trends in lubricant formulation including grease will keep emphasizing on protecting the environment, reducing waste, utilization of highly refined and synthetic base oil, and creating additives that deliver various performances (Gresham 2018).

Numerous researches have been conducted in developing new and improved grease formulation parallel with the current formulation direction. The grease formulations were primarily conducted to evaluate the potential and performance of selected material as one of the grease components either base oil, thickener or additive to see whether it is comparable or better than the conventional grease in delivering the proposed aim. Some studies that were conducted for the purpose of protecting the environment and waste reduction; however, investigating various environmentally friendly and waste-based materials to help in providing alternative material to produce eco-friendly grease and to reduce the abundant waste that threatens the environment, respectively.

1.2 Waste Oil as Base Oil

The generation of waste oil continues to increase annually in response to urbanization and industrialization. Although waste oil holds less than 10% breakdown of the total hazardous waste generated in Malaysia (Aja et al. 2016), some waste oil-related water pollution crises have been reported. However, legal waste oil disposal is rather costly, thus leading to illegal oil disposal and pollution. On the bright side, waste oil still has its economic value and it can be recovered, reclaimed, recycled, reused and converted into a new product such as fuel oils and lubricants, and this will promote environmental protection and reduce the number of wastes oil being disposed of.

Some researches had started in utilizing and employing waste oil in lubricant formulation, including grease to overcome the increasing waste oil generation and environmental concern. Although several studies had focused on this topic, the problem of this approach is the variability of the waste oil composition due to the mixture of various types of waste oil, both mineral and synthetic oil, which will definitely exhibit inconsistent lubricant properties. This is common for waste oil generated by the automotive and machinery industries since there are numerous moving parts which lubricated with different lubricants, either mineral- or synthetic-based. This problem, however, is not apparent to waste oil generated from the power industry, for example, waste transformer oil (WTO). In a power station, the transformer oil type or brand that is used usually the same for every transformer. Hence, the chances of waste oil mixture are minimal.

WTO is a discarded transformer oil due to its quality that is no longer up to the standard. Presently, researches are carried out by reclaiming WTO as an alternative fuel oil either diesel or gasoline, due to its properties that are closed to the fuel oils (Nabi et al. 2013; Mohta and Chaware 2015). The initiative in using WTO to produce lubricant had not gained its emphasis yet. Up to this date, there is no research conducted in producing lubricant from WTO and there is also no prove saying that WTO is unsuitable as lubricant's base oil. Several studies, however, were found to succeed in producing grease by utilizing virgin transformer oil as a base oil to provide unique properties to the grease (Hassan et al. 2013). This, in turn, indicates the viability of WTO as grease base oil and hence creating the opportunity in developing new lubricant formulation including grease, with WTO as base oil. The use of waste oil generated by the power industry will help in solving the variability of oil composition faced with waste oil from the automotive and machinery industries.

2 Experimental Work

Grease formulation involving WTO required four major phases (Fig. 1). The first phase involves only WTO, where WTO is collected, treated and analysed. Grease formulation took place in the second phase, where two types of NLGI grade 2 greases—sodium and fumed silica (FS) grease—are formulated using 67.5–82.5 and 92–94% of WTO, respectively. The greases then produced and analysed for their physical and chemical characteristics.

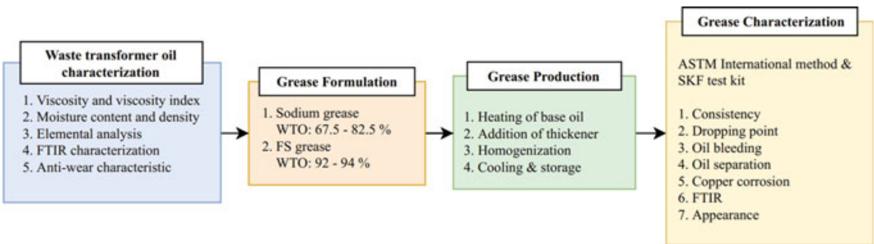

Fig. 1 Major phases of the overall research workflow

3 WTO Analysis

The WTO is first treated through settling, filtration and evaporation processes to remove any impurities presented in the WTO. The WTO condition is evaluated by identifying the WTO fluid properties—i.e. viscosity, density, and moisture content, contaminations and wear debris concentration—through FTIR and elemental analysis.

Viscosity. It is conducted using a glass capillary viscometer (Cannon-Fenske) with Cannon-Fenske tube size 150 and measured according to ASTM D445. The oil viscosity was measured at two temperatures of 40 and 100 °C, and these values are used to calculate the viscosity index.

Moisture content. It is evaluated using 807 KF tritino, a Karl Fischer titrator, to determine the water content presented in the oil.

Density. Density is analysed using a gas pycnometer. The supply of high purity helium gas is used as a medium in the process of density measurement. The result of the test is measured in g/ml.

FTIR characterization. It is conducted using the FTIR instrument, Thermo Scientific Nicolet iS5 FTIR Spectrometer and used to identify the compound existed and provide information on any contamination, additive and any chemical changes presented in the WTO.

ICP-MS. It is used to detect the concentration of wear metals, contaminants, or additive elements within the WTO. The results of the test are reported in terms of the element concentration (ppm or ppb) and compared to the standard provided by the Department of Environment (DOE), Malaysia.

4 Grease Production

The production of grease involves only four steps, which started by raising the base oil temperature, addition of thickener, homogenization of mixture, and lastly, cooling of product and storage. The composition for all grease's formulations are shown in Table 1.

Table 1 Composition of sodium and FS grease

Samples		Grease composition (wt%)	
		WTO	Thickener
Sodium grease	SG_1	82.5	17.5
	SG_2	75	25
	SG_3	67.5	32.5
FS grease	FG_1	94	6
	FG_2	93	7
	FG_3	92	8

Sodium grease. The preparation of sodium grease is initiated by heating the WTO at 120 °C for at least 1 h with continuous stirring to remove traces of moisture. The WTO temperature is then increased to 180 °C. Sodium thickener is added gradually into the WTO, and the mixture is homogenized for at least 3 h until the smooth paste was obtained. The homogenization of the grease sample was conducted at an increased speed for at least 30 min without the presence of heat to allow uniform dispersion of mixture. After homogenization, grease is cooled to room temperature and stored in an enclosed container.

FS grease. The procedure in preparing FS grease was referred to Abdulbari et al. (2008). The preparation steps of FS grease are similar to sodium grease, but the operating temperature is different. After raising WTO's temperature for moisture removal, the temperature is then lowered to 80–90 °C. Fumed silica is added portion-wise into the oil and continuously homogenized at a constant temperature of within 80–90 °C (Abdulbari et al. 2008). The mixture then homogenized for at least 3 h until a gel-like paste is formed. The mixture is also homogenized at an increased speed for at least 30 min without the presence of heat to uniformly disperse the mixture. After homogenization, grease was cooled and stored.

5 Grease Characterization

Greases are characterized both physically and chemically to identify the properties of the grease. Grease consistency, dropping point, copper corrosion and anti-wear properties are the most commonly evaluated grease properties.

Consistency. It is evaluated using the SKF grease testing kit and penetrometer (ASTM D217). When using the SKF grease kit, an amount of grease sample was placed in between two glass plates and placed on top of an NLGI grade scale. Penetrometer (HK-2020) and mechanical grease worker (HK-269G) were used to determine the grease penetration number. Worked grease then was placed below the tip of the penetrometer cone, and the cone is allowed to drop into the grease. The depth of penetration is measured in tenths of a millimetre (mm/10).

Dropping point. It is conducted as described in standard ASTM D2265. Dropping point instrument, HK-2019 High-Temperature Dropping Point Apparatus, is used in this test with maximum operating temperature up to 400 °C. The dropping point then calculated using Eq. 1, where DP stands for dropping point, ODP is a thermometer reading when the first drop reaches the bottom of the test tube, and BT is the block oven temperature when the drop falls.

$$\mathrm{DP}\ (^\circ\mathrm{C}) = \mathrm{ODP} + [(\mathrm{BT} - \mathrm{ODP})/3] \tag{1}$$

Oil bleeding. It is conducted using the SKF's grease test kit with a small volume of grease samples. In this analysis, the grease sample was placed on a blotter paper and heated on a hotplate at 60 °C for 2 hours. The grease's bleed area percentage differences were measured and calculated by using Eqs. 2 and 3 between fresh and aged grease, where S_{Fresh} and $S_{\text{Used,}}$ respectively, stand for the bled area from the fresh and aged sample, D_{AVi} is the average diameter of the bled area and % Diff represents the bled area difference between fresh and used samples. Aged greases in this study refer to the grease that has been aged for 10 days at 70 °C.

$$S_i = 0.785 \times (D_{\text{AVi}}^2 - 100) \tag{2}$$

$$\%\ \text{Diff} = 100 \times \frac{(S_{\text{Used}} - S_{\text{Fresh}})}{S_{\text{Fresh}}} \tag{3}$$

Oil separation. 150 g of grease sample was left untouched in an enclosed container for a month, and the oil separated on top of the grease surface was observed, collected and measured in weight percentage. It is desirable for the grease to released oil for less than 4%, according to Lugt (2013a) during storage.

Copper corrosion. Corrosion test was carried qualitatively through an observational technique to study the WTO and grease corrosiveness towards copper strips. This method was conducted in accordance with ASTM D130 for WTO and ASTM D4048 for greases (ASTM D130-18 2018; ASTM D4048-16e1 2016; Nye Lubricants 2016).

FTIR characterization. It is conducted using the FTIR instrument, Thermo Scientific Nicolet iS5 FTIR Spectrometer and used to identify the compound existed and provide information on any contamination, additive and any chemical changes presented in the prepared grease.

Wear preventive test. It is conducted for both treated WTO and selected greases by using four-ball tester in accordance with ASTM D4172b for the WTO and ASTM D2266 for grease sample (ASTM D4172-18 2018; ASTM D2266-01 2015) to study the friction and wear properties of base oil and grease as a result of a motion between the steel balls and to observe the formation of lubricant film on the worn surface using micrograph and energy dispersive X-ray (EDX) analysis.

6 Result and Discussion

6.1 WTO Analysis

WTO analysis was carried out to investigate the WTO condition. Based on the WTO analysis on its physical (Table 2), contamination and wear debris (Table 3), all were found favourable and comparable to most base oil used in grease formulations (Donley 2012).

WTO treatment is crucial since WTO was contaminated with water and unknown suspended materials that may be possible in causing undesirable issues. The treatment process was proved adequate to reduce the WTO water content from 0.14 to 0.05%. This reduction was also observed through the FTIR spectrum of the WTO

Table 2 WTO's physicochemical properties

Base oil	Test method	Transformer oil	Treated WTO	Untreated WTO
Appearances	Visual	Clear and bright	Bright yellow	Amber
Kinematic viscosity				
@ 40 °C (cSt)	ASTM D445	9.55	10.06	9.95
@ 100 °C (cSt)		2.55	2.57	2.59
Viscosity index (VI)	ASTM D2270	92	97	85
Density (g/mL)	Gas pycnometer	0.895	0.875	0.926
Moisture content (%)	Karl Fischer	0.002	0.05	0.14
Copper corrosion	ASTM D130	1a	1a	–

Table 3 Elemental analysis of WTO

Element	Formula	DOE specs (Hazardous Substance Division 2010)	Treated WTO
Beryllium	Be		<0.5 ppb
Sodium	Na		120.7 ppm
Magnesium	Mg		<0.1 ppm
Aluminium	Al		5.1 ppm
Potassium	K		<0.1 ppm
Calcium	Ca		<0.1 ppm
Vanadium	V		<0.5 ppb
Chromium	Cr	Max. 10 ppm	<0.5 ppb
Manganese	Mn		<0.5 ppb
Iron	Fe		<0.5 ppb
Cobalt	Co		<0.5 ppb
Nickle	Ni		<0.5 ppb
Copper	Cu		<0.5 ppb
Zinc	Zn		281.2 ppb
Arsenic	As	Max. 5 ppm	<0.5 ppb
Selenium	Se		<0.5 ppb
Molybdenum	Mo		<0.5 ppb
Silver	Ag		<0.5 ppb
Cadmium	Cd	Max. 2 ppm	<0.5 ppb
Antimony	Sb		<0.5 ppb
Barium	Ba		<0.5 ppb
Lead	Pb	Max. 100 ppm	384.9 ppb
Sulphur	S		22.8 ppm

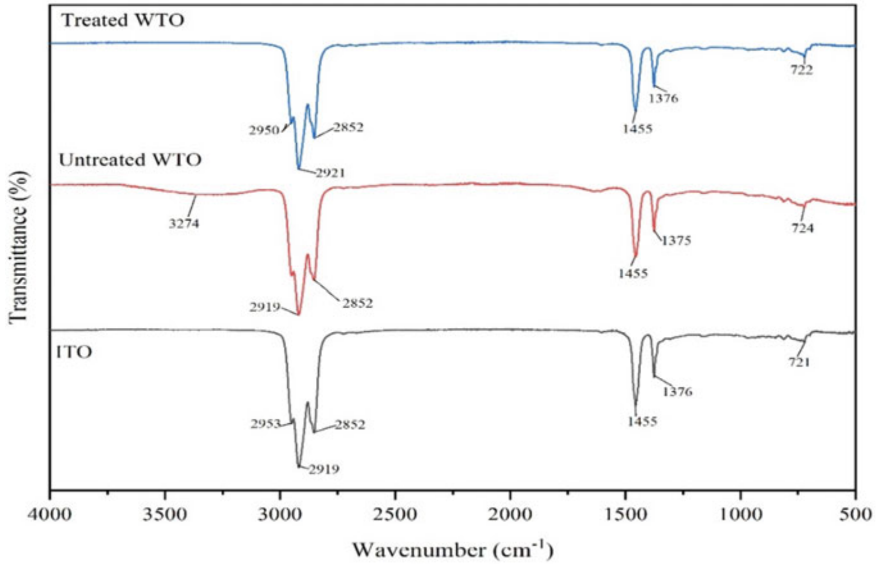

Fig. 2 FTIR spectrum of virgin transformer oil (ITO), treated WTO and untreated WTO

(Fig. 2) before and after the treatment process, which transmittance band indicating the presence of O–H bond that represented water molecule in untreated WTO was undetected after WTO treatment. The contaminants such as wear debris also found in favourable concentration through elemental analysis (Table 3), complying with the standard set by the DOE, Malaysia, for recovered waste oil. The WTO's viscosity, however, unmanaged to be restored to its original value, though its resistance to viscosity change was slightly improved (Table 1).

The tribological study on WTO shows that the WTO's friction coefficient and wear scar diameter were 0.1158 and 743 µm, respectively. This finding is comparable to some of the previous studies (Zuan et al. 2017; Hassan et al. 2016), but the addition of additive is recommended as the WTO film was too thin—due to WTO's low viscosity, to separate the contacting surfaces, thus causing wear to the contacting surfaces.

6.2 Grease Characteristic

Table 4 shows all evaluated grease's properties for both sodium and FS grease.

Consistency. Grease consistency depends on the type and amount of thickener, as well as the base oil's viscosity. According to Rizvi (2008), NLGI grades 2–3 greases exhibit the most grease-like behaviours and function (Rizvi 2008). This study aimed to produce NLGI grade 2 grease, both sodium and FS greases. Such greases were obtained when the WTO percentage is 67.5% in sodium grease and 92% in FS grease.

Table 4 Sodium and FS grease characteristics

Grease	Composition, %		NLGI	Penetration number (mm/10)	Drop point (°C)	Oil bleed (%)	Oil separation (%)	Copper corrosion
	WTO	Thickener						
SG_1	82.5	17.5	0	378	158	−9.54	0.27	1b
SG_2	75	25	1	311	175	−11.67	0.07	1b
SG_3	67.5	32.5	2	275	193	−18.04	0	1b
FG_1	94	6	00	408	264	N/A	0.07	1b
FG_2	93	7	1-0	344	312.3	−28.7	0	1b
FG_3	92	8	2	287	>350	−15.4	0	1a

It was noticed that as the amount of WTO decreased, the grease becomes stiff due to the high amount of thickener in the formulation.

Dropping point. Typically, sodium grease has a dropping point of around 175 °C. WTO-based sodium grease has a dropping point as high as 200 °C. Previous studies in developing grease by utilizing waste oils also exhibited similar dropping points ranging from 165 to 190 °C (Iheme et al. 2014; Ebisike et al. 2016). This, in turn, concluded that at this temperature range, the sodium grease had reached the temperature limit where the grease structure started to break and release of WTO from the thickener matrix.

The highest dropping point observed for FS grease is above 300 °C at 93% WTO content, but when WTO percentage is dropped to below 93%, FS grease dropping point was not existing. This was due to the fumed silica's non-melt characteristic and high melting point (>1600 °C). Abdulbari et al. (2008) and Razali et al. (2017) also presented similar findings with FS-containing grease. On the other hand, FS greases were found decomposed after the test is carried out. Non-melt thickener often burns off over high temperatures even before it reached the grease dropping point because, at a point, the oil evaporated or burns off during the test and leaving the thickener residue to hardened and decayed (Corporation 2002; Abdulbari and Zuhan 2018).

Oil bleeding. Bots (2014) stated that the oil bleeding differences within −15 to +15% between used and fresh are desirable as they indicated that the grease still can be utilized without changing the re-lubrication intervals (Bots 2014). The positive value is indicating that aged grease bleeds more oil than fresh grease and vice versa. Sodium grease exhibited desirable oil bleeding at both ageing temperatures at the WTO percentage of 75 < WTO% < 82.5. As for FS grease, oil bleeding was observed the best at WTO percentage less than 93%.

Overall, it was observed that the decrease in WTO percentage and an increase in temperature led to a decrement of oil bleeding. This is because, when WTO content is reduced, the thickener content is increased, and the microstructure becomes denser and grease permeability is reduced along with the grease's oil bleed (Gonçalves et al. 2015). Not only that, but thermally ageing of grease also affecting the grease oil bleed where thermally aged grease will display much lower oil bleed measurement.

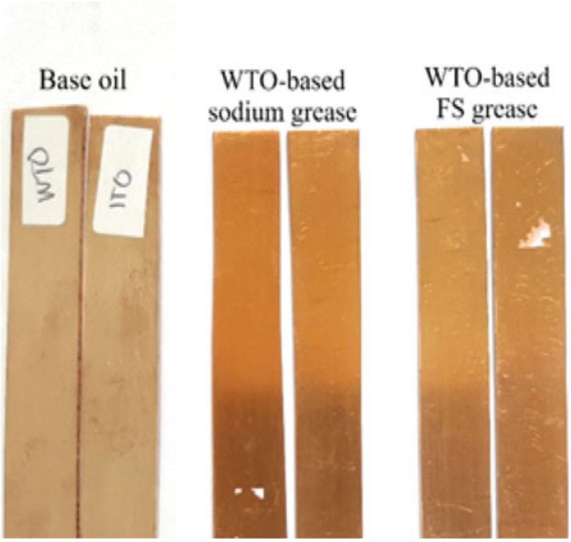

Fig. 3 Graphical result of corrosion test on oil and grease

Oil separation. Excessive oil content and insufficient thickener in grease formulation result in oil separation from grease. It was shown that, from Table 4, only soft consistency greases (≤ NLGI 1) show oil separation on the grease surface. The amount of oil separated complied with an acceptable value of less than 4% (Lugt 2013b). As the WTO percentage is reduced and thickener content increases, the thickener was observed adequate to adsorb/hold the WTO in its matrix, which led to no oil separation occurred.

Copper corrosion. Figure 3 demonstrates the graphical results of the copper corrosion test conducted on base oil and selected greases. The stain on the copper strip indicates that the likelihood of the grease to cause corrosion on copper. In this study, sodium and FS grease corrosiveness are classified in class 1 (slightly tarnish). According to Kholijah et al. (2012), grease corrosion classified in Class 1 considered passed the ASTM corrosion test (Kholijah et al. 2012). From this, the results on grease corrosiveness test demonstrated that all grease constituents, base oil and thickener, were unlikely to cause corrosion to copper-containing metal.

6.2.1 Wear Preventive Characteristic

Oil is responsible for providing lubrication to the metal surface and grease is a sponge-like vessel to hold the oil in it until it is ready to be released for lubrication. The average COF of sodium and FS greases was found to be 0.119 and 0.133, respectively. The COF analysis shows that the thickener does not help in improving the grease resistance to friction since the COF of grease is closed to WTO's COF.

The COF of the present study is high when compared to some commercial greases and the previous grease tribological studies. This is again due to the absence of

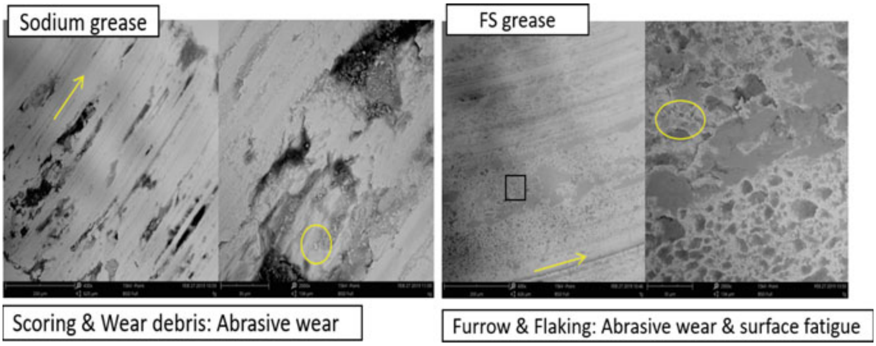

Fig. 4 Wear micrograph of steel balls lubricated with sodium and FS grease

additive in grease. This test was conducted to study the anti-wear properties of the base grease itself without the additive—resulting in high COF due to no sacrificial layer to be worn during operation.

The wear micrograph (Fig. 4) does not show severe wear on the steel ball for both greases. Wear debris was visible which indicated removal of material through abrasion or adhesion mechanism. The wear scar diameter (WSD) measured under the microscope for both sodium and FS grease was 722 and 866 μm, respectively. The EDX analysis of the worn surface shows the presence of iron (Fe), carbon (C) and oxygen (O), contributed by the steel ball, WTO and oxide layer. Both steel ball and WTO contain carbon elements in their composition; thus, there is a chance that the WTO layer was present. Element silicon (Si) was detected indicating the adherence of FS grease on the worn surface (Rawat et al. 2018).

7 Summary

Depending on the application and the operating and surrounding environmental conditions, grease with appropriate properties to serve the application can be chosen. Based on the WTO analysis, the treated WTO was concluded can be used as a base oil in grease formulation. The greases prepared using WTO were proved to have desirable properties of good structure and consistency, high dropping point with stable oil bleeding and no separated oil during the storage period, depending on the percentage of WTO. The oil's viscosity and VI, however, do not define whether or not the oil can be incorporated in the grease formulation since it is depended on the application of the grease. Oil of high viscosity often used in low-speed and high-load application and vice versa. Studies also proved that grease could be produced using low viscosity oil and hence concluded that WTO is viable to use as grease's base oil.

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