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Formulation of lubricating grease using Beeswax thickener

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Abstract. The issues on environmental pollution has brought the industries to seek the alternative green solutions for lubricating grease formulation. The significant challenges in producing modified grease are in which considering the chosen thickener as one of the environmental friendly material. The main purposes of the current research were to formulate lubricant grease using different types of base oils and to study the effect of thickener on the formulated lubricant grease. Used oil and motor oil were used as the base oils for the grease preparation. Beeswax and Damar were used as thickener and additive. The grease is tested based on its consistency, stability and oil bleeding. The prepared greases achieved grease consistency of grade 2 and 3 except for grease with unfiltered used oil. Grease formulated with used oil and synthetic oil tend to harden and loss its lubricating ability under high temperature compared to motor oil' grease. Grease modification using environmental friendly thickener were successfully formulated but it is considered as a low temperature grease as the beeswax have low melting point of 62°C-65°C.

1. Introduction

Most of the lubricants used nowadays lead to environmental pollution [1]. The environmental legislation by government bodies as well as higher consumer awareness are influencing the design process of the lubricating grease to employ more sustainable and environmentally friendly components [2]. Due to this scenario, a new market revolving around eco-friendly products is developing very rapidly, where consumers exhibits higher tendency to use new products and is also willing to pay for green products to conserve the environment from negative effects [3]. Several studies has been done related to the environmental friendly lubricating grease through the utilization of eco-friendly thickener such as cellulose derivatives and beeswax [4,5].

Grease is a semi-fluid product which comprises of a thickening agent along with other elements that provide the grease with special properties dispersed in a lubricating base oil. The common percentage compositions for base oils, thickeners and additives are 70-95%, 3-30% and 0-10% respectively [6]. Many studies has been done related to eco-friendly grease, where most researchers utilized vegetable oil as the base oil [7–9]. In contrast, the amount of studies done on thickeners are scarce. Thickeners are known to play a vital role in grease composition for which it determines the consistency of grease.

Used oils are generally generated during the engines service and maintenance however, it is often contaminated with impurities in the course of usage and handling. In addition, used oils often points out as a serious pollution problems due to irresponsible act of illegal dumping and unmanageable disposal resulting in groundwater, surface water and soil contamination[10]. Thus, in this study, used oils were utilized as one of the base oils to prevent the aforementioned problems.

The thickener's main function is to remain in contact with or hold lubrication from moving or leaking out under the force of gravity and often referred to as a sponge that holds the lubricants [11]. Beeswax is a natural substance obtained from the honeycombs of bees after the honey has been removed by draining or centrifuging. It possesses characteristic of insoluble in water, sparingly soluble in alcohol and very soluble in ether. In this study, beeswax was used as the thickeners in grease composition. Damar (*Shorea dipterocarpaceae*) are solid resins, generally hard and durable, were used as grease additive. It provides a unique depth of gloss, superior color and gloss retention, adhesion and flexibility [12], and it has melting point ranging from 85°C-115°C.

Lubricating grease functions in reducing the wear and tear of the surfaces by avoiding direct metal to metal contact between the rubbing surfaces and lowering the expansion of metal due to frictional heat and destruction of material. Motor oils and used oils, beeswax as well as damar were used to produce grease. This research study was carried out to formulate grease using different types of base oils and to study the effect of thickener on the formulated grease.

2. Methodology

2.1. Materials
Different types of base oils were used which are motor oils and used oils. The grade of the motor oils chosen were Toyota Premium Mineral (PM), Toyota Synthetic Formulation (SF) and Perodua Semi Synthetic (SS). Used oil collected from workshop was selected to be another alternative of base oils. Beeswax was chosen to act as a thickener, is a natural substance obtained from the honeycombs of bees after the honey has been removed by the process of draining or centrifuging meanwhile the additive, Damar (*Shorea dipterocarpaceae*) a solid resin is the sap from naturally wounds to the surface of the tree.

2.2. Preparation of grease samples
The compositions to produce 200g of grease samples were fixed with ratio of 14:5:1 of base oil, thickener and additive, respectively. 50g of beeswax was slurred together with half of the amount of base oil (70g) in a 500-mL beaker at continuous stirring rate. The speed of the mixer was adjusted according to the viscosity of the paste and for each of the sample, the mixing was continued until a smooth paste was formed at a constant temperature of 50°C-60°C. The other half amount of base oil and Damar (10g) were added slowly into the paste and the process continuous until all the materials blend together. The samples for each formulated grease were labelled as follows in accordance to its base oils types:

Table 1. Grease samples identification.

Type of base oil	Sample identification
Toyota Premium Mineral	PM
Toyota Synthetic Formulation	SF
Perodua Semi-Synthetic	SS
Used oil (unfiltered)	WO
Used oil (filtered)	FWO

2.3. Consistency test
The tests on each sample were performed using the SKF Grease Test Kit TGKT 1. A fixed grease volume was spread between the two glass plates 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 [13].

Table 2. NLGI classification system based on consistency.

NLGI Number	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

2.4. Stability test

The test was done as complied with the Standard Test Method for Oil Separation from Lubricating Grease during Storage (ASTM-D1742). It is known as oil separation test in which it determines the stability of the grease formulated. All grease samples were stored in enclosed container and the initial level of the grease is observed and recorded. The samples were left for 2 months at room temperature. The amount of oil separated was measured using the measuring cylinder and weighed. Theoretically, the lesser the oil separated, the better the stability or quality of the grease. The sample is considered stable if and only if the amount of the oil separated less than 4% [14].

2.5. Oil bleeding test

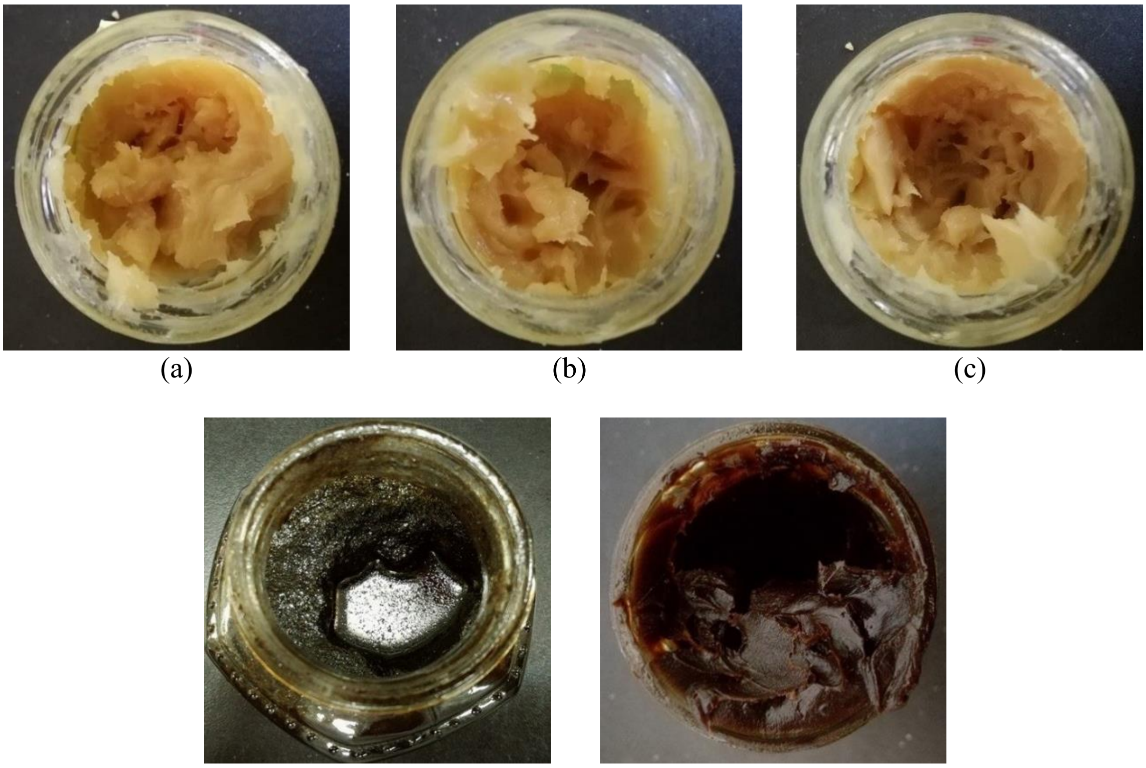
The tests on each sample were performed using the SKF Grease Test Kit TGKT 1. A fixed amount of grease was put on a piece of blotter paper provided. The samples were heated for two hours at constant temperature of 60°C according to the recommended operating condition of the grease test kit [15]. The base oil released from the grease created a stained on the paper. The bleed area and the percentage difference between bleed area of fresh and used samples were calculated by using equation (1) and (2).

$$S_{\dots} = 0.785 \times (D^2_{AV\dots} - 100) \quad (1)$$

$$\%Diff = 100 \times \frac{(S_{Used} - S_{Fresh})}{S_{Fresh}} \quad (2)$$

3. Results and discussion

3.1. Modified grease appearances



(a) (b) (c) (d) (e)

Figure 1. The appearances of modified grease samples. (a) Toyota premium mineral-oil-based grease, (b) Toyota synthetic-oil-based grease, (c) Perodua semi-synthetic-oil-based grease, (d) unfiltered used-oil-based grease, (e) filtered used-oil-based grease.

The modified grease samples prepared were semi-solid and their color depend on the color of base oil which vary from light yellow to black as in Figure 1. The properties of modified greases are as in the following subtopics.

3.2. Formulated lubricant grease consistency

The outcomes from the testing done upon each of the grease samples in accordance to the NLGI consistency grade (Table 2). The grades defined the consistency of the greases and their appearance at 25°C. SF-based grease, SS-based grease, and FWO-based grease were recorded NLGI grade 2 and PM-based grease of grade 3, where grease with these consistencies were most commonly used in general industrial, automotive and others [16]. Grease 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 through dispensing systems [17]. In addition, most of the multipurpose grease also of this grade of NLGI 2-3 consistency.

Table 3. Consistency of prepared modified grease.

Sample	NLGI	Consistency
PM	3	Firm
SF	2	Buttery
SS	2	Buttery
WO	00	Semifluid
FWO	2	Buttery

Comparing the WO-based grease and the other samples, very large difference in consistency observed for WO-based grease of which it is of NLGI grade 00. WO-based grease is softer than the other prepared greases. By comparing WO-based grease and FWO-based grease, FWO-based grease is formulated with filtered used oil. The presence of contaminants in the unfiltered used oil (WO) might be affecting the grease consistency for which most of these contaminants is removed before being used in FWO-based grease formulation [18]. Therefore, it was inferred that the WO-based grease consistency can be increase by decreasing the oil-to-thickener ratio [19].

3.3. Oil separation during storage
Greases tends to release their base oils during storage (static bleeding). Static oil bleeding can be affected by storage conditions and is more pronounced if the grease is soft in consistency (NLGI grades 00, 0 and 1) [20]. Oil separation often relates to the base oil and thickener combination with kinematic viscosity of the base oil [21]. All grease samples were stored at room temperature for 2 months and were observed for any changes of the behavior and properties. Table 4 shows oil separation only occurred to WO-based grease.

Table 4. Grease oil separation upon storage at 25ºC.

Sample	Oil separated (g)	%loss	Description
PM	-	-	Stable
SF	-	-	Stable
SS	-	-	Stable
WO	16.20	8.10	Unstable
FWO	-	-	Stable

WO-based grease was found to be unstable and recorded excessive oil separation which is more than 4%. Contaminants present in the unfiltered used oil can draw out the base oils from the thickener system over time [20]. However, the oil puddle on top of the grease does not result in the grease being unsuitable for use. It can be either removed by decanting the free oil from the surface or by manually stirring it back into the grease.

Prepared grease other than WO-based grease shown to have very good oil separation resistance. From the appearance of the other grease samples, the base oil and the thickening agent chosen seems to have a strong physically-acting bond forces that build a stable three-dimensional network structure [22]. Interestingly, filtered used oil-base grease (FWO) has good oil separation comparable to the fresh motor oils-base grease. This is might be due to the pre-treatment of used oil that was done such as settling, filtration and dehydration for which most of the contaminants in the used oil were being removed.

3.4. Oil bleeding of the formulated grease
This test method is not suitable for greases softer than NLGI grade 1 consistency due to the tendency for softer greases to sweep away while the test is performed. Therefore, the testing was not applicable on WO-based grease of grade 00. Comparison of grease bled between fresh samples and used samples grease were made. bled area difference (%Diff) between the used and the fresh sample were calculated

to indicated either the lubricating ability of the grease. Positive values mean the grease lubricating ability is increased or vice versa.

Based on the results in table 5, PM-based grease and SS-based grease shows the positive value in the percentage of area different which indicates that the grease lubricating ability can be maintained at high temperature. Other greases indicated reduction on their lubricating ability. SF-based grease and FWO-based grease losses its lubricating ability when subjected to high temperature for which it causes these greases harden as time goes. The rate of oil released from the grease will increase with time and vary based on the temperature at which it is stored [23]. It was observed that the formulated greases have low working temperature range which was found to have similarity with Kreivaitis' [5] study, as he stipulated that the dropping-point temperature of the beeswax-thickened greases were within the range of 52ºC to 66ºC. However, the formulated grease high temperature tolerance can be improved by adding proper additive to increase its dropping point temperature properties.

Table 5. Oil bleeding test at 60ºC.

Sample	$D_{AV\ fresh}$ (mm)	$D_{AV\ used}$ (mm)	S_{Fresh} (mm^2/s)	S_{Used} (mm^2/s)	%Diff
PM	34.0	36.5	828.96	967.31	16.68
SF	45.0	43.0	1511.13	1372.97	-9.14
SS	40.0	42.0	1177.50	1306.24	10.93
WO	N/A	N/A	N/A	N/A	N/A
FWO	35.5	34.0	910.79	828.96	-8.98

Table 6 shows the data analyzed from the tests carried out on each of the grease samples. The tests carried out includes the consistency test, stability test and oil bleeding test. Consistency test were done to determine the NLGI consistency grade for grease samples. In stability test, any changes in grease samples were analyzed after 2 months of storage at room temperature. Oil bleeding tests were carried out for two hours at constant temperature of 60°C where the base oil released from the grease of fresh and used samples created oil stained on the paper. Both the diameter of the stained and the bleeding properties were measured.

Table 6. Overall results of formulated lubricant grease analysis.

Sample	Color	Appearance	NLGI	Stability @ 25ºC	%Diff oil bleed @ 25ºC
PM	Light yellow	Firm	3	Stable	16.68
SF	Light yellow	Buttery	2	Stable	-9.14
SS	Light yellow	Buttery	2	Stable	10.93
WO	Black	Semifluid	00	Unstable with 8% oil separated	Not applicable
FWO	Dark brown	Buttery	2	Stable	-8.98

4. Conclusion

Beeswax-thickened greases are potentially an environmental friendly type of grease. Based on the result obtained, most of the formulated grease were successfully formulated by utilizing beeswax as thickener with NLGI consistency range of grade 2 – 3. However, these greases are not meant to be subjected to high temperature as it tends to lose it lubricating ability and harden as it exposed to high temperature for a period of time.

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