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Synthesis and properties evaluation of sodium grease formulated from used transformer oil as base oil

N S A Japar¹, M A A Aziz^{1*} and N Hamid²

¹ Faculty of Chemical and Process Engineering Technology, University Malaysia Pahang, 26600 Kuantan, Pagang, Malaysia.

² Foresight and Strategic Unit, Vice Chancellor Office, University College of Yayasan, 25200 Kuantan, Pahang, Malaysia.

*E-mail: maizudin@ump.edu.my

Abstract. This study aimed to prepare sodium grease using used transformer oil as a base oil and investigate the prepared grease properties. Oil, whether it is new or used, is a significant contributor to water pollution due to its toxicity towards the environment. In this study, used transformer oil (UTO) is utilized as grease's base oil. The properties of the grease were evaluated in terms of their consistency, drop point, oil separation, and bled oil in accordance with ASTM D217, D2265, D1742, and IP 121, respectively. The grease also characterized by using the FTIR-ATR. The results show that the grease can be formulated using UTO with significant grease properties of >180 °C dropping point, -15% to +15% of oil bleeding at both 25 °C and 70 °C, no separated oil during storage, and slightly tarnish on copper strip. The best UTO-based sodium grease formulation was 67.5:32.5 of oil-to-thickener ratio and it was classified as NLGI 2 grease. Moreover, the FTIR spectrum shows no contaminants or unknown chemical changes or additive in UTO and the prepared grease. The addition of MoS₂ in grease shows no significant improvement in sodium grease properties. Thus, UTO was able to work as grease base oil and exhibited overall good properties but, UTO-based grease analysis on its tribology is necessary to study the prepared grease performances during operation.

1. Introduction

Transformer oil is commonly used for insulation and cooling purpose. Petroleum oil, synthetic ester, and silicon oils are traditionally used as transformer oil. Transformer oil is usually a highly-refined mineral oil that is stable at high temperatures and has excellent electrical insulating properties. In the operation of an electrical transformer, the transformer oil is subjected to mechanical and electrical resistance, which results in oil deterioration and degradation. The discarded transformer oil is known as used transformer oil (UTO). The UTO generated in Malaysia is currently does not being utilized into new products or undergo an oil treatment process to reclaim the oil yet. Moreover, the quantity of UTO generated is increasing day by day due to the installation of new transformers and from scrap of old transformer [1]. Malaysia's power industry transformer unit has been introduced with only one type of transformer oil product. This transformer oil is a PCB-free uninhibited mineral oil made from a severely hydro-treated wax-free naphthenic oil. Therefore, the use of UTO generated by this industry could help to ease up lubricating grease composition control since there is no unknown additive associated with this UTO.

Current researches related to UTO are more focused on reclaiming it to produce diesel-like fuel (DLF) [2–4]. There are not many initiatives in using UTO as lubricating grease alternative base oil. The



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majority types of waste oils being used in lubricating grease formulation are either waste engine oil (WEO) or waste cooking oil (WCO) [5–7]. For example, Ebisike et al. [6] successfully produced sodium grease from spent engine oil, and the grease produced exhibit good properties of dropping point up to 200 °C and classified as NLGI 2 – 3 grease. Razali et al. [8] also formulated grease from waste oil with two types of thickeners (red gypsum and fumed silica), and the grease had a drop point of more than 240 °C. Formulation of grease using WCO was conducted by Abdulbari and Zuhan [7] where they found the formulated grease had no drop point and slightly corrosive to copper.

However, several studies were successfully conducted where transformer oil was utilized as grease base oil [9–11]. For example, transformer oil was utilized by Hassan et al. [12] and blending the oil with lube oil to produce grease with excellent insulating properties. Studies conducted by Cao et al. and Ge et al. [10,11] also found that transformer oil was able to provide insulating properties to the grease and improved with the addition of selected additive into the grease formulation. Therefore, it was expected that UTO could be utilized as lubricating grease base oil in this study.

Therefore, the present study aims to prepare sodium grease using used transformer oil as grease base oil and to investigate the prepared grease characteristics. The greases were prepared by stirring and homogenizing variation of grease component proportions of base oil and thickener, and a fixed amount of additive. The greases properties were evaluated and compared between greases containing fresh and used transformer oil, and greases with and without the presence of the additive.

2. Experimental work

2.1. Materials

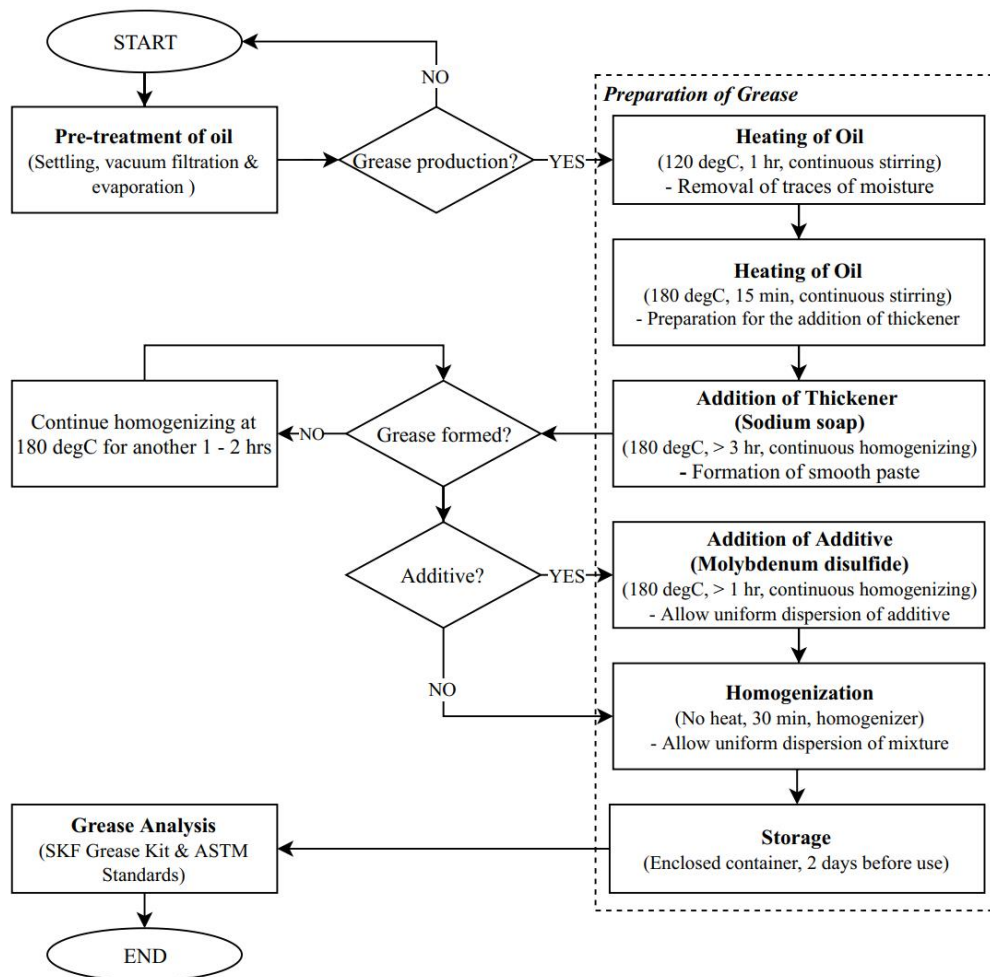
Used transformer oil (UTO) collected from an electrical power station located in Selangor, a fresh uninhibited industrial-grade transformer oil (FTO), sodium stearate, and molybdenum disulfide (MoS_2) with 98.5% purity.

2.2. Pre-treatment of used transformer oil (UTO)

Pre-treatment of UTO was carried out through settling, vacuum filtration, and heating to remove unwanted contaminants such as water and suspended dirt in the oil. Water was separated from UTO through gravity-based separation by allowing the water to settle at the bottom of the storage container within 2 weeks. The water was then removed from the container, leaving only UTO. Filtration of UTO via vacuum filtration using a vacuum pump and a glass microfiber filter with 1.2 μm pore size was then carried out to remove any solid particles suspended in the oil. Finally, filtered UTO was heated at 120 °C for at least 2 hours with continuous stirring to remove moisture and any volatile compound presented in the oil through the evaporation process. Heated UTO was then cooled down to room temperature before it can be stored in a new cleaned storage container prior to use.

2.3. Preparation of sodium grease

Grease formulation using both fresh transformer oil (FTO) and used transformer oil (UTO) as base oil was formulated. There were two formulations involved, which were formulated grease with and without the addition of additive (MoS_2), respectively. The preparation of sodium greases was started by heating the base oil at 120 °C for at least 1 hour with continuous stirring to remove traces of moisture. The oil temperature was then increased to 180 °C for 15 minutes and stirred continuously. Sodium stearate was added gradually into the oil, and the mixture was stirred for at least 3 hours until the smooth paste was obtained. The homogenization of the grease sample was conducted for at least 30 minutes without the presence of heat to allow uniform dispersion of mixture. As for the additive, MoS_2 was added into the mixture after the smooth paste was formed, and MoS_2 was stirred into the mixture for another 1 hour to allow even dispersion of additive. After homogenization, grease was stored in an enclosed container for 2 days to allow the grease to cool down. Grease preparation steps were as in figure 1, and the formulation of FTO-based grease (FG/FGM) and UTO-based grease (SG_i/SGM_i) was as shown in table 1.

**Figure 1.** Flowchart of the study procedure.**Table 1.** Formulation of the grease.

Sample		Grease composition (wt%):			
		FTO	UTO	Sodium soap	MoS ₂
FG	(No additive)	70	—	30	—
FGM	(With additive)	68.6	—	29.4	2
SG ₁	(No additive)	—	90	10	—
SG ₂		—	90	10	—
SG ₃		—	82.5	17.5	—
SG ₄		—	75	25	—
SG ₅		—	67.5	32.5	—
SG ₆		—	60	40	—
SG ₇		—	60	40	—
SGM ₁	(With additive)	—	88.2	9.8	2
SGM ₃		—	80.85	17.15	2
SGM ₄		—	73.5	24.5	2
SGM ₅		—	66.15	31.85	2
SGM ₆		—	58.8	39.2	2
SGM ₇		—	58.8	39.2	2

2.4. Grease characterization

2.4.1. Consistency. This test was carried out using the SKF Grease Test Kit TGKT 1. The consistency of the grease was measured using a calibrated measuring scale of NLGI grade. This test method was in accordance with ISO 2137, for which only a small amount of grease sample is required. The consistency level of the greases was classified by NLGI grade (table 2) [13].

Table 2. NLGI classification system based on consistency.

NLGI Grade	Worked Penetration, mm/10	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	
4	175-205	Stiff
5	130-160	Stiff
6	85-115	Hard solid

2.4.2. Dropping point. The dropping point test was evaluated according to ASTM D2265 for a wide temperature range [14]. In this test, the sample in the grease test cup was placed in a test tube and placed inside the aluminum block oven. A thermometer was placed in the test tube and positioned that it measures the temperature in the sample cup without coming in contact with the grease. As the temperature increases, at some point, a drop of material fell from the cup, and the reading of thermometer (ODP) and block oven temperature (BT) were recorded to the nearest degree range [14]. The dropping point (DP) of the grease sample was calculated using equation (1).

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

2.4.3. Oil bleeding. This analysis was conducted using the SKF Grease Test Kit where an only small volume of samples was required [15]. In this analysis, grease sample was placed on a blotter paper and heated on a hotplate at 60 °C for 2 hours [16]. The grease's bleed area percentage differences were measured and calculated by using equation (2) and equation (3) between fresh and aged grease. Where, S_i is the bled area from fresh and aged samples, D_{avi} is the average diameter of the bled area, and %Diff represents the bled area difference between fresh and used samples. Aged greases are greases that have been aged for 10 days at both 25 °C and 70 °C [17].

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

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

2.4.4. Oil separation. The test was carried out on grease that has undergone a 1-month storage period. The amount of oil separated on top of the grease sample during storage was collected and measured in terms of their weight percentage.

2.4.5. Corrosion test. Corrosion tests on both base oil and greases were carried out according to ASTM D130 and ASTM D4048, respectively [18, 19]. This test was performed by preparing a copper strip and totally immersing the strip in a sample of oil or grease. The sample was heated in an oven or liquid bath at a specified temperature for a period of time. The condition for the test was set at 100 °C for 3 hours in a water bath for base oil and at 100 °C for 24 hours in an oven for grease sample. At the end of the test period, the strips were removed from the sample, washed and compared to the ASTM Copper Strip Corrosion Standards (figure 2) [19].

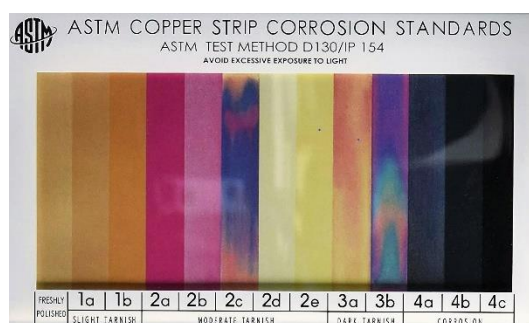


Figure 2. ASTM copper strip corrosion standards.

2.4.6. FTIR characterization. Fourier transforms infrared (FTIR) spectroscopy were used to identify the chemical compound that existed in the grease. FTIR spectrum can provide information on any contamination, additives, and changes presented in the grease. In this experiment, FTIR spectrums were measured using Thermo Scientific Nicolet iS5 FTIR Spectrometer via the FTIR-ATR approach at wavenumber of 500 – 4000 cm^{-1} .

3. Results and discussion

3.1. Physicochemical properties of oil

Table 3 shows the physicochemical properties of FTO and UTO. Grease mainly consisted of more than 80% of base oil where it provides lubrication when applied in an application. Base oil viscosity is the most important properties to be considered when formulating grease. High viscosity oil flows slowly compared to low viscosity oil. In low-speed applications, high viscosity oil is more favourable, and low viscosity oil is recommended for high-speed applications. The choice of base oil viscosity for grease formulation is depended on the grease intended application. Based on table 3, FTO and UTO were a low viscosity oil of 9.84 cSt and 9.93 cSt at 40 °C, respectively when compared to conventional mineral oil hence, these oils can be used in grease formulation for high speed and low load application [20]. However, the oil viscosity can be improved by the addition of proper additive into the oils. The moisture content of UTO after the pre-treatment process was reduced to 0.05%, which was within the range of maximum water content (0.02 to 0.06 %) that can be held by most industrial oil [21].

Table 3. Physicochemical properties of FTO and UTO.

Properties	Fresh transformer oil	Used transformer oil
Appearances	Clear & bright	Bright Yellow
Kinematic viscosity		
at 40 °C, cSt	9.84	9.93
at 100 °C, cSt	2.55	2.55
Viscosity index (VI)	80	77
Moisture content, %	0.002	0.05

3.2. Consistency of greases

Based on table 4, FG, FGM, SG₄, SG₅, SGM₄, and SGM₅ were classified as NLGI 1 – 3 greases, where these consistencies were most common for commercialized grease [22]. NLGI 2 greases were suitable for moderately loaded with medium speed applications. Grease of this class of consistency is formulated to give a good balance of properties required for easy pumping through dispensing systems [23]. Some of the formulated greases that classified with consistency lower than NLGI 1 are considered as soft greases, and this might be due to the insufficient amount of thickener in the formulation to hold the oil in its matrix. However, these soft grease consistency can be increased by decreasing the oil-to-thickener

ratio [24]. Conversely, greases with softer consistency do not mean it cannot be used in any application. Greases of such consistencies can be used at low operating temperatures and high-speed applications. Therefore, the grease consistency may be adjusted in the formulations according to the application's operating condition. Grease classified with NLGI > 3 was considered as hard grease for which this grease usually utilized at an application where leakage is a concern.

The addition of MoS₂ in sodium grease shows significant changes in the grease's consistency. For example, FG and FGM were formulated with the same oil and thickener composition of 70:30, but FGM contained 2 percent MoS₂, and FGM's consistency is one NLGI grade stiffer than FG. These results were not inlined with Mohammed [25], where he found that the addition of MoS₂ in different types of greases clearly reduces the grease consistency.

Table 4. Formulated grease properties.

Grease sample	Consistency (NLGI Grade)	Dropping point (°C)	Oil bleeding (%)		Oil separation (%)
			@ 25°C	@ 70°C	
FG	2	194.7	- 7.54	-36.81	0
FGM	3	193.9	- 13.72	-58.79	0
SG ₁	000	138	Too soft	Too soft	Too soft
SG ₂	000	135			
SG ₃	0	158	16.53	-9.54	0.27
SG ₄	1	175	9.78	-11.67	0.07
SG ₅	2	193	-8.62	-18.04	0
SG ₆	4	207	-28.70	-35.57	0
SG ₇	4	204	-27.85	-35.84	0
SGM ₁	00	143	Too soft	Too soft	Too soft
SGM ₂	00	141			
SGM ₃	0	170	10.42	3.31	0.33
SGM ₄	2	181.2	2.21	-3.67	0.12
SGM ₅	3	198.4	-11.16	-16.64	0
SGM ₆	5	203.1	-58.02	-60.06	0
SGM ₇	5	201.2	-59.08	-61.43	0

3.3. Dropping point of the grease

Table 4 also shows the results of the dropping point test for the formulated greases using both FTO and UTO with and without the additive. According to Totten [26], dropping point is dependent on the type of thickener and the cohesiveness of the oil and thickener of a grease. Based on the results, the grease's dropping point temperature increases with the amount of thickener. This is in line with Buhlak et al. [27], it was found that when the thickener consists of 90:10 percent of sodium-to-lithium soap mixture, the drop point of formulated greases with 20% and 25% of thickener has increased from 119 °C to 128 °C, respectively.

Sodium greases do not have a high dropping point, typically 175 °C. In this study, the dropping point of the prepared greases was increasing with the amount of thickener. The drop point of both FTO-based and UTO-based greases shows high dropping point of more than 175 °C at thickener content of above 17.5%. This result is in agreement with Ebisike et al. [6], where grease formulated using spent engine oil and sodium soap exhibit drop point of 177 °C – 182 °C. Grease produced by Iheme et al. [28] using mineral oil and sodium soap also had comparable high dropping point of 165 °C – 190 °C to this study. Conversely, the addition of MoS₂ into the formulation does not shows an improvement on the dropping point, but it seems to maintain the dropping point within 186 °C ± 15 °C. This result is similar to studies conducted by Mohammed [25], where he stipulated that there was no change in the grease drop point when MoS₂ was added into the formulation of different types of greases.

3.4. Oil bleeding of the grease

Oil bleeding is the tendency of grease to bleed oil. A certain amount of bleeding is desirable in greases to provide continuous lubrication. Oil bleeding from grease depends on the thickener structure, base oil viscosity, and grease firmness. Bots stated that the oil bleeding differences of -15% to $+15\%$ are desirable as it indicated that the grease still can be utilized without changing the re-lubrication intervals [29]. The positive value stipulated for more oil bleeding occurs after grease is used where it indicated the possibility of a broken thickener structure during operation hence lead to the need for frequent re-lubrication. A negative value represents the drop of oil bleeding with respect to used grease which indicated grease dry out during operation [29].

Figure 3 shows that only some of the formulated greases achieved the recommended oil bleeding values. The amount of thickener in the formulation was found to affect the grease's oil bleed. Thickener helps to hold the oil in its system but, an excessive amount of thickener was found to hinder the oil from bleeding from the grease. For example, at SG₃, positive value of oil bleeding exceeding $+15\%$ shows that more oil was bled due to its soft consistency. As the thickener was increased to 32.5% , the oil bleeding remained within the range. However, at 40% of thickener content, high negative oil bleed values were obtained, which indicates the grease was dried out during the operation as if the oil was unable to bleed out from the thickener or the oil itself has evaporated. This is in line with Dixena et al. [30] where they stated that "an increase in oil bleed tendency was observed with decrease in thickener content and vice versa in both the thickener types PP and mPP over entire temperature range studied."

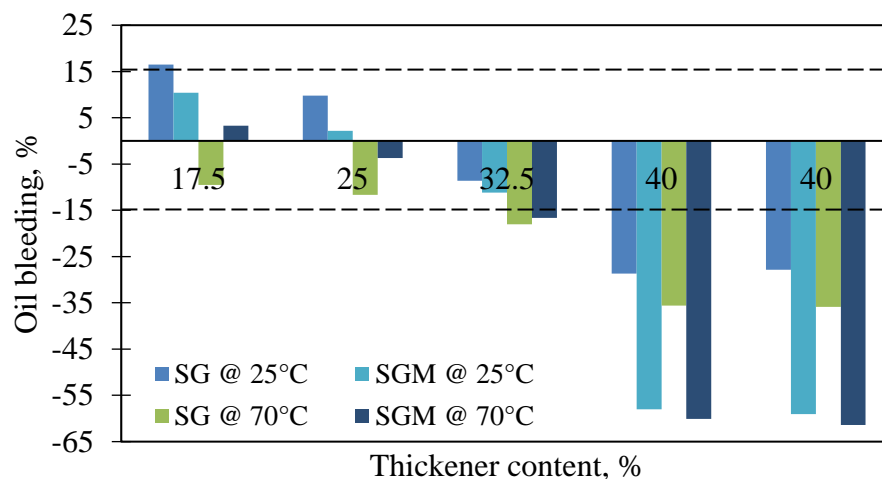


Figure 3. Oil bleeding of grease.

The presence of MoS₂ in grease formulation shows different results where it was found to reduce the tendency of grease to bleed oil. For example, SG₃ and SGM₃ share the same percentage of oil-to-thickener content of $82.5:17.5$ percent. SG₃ oil bleed was observed to exceed the recommended values at $25\text{ }^{\circ}\text{C}$ but, with the presence of MoS₂, SGM₃ oil bleed falls within the range from 16.53% to 10.42% . However, oil bleed of greases with 40% of thickener content has exceeded the range. From this, it can be drawn that MoS₂ does not improved or helps in stabilizing the grease oil bleed, but it reduced the tendency of the grease to bleed oil. This finding is similar to Sinitsyn et al. [31] where they found significant changes in grease oil bleed from 31.2% to 23.5% when the amount of MoS₂ was increased from 0% to 3% , respectively. Therefore, MoS₂ can be added to the formulation of grease with high oil bleeding percentage.

3.5. Oil separation of the grease

Oil separation on all grease sample surfaces was observed and tabulated in table 4. According to the ASTM D1742 standard [32], greases of NLGI less than one (1) are not suitable to be tested using the

standard test method. However, out of all greases, only SG₃₋₄ and SGM₃₋₄ show oil separation of top of the sample surface. The oil separated from SG₁₋₂ and SGM₁₋₂ were not able to be identified as the grease is very soft and in semifluid form. The amount of oil separated from SG₃₋₄ and SGM₃₋₄ which were 0.27%, 0.07%, 0.33% and 0.12%, respectively were within the desirable value of less than 4% [33]. The oil separation was observed to reduce when the percentage of thickener content was increased.

3.6. Detection of copper corrosion from the grease

According to Verdura et al. [34], “This test does not determine the ability of a grease to inhibit copper corrosion caused by factors other than the grease itself; neither does it determine the stability of grease in the presence of copper. The sole determination is the chemical staining of copper by lubricating grease”. Table 5 shows the corrosiveness results of the base oil and the formulated greases towards copper. FTO and UTO were found only slightly tarnish towards copper, thus concluded that the base oil appeared to be not corrosive towards copper strips. According to Mohammed [25], sodium grease was classified as moderately corrosive (2a) towards copper when no additive added, and the addition of 3% MoS₂ into the formulation was found to improve the grease corrosiveness.

In this study, all grease corrosiveness was classified as class 1 on copper strips. According to Mohammed [25] and Sinitsyn et al. [31], MoS₂ acted as a corrosion inhibitor in grease formulation. However, referring to table 5, it was found that all UTO-based greases without MoS₂ (SG_i) demonstrated a slight tarnish to copper strips. Therefore, blending 2% of MoS₂ into the prepared greases (SGM_i) shows no significant effect on grease corrosiveness towards copper strips as both SG_i and SGM_i were classified as class 1 grease corrosiveness. It was concluded that FTO, UTO, sodium stearate and MoS₂ were not corrosive to copper strips when utilized in grease formulations.

Table 5. Results of oil and grease corrosiveness test.

Sample	Corrosiveness
FTO	1a – slight tarnish
UTO	1a – slight tarnish
FG	1a – slight tarnish
FGM	1b – slight tarnish
SG ₁	1a – slight tarnish
SG ₂	1a – slight tarnish
SG ₃	1b – slight tarnish
SG ₄	1b – slight tarnish
SG ₅	1b – slight tarnish
SG ₆	1b – slight tarnish
SG ₇	1b – slight tarnish
SGM ₁	1b – slight tarnish
SGM ₂	1a – slight tarnish
SGM ₃	1b – slight tarnish
SGM ₄	1a – slight tarnish
SGM ₅	1a – slight tarnish
SGM ₆	1b – slight tarnish
SGM ₇	1b – slight tarnish

3.7. FTIR characterization of the grease

Figure 4 shows the FTIR spectrums of selected sodium greases that represent all samples. FTIR spectroscopy was used to determine the functional group of each chemical compound presented in greases. The measurement was within 500 – 4000 cm⁻¹. The FTIR spectrums show that all samples have the same strong peaks in the region of 2849 – 2951 cm⁻¹, which indicates the CH₂ group asymmetric and symmetric vibration. Weak peaks were observed at 3371 – 3390 cm⁻¹ that attributed to O-H stretching vibration [35]. The functional group of symmetrical stretching vibration of COO group was observed at

1558 cm^{-1} . A peak at 1455 cm^{-1} indicates the asymmetric CH_3 deformations [36]. The functional group of alkane C—H bond stretch of CH_3 bond was observed at about 1376 cm^{-1} [37]. Overlapping of CH_2 rocking vibration and long aliphatic hydrocarbon vibration at about 722 cm^{-1} , which also indicates the presence of alkanes [38, 39].

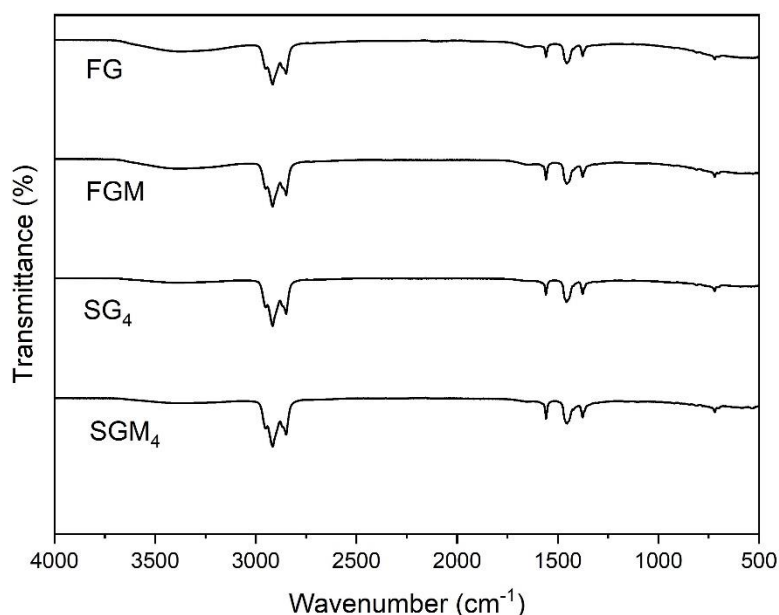


Figure 4. FTIR spectra of sodium greases.

FTIR spectra of all types of grease show no significant differences either being formulated with FTO or UTO, with or without MoS_2 . There were no clear peaks in the grease, which indicates the presence of contaminants or any by-products such as dissolved gases, oxidation products, and etc. The spectra show similarity due to the same chemical used in the grease for which the amount varies. These FTIR spectra prove the absence of any contaminants in the greases.

4. Conclusion

The utilization of used transformer oil (UTO) in grease formulation and the characterization of the grease were evaluated. The best formulation for grease employing UTO is 67.5:32.5 (UTO:Thickener) with 199 °C dropping point, −8.62%, and −18.04% of oil bleeding at both 25 °C and 70 °C. The grease was classified as NLGI 2 grease. Corrosion analysis of the grease indicates that the addition of MoS_2 into the formulation does not affect the grease corrosiveness towards copper strips. FTIR spectra of all greases show no significant difference. Further grease analysis with regards to its tribology is required to study the grease's performances (wear and friction properties) during operation.

Acknowledgements

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