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Sodium grease formulation from waste engine oil

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Abstract. The present study was conducted to formulate sodium soap grease using waste engine oil (WEO). Greases were formulated by homogenizing sodium soap thickener in WEO for 2 hours at 150 ℃ Different grease composition of oil-to-thickener ratio was designed which are 90/10, 80/20, 70/30 and 60/40. The formulated greases were analysed by conducting the consistency, oil bleeding, oil separation and FTIR characterization tests. The present study found that grease can be formulated using WEO and the thickener percentage have significant effect on formulated greases properties. Grease formulated with 70 – 80% of base oil and 20 – 30% of thickener was the best grease formulated as it shows desirable grease properties.

1. Introduction

Lubricating grease is a semi-solid material representing a dispersion of a thickener in a liquid lubricant [1]. It consist of three component which are oil, thickener and additives. Additives and base oil play the major roles in the grease formulation while the thickener is referred to as a sponge that hold the lubricant together. Mineral oil are often used in grease production as their fluid component. However, a better stability can be find in synthetic base oil during extreme temperature (high or low) [2]. To set the grease apart from the fluid lubricants, the thickener is used. Thickeners are molecules, polymers or particles that are partially soluble in lubricating fluid.

Earliest production of grease is from the early Egyptian or Roman eras by combining lime with oil. The product of this saponification produce calcium grease when the lime saponified the triglyceride that comprises oil. Starting from that, the development of thickeners have a big impact in grease technology. Lithium thickener has dominated approximately 70% of the grease today [3]. They searched for additives that are used to extend the capabilities of the basic product. However, this concept which is over 60 years old invented by Clarence Earle in the 1940's are the base of a current grease production. The formulator in the 1970's and 1980's target was one single grease which would be capable of a superior performance in a wide range application, temperature and environment. Unfortunately, it was too expensive to used multi-purposed grease for a basic application.

In modern days, the impact of grease production has increase environmentally and economically. As the production and consumption of oil is high every day, the disposal of the waste can give negative impact to the environment. This also bad for the environment as all the waste oil are categorized as the scheduled waste [4]. As the raw material for the grease production is a base oil, using waste oil can

reduce the numbers of waste oil that are being disposed to the landfill. It also can help to save the production cost of the grease as the composition of grease consist of 70%-90% of base oil.
Recycling waste oil to replace the base oil can reduce the production cost as the waste oil is much cheaper in price [5]. The main purpose of this study is to design formulation of soap base grease and the production of grease by utilizing waste engine oil.

2. Methodology

2.1. Materials
In this study, waste engine oil is used as base oil. The thickener used in this experiment will be sodium soap thickener. The sodium hydroxide will undergoes saponification process with stearic acid to produces sodium soap thickener. Stearic acid is one of the material needed for saponification process that have a long-chain fatty acid consisting of 18 carbon atoms without double bonds. In nature stearic acid occurs primarily as a mixed triglyceride, or fat, with other long-chain acids and as an ester of a fatty alcohol.

2.2. Pre-treatment of waste oil
Waste oil will undergoes pre-treatment in order to remove any impurities and water in the oil. Two step involves in the treatment which are filtering process and heating process. For filtering process, the oil will be filtered by using filter paper to remove any small impurities in the oil. Then the oil will undergoes second process which is heating the oil until the temperature higher than 90˚C with continuous stirring to remove water in the oil. This process will take about 1 hour with a temperature higher than 150˚C.

2.3. Formulation of soap base grease
In this formulation waste oil was utilized around 60 to 90 wt% and 10 to 40% of sodium soap thickener was dispersed in the waste engine oil (WEO). The WEO was heated to > 90˚C and stearic acid was melted at 60˚C. 70% of the heated oil was transferred into the melted stearic acid and homogenized for about 15 minutes. 6M NaOH solution was then added into the mixture drop by drop to control excessive foaming at low speed. After the addition of NaOH, the temperature was increased to 150 ℃ and homogenized for 30minutes. The oil balances were then added completely to the mixture and homogenized for 2 hours at high speed of 8000rpm and constant temperature. Finally, heat was removed from the sample and homogenization was continued for another 30 minutes. Grease sample was stored in an enclosed container and allowed to cool to room temperature for 2 days.

2.4. Analysis of grease

2.4.1. Consistency. Consistency test was carried out to evaluate the level of softness and firmness of the grease ranging from NLGI number 000 to 6 using SKF grease testing kit.

2.4.2. Oil bleeding. Oil bleeding test was conducted to study the oil bleeding area if the grease for fresh and used grease. In this test, greases were heated on filter paper for 2 hours at 60 ℃ [6]. Oil stained created after the test was measured, calculated and compared between fresh and used grease. It is desirable for the grease to bleed oil within the range of +15% to -15% of oil bleed different between fresh and used grease [7]. This range shows that in an operation, the grease can still be used without changing the re-lubrication intervals. Positive values shows that bleeding is increased due to the broken of grease's thickener structure. Negative values represent the reduction of oil bled at which during the grease was used in an operation, the grease dried out before the re-lubrication interval and the surface is 'hungry' for lubrication. In this analysis, used grease is the grease that was aged in an oven for 10 days at 70 ℃.

2.4.3. Oil separation. Oil separation test is a test to determine the grease tendency to separate oil during storage which correlate directly with the oil separation occurs in 35-lb pails of grease during storage. This test was carried out at 25 ℃ after the grease being stored for 2 months where oil separated on top of the grease were collected and measured. It was desired for the oil separated from the grease to be < 4% by weight [8].

2.4.4. FTIR Characterization. The base oil and the greases were characterized using Fourier-transform infrared (FTIR) spectroscopy to study the compound existed in the oil and grease.

3. Results and discussion

3.1. Consistency

Table 1 shows the consistency of each formulated greases in accordance to NLGI number ranging from 000 to 6. It was found that as the thickener percentage in grease formulation increases, the consistency of the grease becomes more firm. Commonly, the grease available in the market possess grease consistency of NLGI no. 1 to no. 3 [9]. From this test, grease of 20 – 30% of thickener content shows the most desired consistency of NLGI no. 1 to no. 3.
10% of thickener content possessed softer consistency compared to the other grease. This is due to the insufficient amount of thickener to hold the oil in its thickener system which finally led to softer consistency. This also applies towards grease with consistency more firm than NLGI no. 3 at which the amount of thickener were too excessive which cause the lack of oil content in grease. As the matter of fact, grease consistency can be altered by adjusting the oil-to-thickener ratio [10].

Table 1. Consistency number for grease formulation based on NLGI number.

Base oil (%)	Stearic acid (%)	Sodium hydroxide (%)	NLGI number
90	5	5	0
80	10	10	1 to 2
70	15	15	2 to 3
60	20	20	3 to 4
50	25	25	3 to 4

3.2. Oil separation

Oil separation usually occur when grease are being kept for a long period of times. The grease can be considered as good grease if the oil separation is under 4%. From this study, it can be seen that all formulated greases separated oil < 4% **(Table 2)**. There are no oil puddle observed for grease with thickener more than 30% where it can be concluded that the oil were fully hold by the thickener. Oil puddle is formed on top of grease formulated with less than 30% of thickener due to their soft consistency. But, oil contamination such as dust, dirt can also be the factor of oil being drew out from thickener system over time. The oil separation could be influenced by many factors such as the percentage of the base oil, the ability of the thickener to hold the oil, and the homogenize level of the finished grease.

Table 2. Oil separation of the formulated grease.

Base oil (%)	Stearic acid (%)	Sodium hydroxide (%)	Oil separation (%)
90	5	5	1.4
80	10	10	0.6
70	15	15	0.0
60	20	20	0.0
50	25	25	0.0

3.3. Oil bleeding

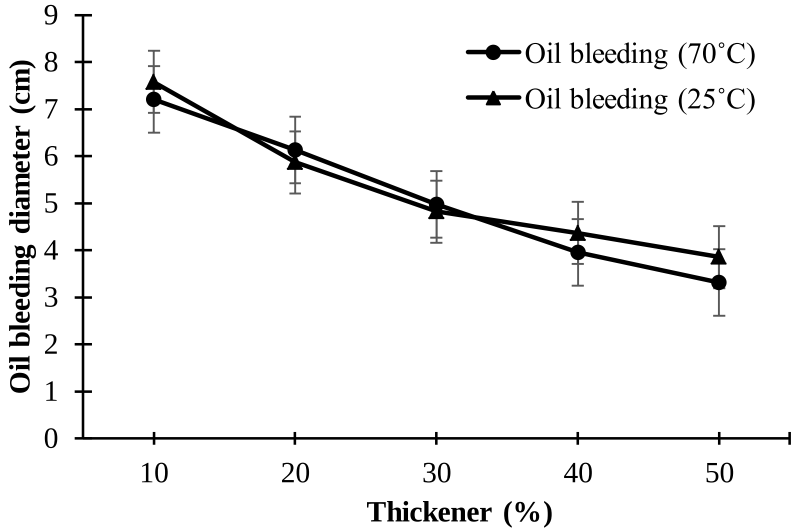
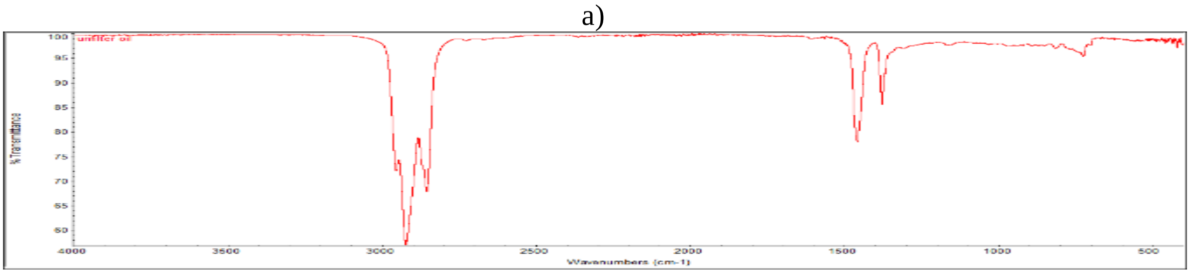


Figure 1. Graph of oil bleeding versus thickener percentage.

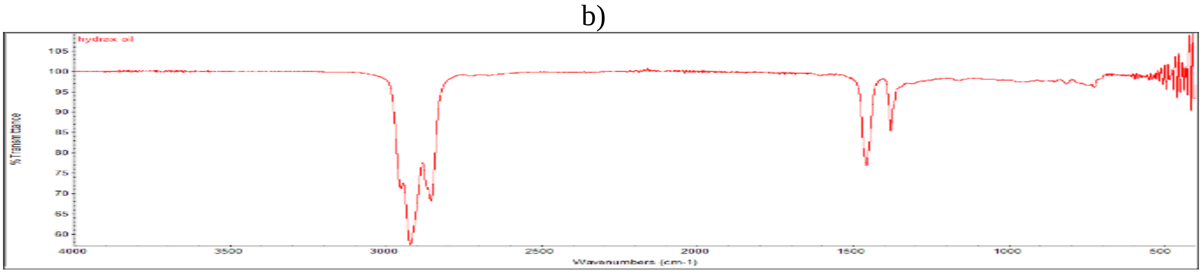
Figure 1 shows that as the amount of thickener had significant effects on the formulated grease's oil bleeding. In this analysis, oil bleeding was tested in two different condition which are at room condition of 25 ℃ and at 70 ℃. The graph above shows that the oil bleeding are relatively linear to thickener percentage regardless of grease ageing condition. Oil bleeding happened when there is stress occur to the grease. Although this stress are extremely weak when be compared to the mechanical stress acted upon grease during usage, but it still can result in the release of a small amount of oil from the grease [11].

3.4. FTIR analysis

a)



b)



c)

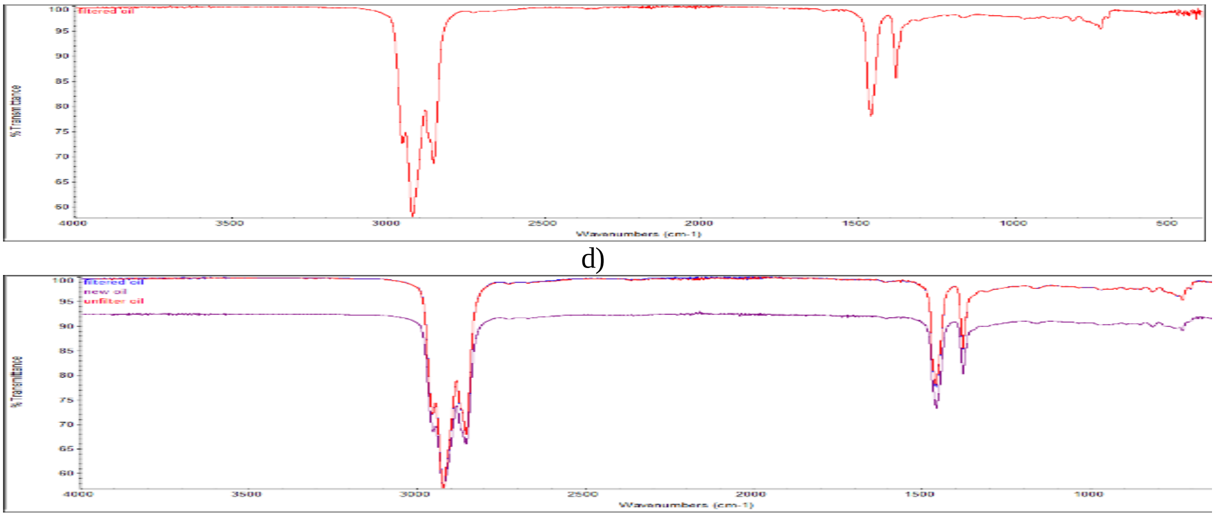


Figure 2. *FTIR analysis of (a) unfiltered waste oil, (b) new oil, (c) filtered waste oil, (d) comparison between oil.*

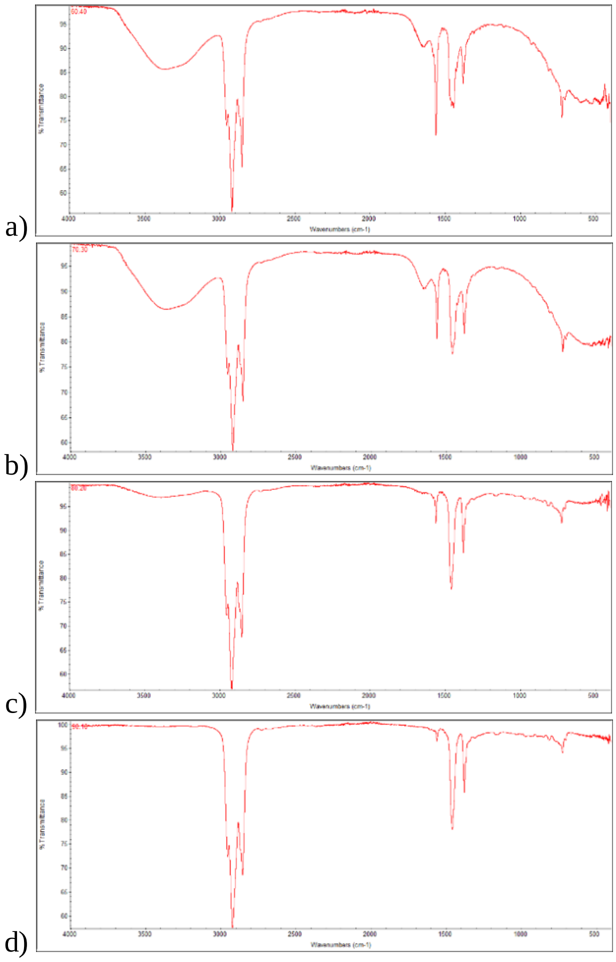


Figure 3. FTIR analysis of grease formulation: (a) 60:40, (b) 70:30, (c) 80:20, (d) 90:10

Figure 2 shows the FTIR analysis results for base oil and their comparison against each other. It was observed that there are no significant difference between new engine oil and waste engine oil (WEO) either it was filtered or unfiltered. It was also observe that there are no known contaminants presence in WEO.

Figure 3 shows the formulated greases FTIR analysis results. The FTIR spectrum for each greases are almost similar due to the same type chemicals were used in the formulation. However, the spectrum shows some variation on peaks as the thickener percentage were increased. Absorption peaks at 2973 cm^{-1} indicates the asymmetric stretching vibration of CH_3 group from the base oil. It was observed that there were visible peaks at 3400 cm^{-1} as the thickener percentage increases for which it indicates the presences of water or glycol compound in grease. This peaks was belongs to the thickener itself for which there are some traces of water produced when the soap was first formed during the reaction of NaOH and stearic acid [12,13].

4. Conclusion

Grease can be formulated from waste engine oil. FTIR spectroscopy has found that there were no significant differences between new oil, waste oil, and all formulated greases. Results of grease analysis shows that the thickener percentage have significant effect on formulated greases properties including consistency, oil bleeding and oil separation. Out of all formulation, grease formulated with 70 – 80% of base oil and 20 – 30% of thickener was the best grease formulation as it shows desirable grease properties of consistency within NLGI no.1 to no. 3, < 4% oil separation, and acceptable oil bleeding. This, it was proven that grease could be formulated using waste engine oil.

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