



Full Length Article

Formulation of fumed silica grease from waste transformer oil as base oil



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ABSTRACT

This study aimed to formulate fumed silica (FS) grease using waste transformer oil (WTO) and to investigate the grease properties. Various unexplored application of the reused WTO due to its reusable characteristic. WTO and fresh transformer oil (ITO) were used as grease base fluid and the properties of oil and grease were evaluated using ASTM International standards and SKF's Grease Test Kit. The oil and grease chemical compound were determined using Fourier Transform-Infrared Spectroscopy at wavenumber of 500–4000 cm^{-1} . It was found that WTO have high viscosity index of 96 and low moisture content of 0.05% after pre-treatment than ITO. FS greases with and without MoS_2 were classified as NLGI 2 greases when FS content was 8%. Grease formulated with MoS_2 shows better performances in term of corrosiveness, oil bleed within -15% to $+15\%$, oil separation less than 4%, and dropping point of $>300\text{ }^\circ\text{C}$. FTIR results shows no significant different between ITO and WTO, and between all formulated greases. Based on the findings, it was concluded that WTO can be utilized as an alternative base oil in grease formulation due to the good properties exhibited by the formulated WTO-based FS grease blended with MoS_2 .

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1. Introduction

Grease is known as a semisolid product that consists of base fluid, thickener, and additive. Nowadays, there are variety types of grease existed. The rapid growth of the industries has led to higher demands in lubrication as well as increasing amount of waste oil generated. Waste oil is categorized as schedule waste and is known for its ability to threaten public health and environment.

Green product had gained its attention in the recent years. In Malaysia, waste oils is generated approximately 150 million liters annually [1] and an estimated 36 MTA of waste transformer oil (WTO). The idea of reusing waste oil to produce new product has been implemented in Malaysia [2] but unfortunately, WTO has not making its way to be reused in any application. Even though there are only small amount of WTO disposed every year compared to waste engine oils but, it is important to find the potential use of WTO in order to avoid the amount to increase.

Grease consist of almost 90% of base oil which is mainly derived from petroleum oil. However, the amount of petroleum oil is continuously decreasing due to the limited petroleum reserves. Researchers been studying other alternative raw materials which have the potential to use as a replacement of the petroleum oil

such as vegetable oil, waste cooking oil, waste engine oil, ionic liquid and more [3–6].

Transformer oil is an insulating oil which act as heat transfer medium in the transformer. In general, transformer oil is produced from wax-free naphthenic oils. After certain period of being used in the transformer, the oil is removed and form a waste known as WTO. Several studies on the utilization of transformer oil in grease formulation have been conducted by blending the oil into lubricating oil and by using transformer oil solely as base oil to study the potential of the transformer oil to provide dielectric and insulating properties for the greases, respectively [7–9]. However, there are no study conducted regarding the use of WTO oil in the formulation of grease.

Earlier findings of testing the possibility of grease structure to form when WTO is utilized in the formulation, it was found that the grease structure is comparable to the preferable grease texture at specific formulation. Based on that, the current study aimed to investigate the potential of WTO as a grease's base oil substitute and to evaluate the formulated greases structure and properties.

2. Experimental work

2.1. Materials

Waste transformer oil (WTO) collected from electrical power plant located in Selangor, a fresh uninhibited industrial grade

transformer oil (ITO), fumed silica (FS) powder, molybdenum disulfide (MoS_2) with 98.5% purity.

2.2. Grease formulation

2.2.1. Pre-treatment of WTO

In this experiment, WTO was pre-treated through several treatments to remove unwanted contaminants such as water and suspended solid. WTO was first settled in an untouched container for a week at room temperature to separate the WTO and water layers by gravity forces. Water layer separated at the bottom of the container was then removed, leaving only the WTO inside the container. WTO was then transferred into new cleaned enclosed container.

Secondly, WTO was vacuum filtered at room temperature using vacuum pump and glass microfiber filter with pore size of $1.2 \mu\text{m}$ to remove any visible solid particles suspended in the WTO. The WTO was heated on a hotplate at 120°C for at least 2 h with continuous stirring to remove moisture and any volatile compound present in the WTO through evaporation process. After evaporation process, WTO was cooled down to room temperature before being stored for further use.

2.2.2. Preparation of grease containing ITO (IG_i)

Grease containing ITO was formulated to compare its properties with grease formulated using WTO. There were two formulations involved, for which the greases were formulated with and without the addition of additive which was MoS_2 .

ITO was heated to $80\text{--}90^\circ\text{C}$ and FS was added portion wise for 30 min with continuous homogenization and MoS_2 was added afterwards. Homogenization was continued for a total time of 1 h to disperse the thickener and additive [10]. After homogenization, grease was stored in enclosed container for 2 days to allow cooling of grease. The formulation of grease containing industrial transformer oil (IG_i) was shown in Table 1.

2.2.3. Preparation of grease using WTO (WG_i)

This experimental grease formulation consisted primarily of WTO and FS. There were two formulations involved, for which the greases were formulated with and without the addition of additive which was MoS_2 .

WTO was heated to $80\text{--}90^\circ\text{C}$ and FS was added portion wise for 30 min with continuous homogenization and MoS_2 was added afterwards. Homogenization was continued for a total time of 1 h to disperse the thickener and additive [10]. After homogenization, grease was stored in enclosed container for 2 days to allow cooling of grease. The formulation of grease containing waste transformer oil (WG_i) was shown in Table 1.

2.3. Grease analysis

All formulated greases were analyzed through analytical testing to determine their properties.

2.3.1. Consistency test

The test on each grease was performed using the SKF Grease Test Kit TGKT 1. A fixed grease volume was spread between the two glass plates and pressed for 15 seconds by means of the weight. The consistency of the grease strain was observed and evaluated using calibrated measuring scale NLGI grade. This test method was in accordance of ISO 2137 which specifies methods for determining the consistency of lubricating greases when only small samples were available. The results of NLGI for each grease indicates their consistency level [11].

2.3.2. Dropping point test

The dropping point test was conducted as described in standard ASTM D2265 (Standard Test Method for Dropping Point of Lubricating Grease over a Wide Temperature Range) [12]. In this test, grease sample was placed in grease test cup supported in a test tube and placed in an aluminum block oven at a preset constant temperature. A sample thermometer is placed in the tube and so positioned that it measures the temperature. As the temperature increases, at some point a drop of material will fall from the cup to the bottom of the test tube. The reading on the sample thermometer is recorded to the nearest degree as the observed dropping point. At the same time, the temperature of the aluminum block oven is also recorded to the nearest degree. The drop point was then calculated using Eq. (1).

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

where, DP stands for dropping point, ODP is a thermometer reading when first drop reaches the bottom of the test tube, and BT is the block oven temperature when the drop falls.

2.3.3. Oil bleeding

This method was in accordance to IP 121 where large volume of sample was required [13,14]. Oil bleeding test was conducted using the SKF Grease Test Kit TGKT 1, an alternative method where only small volume of samples were required [15]. The sample was put on blotter paper and heated for 2 h at 60°C according to the SKF's manual [13]. The oil stain created on the paper were measured based on the bleed area and the percentage difference between bleed area of fresh and used samples by using equation Eqs. (2) and (3). Used greases refer to grease that have been aged at two controlled condition of 10 days at room condition and at 70°C [16].

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

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

where, S_i stands for the bled are from fresh and used sample, D_{AV} is the average diameter of the bled area, and %Diff represents the bled area difference between fresh and used sample.

2.3.4. Oil separation

Oil separation test was carried to identify the tendency of oil to be separated from grease during storage in accordance to ASTM

Table 1
Formulation of the greases IG_i and WG_i .

Components (%wt)	Sample notation									
	IG_1	IG_2	WG_1	WG_2	WG_3	WG_4	WG_5	WG_6	WG_7	WG_8
Industrial transformer oil	92	91.14	–	–	–	–	–	–	–	–
Waste transformer oil	–	–	95	93	92	91	93.1	91.14	90.16	89.18
Fumed silica	8	6.86	5	7	8	9	4.9	6.86	7.84	8.82
Molybdenum disulfide	–	2	–	–	–	–	2	2	2	2

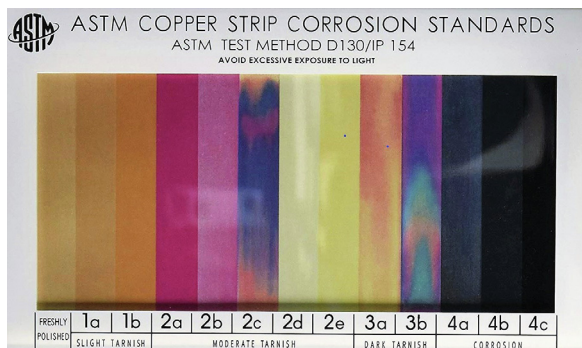


Fig. 1. ASTM copper strip corrosion standards.

D-1742 (Standard Test Method for Oil Separation from Lubricating Grease during Storage). The grease sample was stored in an enclosed container at room temperature for 1 month. The quantity of oil separated was collected and measured in weight percentage.

2.3.5. Corrosion test

Corrosion test was carried out to determine the level of corrosiveness of the base oil and the formulated grease towards copper strips. This method was carried according to ASTM D-130 (Standard Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test) and ASTM D-4048 (Test Method for Detection of Copper Corrosion from Lubricating Grease by the Copper Strip Tarnish Test), respectively [17,18]. In this test, a prepared copper strip is totally immersed in a sample of grease or oil and heated in an oven or liquid bath at a specified temperature for a definite period of time. The condition for the test was set at 100 °C for 3 h in water bath for base oil and 24 h in an oven for grease. At the end of the test period, the strips were removed from sample, washed and compared to the ASTM Copper Strip Corrosion Standards (Fig. 1) [18].

2.3.6. FTIR analysis

Fourier transform infrared (FTIR) spectroscopy identifies the type of base oil and thickener in grease sample. By comparing the fresh grease reference to the used grease, FTIR spectrum can provide information regarding contamination and any changes in a grease sample. In this experiment, greases and oils were characterized at wave number from 500 to 4000 cm^{-1} .

3. Result and discussion

3.1. Physicochemical properties of oil

Table 2 shows the physicochemical properties of ITO and WTO. ITO's properties was as in data sheet provided by the industry. Base oils are present in majority of >80% in greases formulation where it functions to take care of lubrication by seeping out from the thickener matrix after being introduced to load [19]. High viscosity oil-based greases flow slowly compared to low viscosity oil-based greases. Wherever application speeds are low, high viscosity oil-based greases are recommended and vice versa. The choice of oil viscosity depends on the intended application. It is clear that in this study the transformer oils possessed lower viscosity of 10 cSt at 40 °C.

3.2. FTIR characterization of oil

FTIR had been used to determine the functional group of ITO and WTO. The measurement was within 500–4000 cm^{-1} . Figs. 2

Table 2
Physicochemical properties of ITO and WTO.

Properties	Test	Industrial transformer oil	Waste transformer oil
Appearances	Visual	Clear & bright	Bright Yellow
Kinematic viscosity at 40 °C, cSt	ASTM D445	9.84	10.20
at 100 °C, cSt		2.55	2.61
Viscosity index	ASTM D2270	81	81
Density, g/mL	Gas pycnometer	0.895	0.875
Moisture content, %	Karl Fischer method	0.002	0.05

and 3 shows that the oils have high intensity bands in the region of 2852–2950 cm^{-1} , as a result of the CH_2 and CH_3 asymmetric stretching. A strong band at 1455 cm^{-1} due to the C–H asymmetric bending vibrations which indicates the presence of alkanes [20]. Moreover, a strong band obtained at 1376 cm^{-1} also indicate the functional group of alkane C–H bond stretch of CH_3 bond [21]. A weak band at 721 cm^{-1} represented the C–H out of plane bend indicating the presence of alkanes [22].

By comparing the FTIR spectrums of both ITO and WTO, there was no significant differences between fresh and waste transformer oil. In addition, there was no clear peaks indicating the presence of contaminants or by-products especially in WTO, such as dissolved gases, moisture etc. for which it usually resulted from the thermal stress in the transformer [21]. Thus, there were no further WTO treatments conducted to remove any dissolved contaminants other than the pre-treatment of oil.

3.3. Consistency of formulated grease

Table 4, shows the test results of formulated greases on its consistency, dropping point, oil bleeding and the oil separation.

The outcomes from the consistency test done upon each of the grease samples is in accordance to the NLGI consistency grade (Table 3). The grades defined the consistency of the greases and their appearance at 25 °C. Based on Table 4, IG₁, IG₂, WG₃, WG₄, WG₇, and WG₈ were recorded with NLGI grade 2 to NLGI grade 3, where grease with these consistencies were most commonly used in the industries [23]. Greases with NLGI grade 2 were suitable for rolling bearings moderately loaded with medium speed applications. Grease of this class of consistency are formulated to give a good balance of properties required for easy pumping

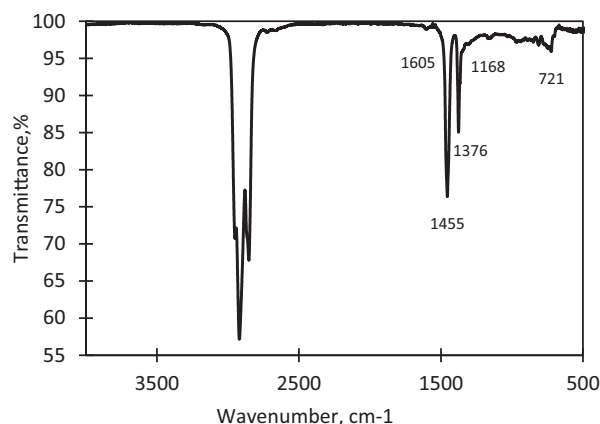


Fig. 2. FTIR of ITO.

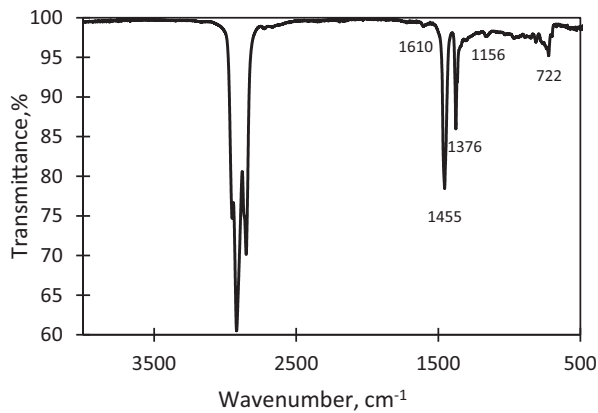


Fig. 3. FTIR of WTO.

through dispensing systems [24]. In addition, most of the multi-purpose grease also of this grade of NLGI 2-3 consistency.

Some of the formulated greases possessed NLGI grade consistency softer than NLGI 2 for which it might be due to the insufficient amount of thickener in the formulation to hold the oil in the thickener system. However, these soft greases consistency can be increase by decreasing the oil-to-thickener ratio [25]. Conversely, greases with softer consistency does not mean it cannot be used in any application. Greases of such consistencies can be used at low operating temperatures and high-speed application. Therefore, the grease consistency may be adjusted in the formulations according to the application's operating condition.

3.4. Dropping point of grease

Table 4 shows the results of dropping point test for the formulated greases using both ITO and WTO. Based on the results, the formulated greases dropping point temperature increases as the

Table 3
NLGI classification system based on consistency [23].

NLGI grade	Worked penetration, tenth of millimeter	Consistency
000	445–475	Very soft
00	400–430	
0	355–385	Soft
1	310–340	Soft
2	265–295	Creamy texture (buttery)
3	220–250	Semi-solid
4	175–205	Stiff
5	130–160	Stiff
6	85–115	Hard solid

Table 4
Formulated grease properties.

Grease sample	Consistency (NLGI grade)	Dropping point (°C)	Oil bleeding (%)		Oil separation (%)
			Room condition	At 70 °C	
IG ₁	2–3	>350	–7.75	–14.2	No separation
IG ₂	2–3	>350	–8.39	–11.4	No separation
WG ₁	000	203.3	Not tested (too soft)		0.14
WG ₂	1–0	312.3	–11.01	–28.7	No separation
WG ₃	2	>350	–7.87	–15.4	No separation
WG ₄	3–4	>350	–28.98	–28.40	No separation
WG ₅	000	270.7	Not tested (too soft)		0.17
WG ₆	1	>350	–7.08	–14.8	No separation
WG ₇	2	>350	–0.28	–11.7	No separation
WG ₈	3–4	>350	–1.16	–15.3	No separation

amount of FS increased in the formulation. Furthermore, the addition of additive into the formulation also affecting the grease's dropping point, as MoS₂ was known for its better performance under both low and high temperature [26,27]. FS and MoS₂ is known for their high melting point of 1600 °C and 1700 °C, respectively. These results are in agreement with the previous studies by Abdulbari et al. [10] and Mohd Najib et al. [28], where there was no dropping point exist for grease formulated using FS even though the temperature exceed 350 °C and 240 °C, respectively. Therefore, the high dropping point of the formulated greases show that the greases are thermally stable and have good heat resistance over high temperature.

3.5. Oil bleeding of grease

Oil bleeding properties of grease refers to the tendency of grease to bleed oil. A certain amount of bleeding is considered desirable in greases since this tends to provide continuous oil lubrication to the applied area. Bleeding properties usually will depend on the grease's thickener structure, base oil viscosity and grease firmness. In this test, oil bleeding properties of grease was conducted by evaluating the oil bleeding difference between oil bleeding area of fresh grease and aged grease in both room condition and at 70 °C. According to Bots [29], when the bleeding difference is within plus or minus 15 percent (+15% > X > –15%), the grease still can be used without changing the re-lubrication intervals.

By referring to Table 4, the grease formulated without additive shows less oil bleeding at 70 °C compared to at the room condition for which, this indicates that the greases were already begun to dry out at this temperature. Lugt [30] stated that when the oil bleeding shows that there is 50% oil left in grease composition, the change in grease type and re-lubrication interval is required. Interestingly, the addition of MoS₂ in all grease formulation shows significant improvement in term of oil bleeding difference at both room condition and 70 °C. This indicates that MoS₂ were able to prolonged the grease lifetime. These results were in agreement with Hangzhou [31] where he found that MoS₂ could delay the process of grease deterioration. Similar to Bhardwaj et al. [32] which stipulated that the grease blended with MoS₂ shows lesser damage to thickener structure and lead to long grease life.

3.6. Oil separation from grease

After storage of 1 month, all grease samples were observed for oil separation on the surface of grease. The amount of oil separated from grease was shown in Table 4. According to ASTM standard [33], grease of NLGI less than 1 is not suitable for the standard test method. However, when the test was conducted, it was shown that the greases of consistency NLGI 000 were the only grease with oil separation, as expected due to their soft consistency. However, the

amount of oil separated from the grease was within the acceptable value of less than 4% [30]. FS function as thickener and tends to hold and attract the oil in its thickener system. WG₁ and WG₅ only contained 5% amount of FS for which apparently, it was not enough to hold 95% of oil. Insufficient amount of FS in the formulation results in the formation of oil layer on the grease surface [28].

3.7. Detection of copper corrosion from grease

Tables 5 and 6 show the corrosiveness results of the base oil and the formulated grease with and without additive. In this study, it was found that the ITO and WTO were only slightly tarnish towards copper strips at which the appearances were close to the freshly polished strips (Fig. 4). The corrosivity of the formulated grease using WTO with and without additive were similar (Classification 1 – slight tarnish). However, greases formulated using ITO seems to have different corrosivity level of classification 1 (IG₂) and classification 2 (IG₁). IG₁ was formulated using ITO without MoS₂. When compared to WG₁₋₄, FS might not responsible for the grease corrosivity, which is in lined with results obtained by Abdulbari et al. [10]. Aside from that, the addition of MoS₂ in grease formulated using ITO (IG₂) was found only slightly tarnish towards the copper strips. This viewpoint is not in line with other findings where the addition of MoS₂ were commonly found to increase the corrosivity of the grease [27,34]. However, this study found that the addition of MoS₂ in grease formulation may help in protecting metals from corrosion.

3.8. FTIR characterization of grease

FTIR analysis had been conducted to determine the functional group of grease samples formulated using both ITO and WTO, and with and without the addition of additive, respectively. The measurement was within 500–4000 cm⁻¹. Fig. 4 shows the grease samples spectra as the representative of all grease samples. There were no notable differences between all formulated greases.

By referring to Fig. 5, all grease samples have high intensity bands in the region of 2852–2951 cm⁻¹, as a result of the CH₂ and CH₃ asymmetric stretching. A strong band at 1455 cm⁻¹ and 1376 cm⁻¹ represented the C–H asymmetric bending vibrations indicating the functional group of alkane [20,21]. The presence of peaks at 1098 cm⁻¹ to 1100 cm⁻¹ were related to the Si–O–Si stretching vibration from the fumed silica thickener [35,36]. A weak band at 841 cm⁻¹ and 721 cm⁻¹ represented the C–H out of plane bend indicating the presence of alkanes [22].

Table 5
Corrosiveness of base oil.

Sample	Result
ITO	1a – slight tarnish (light orange)
WTO	1a – slight tarnish (light orange)

Table 6
Grease corrosiveness test.

Sample	Result
IG ₁	2a – moderate tarnish (claret red)
IG ₂	1b – slight tarnish (dark orange)
WG ₁	1b – slight tarnish (dark orange)
WG ₂	1b – slight tarnish (dark orange)
WG ₃	1a – slight tarnish (light orange)
WG ₄	1b – slight tarnish (dark orange)
WG ₅	1a – slight tarnish (light orange)
WG ₆	1a – slight tarnish (light orange)
WG ₇	1b – slight tarnish (dark orange)
WG ₈	1a – slight tarnish (light orange)

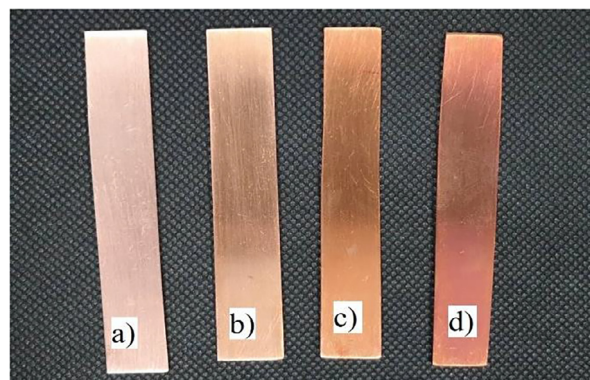


Fig. 4. Copper strips corrosion a) freshly polished b) class 1a–slightly tarnish c) class 1b–slightly tarnish d) class 2a–moderate tarnish.

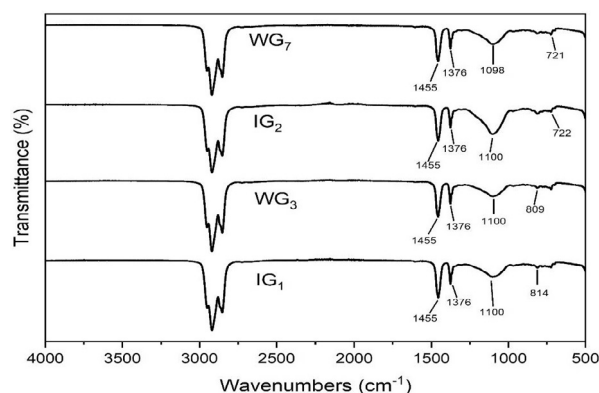


Fig. 5. FTIR characterization of formulated greases.

4. Conclusion

The formulation of grease using WTO is a new idea. Transformer oil is derived from petroleum resources which was in the same categories as lubricating oil. The possibility for WTO being used as new product (grease's base oil) could be a promising base oil alternative to replace the conventional fresh petroleum oil – which currently decreasing due to the declined of petroleum reserves and to create a green product. The physical properties of the formulated FS greases using WTO blended with MoS₂ is encouraging for future application. Further experimental work related to tribology is required to study the performance of the formulated greases for its ability to perform the grease's primary key function.

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Declaration of interest

None.

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