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Preparation of Grease using Organic Thickener

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Abstract

This paper aimed to study of the behaviour of organic thickener in the grease formulation. In this study different types of organic thickener (polypropylene, chitosan, cellulose) with different percentage has been tested in grease formulation. The percentage of base oil and thickener used were 65 to 75% of base oil and 25 to 35% of thickener. The formulation was done by dispersing the thickener in the base oil at temperature of 110°C for 3 hours. Several tests were conducted to evaluate the grease properties, including its consistency, oil bleeding, and oil separation. It was found that polypropylene-thickened grease shows most desirable properties with consistency of NLGI no. 1, > -15% of oil bleeding and < 4% of oil separation, compared to other organic thickener. It was shown that 70 – 75% of base oil with 25 to 30% of polypropylene thickener were the best formulation. However, all of the formulated greases were unable to withstand high temperature as it tends to fully liquefied.

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Keywords: Grease; Organic thickener; Chitosan; Cellulose; Polypropylene

1. Introduction

Grease is classified as semi-solid lubricant consisting 70-90% of base oil, 10-30% of thickener, and 5-10% of additives. It is usually utilized when liquid lubricants are impractical to be used; such as when a liquid lubricant cannot stay in the contact or for sealing purposes to protect the contact from contamination by debris, water or

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corrosive environment. Grease thickeners are divided into two categories which are soap thickener and non-soap thickener (inorganic or organic).

Non-soap thickened grease shares about 17% of the market share along with 83% of soap thickened grease. However, clay and polyurea thickeners often gaining attention for non-soap thickener due to their excellent properties of high dropping point, thermal stability, better consistency and water stability. Organic thickeners can be divided into two types which are pre-made, largely oil-insoluble materials, and in situ production by the reaction of the two or more ingredients. As a matter of fact, there is only one predominantly used organic thickener which is polyurea [1]. As polyurea thickener possess many desirable properties, this thickener is more expensive compared to soap thickener and have a poor pumpability characteristic.

It is a very interesting point that nowadays an organic material is being used in grease formulations, as they have strong bond which can hold the base oil. Grease which made from polymer has effectively proved in reducing friction between both objects. When used as thickener, the polymers form short and thick elements connected to each other, which trap the oil and modify its structure. The polymers are also added to improve the viscosity index and promote the adhesion of lubricating [2].

In addition, several studies related to the used of organic thickener has been conducted to find other potential alternative other than polyurea which are the used of chitin, acylated derivatives, cellulose and chitosan [3–5]. Chitosan is the second most abundant biopolymer after cellulose which possess interesting properties due to its biodegradability and non-toxicity. It is somewhat similar to vegetable oil [6].

In this study, it was aimed to evaluate the organic thickeners behaviour in grease formulation, which are chitosan, cellulose and polypropylene, and to study the grease properties in accordance to the thickener type.

2. Methodology

2.1. Materials

Fumed silica powder (Aerosil 200 type, surface area 200 m^2/g, 99% SiO_2 purity). Polypropylene ($(C_3H_6)_n$, 99% purity). Chitosan powder (deacylated chitin, low molecular weight) and cellulose ($(C_6H_{10}O_5)_n$, 99% purity). Castor oil as the grease's base oil.

2.2. Grease formulation

The manufacturing of this grease was done in batch process. The castor oil was preheated up to 110°C to remove any traces of moisture. The organic thickener was added into the base oil and continuously stirred until the thickener powder dispersed evenly in the base oil, which was around 3 hours. After mixing, the sample was cooled to room temperature which normally takes about 2 days. The organic thickener which being used are polypropylene, chitosan and cellulose. The percentages of base oil and thickener in grease formulation are 65 – 75% of base oil and 25 – 75% of thickener.

2.3. Grease testing

Several tests on grease's properties was conducted in accordance to ASTM's standard testing method for lubricating grease namely consistency test, oil separation test and oil bleeding test.

Consistency test was carried out by using SKF grease testing kit to study the level of softness and hardness of the formulated grease ranging from NLGI number 000 to 6 (Table 1). The higher the NLGI number represent more firm grease consistency.

Table 1. The classification of grease based on its penetration number.

NLGI Number	Worked Penetration (10^{-1} mm)	Appearance at 25 °C
000	445-475	Very fluid
00	400-430	Fluid
0	355-385	Semi-fluid
1	310-340	Very soft
2	265-295	Soft
3	220-250	Medium hard
4	175-205	Hard
5	130-160	Very hard
6	85-115	Extremely hard

Oil bleeding test was done in accordance with ASTM D4425. It determines the amount of oil separation after the grease is mechanically sheared as would occur in rolling element bearings or is exposed to high levels of centrifugation stress; which typically found in shaft couplings and universal joints, or pumped through a central system distribution block. The oil bleeding test method is an indicator to measure the point at which the grease changes its phase from semi-solid to liquid when introduced to heat.

The procedure is done by put a fixed amount of used and fresh grease on a piece of a blotter paper and heated to 60°C for 2 hours [7]. Oil stained created by the grease was measured and compared to each other. It is desirable for the grease bled area in between of +15% to -15% [8].

The oil separation test was conducted as described in ASTM D1742 to examine oil separation from grease during storage. This test method covers the determination of the tendency of lubricating grease to separate oil during storage in both normally filled and partially filled containers. The beaker was properly covered with aluminum foil and being left for overnight to see if any oil separation occurs [9].

3. Results and discussion

3.1. Consistency test

The NLGI (National Lubricating Grease Institute) grade is a widely accepted parameter to classify lubricating greases as a function of their consistencies obtained as penetration indexes. The NLGI grade increases with thickener concentration [3]. Table 2 shows the result of consistency test that has been done on all these three greases, while Table 3 shows the structure of grease at different NLGI value.

Table 2. NLGI value of greases after undergo consistency test.

Grease	Base oil to thickener percentage	NLGI number
Polypropylene	65/35	1
	70/30	1
	75/25	1
Cellulose	65/35	00
	70/30	000
	75/25	000
Chitosan	65/35	00
	70/30	000
	75/25	000
Fumed silica (inorganic thickener) – as comparison reference		2

Table 3. Structure of grease corresponds to their NLGI value.

NLGI number	Grease appearance	NLGI number	Grease appearance
000 (Chitosan)		1 (Polypropylene)	
00 (Cellulose)		2 (Fumed silica)	

From the consistency test that has been done, it can be seen that polypropylene seems to be the least oil bleed among them all. This is due to its structure which entangles and has strong bond with the base oil compared to cellulose and chitosan. Meanwhile, The NLGI value for both chitosan and cellulose is the lowest, which falls under NLGI number below than 0. This is because as force is exerted they loses their stability and cannot longer contain oil. Hence, in the section of oil bleeding test, polypropylene is the least in oil bleeding compared to cellulose and chitosan grease. However, it can be seen that the NLGI grade for all of these organic greases are ranging from 000 to 1, while NLGI for fumed silica falls under grade 2. Comparing these organic greases with fumed silica, it can be concluded that all the organic greases are categorized under soft grease. This is because common NLGI grade grease for industrial application is 2 and above. Thus, as these organic greases are soft grease, so they only can be applied for low temperature application, such as for house application [10].

3.2. Oil bleeding test

The percentage difference indicates the bled area difference between the used and fresh sample. If the percentage difference is negative, then bleeding is reduced. In contrast, if the result is positive, it means the bleeding is increased.

From oil bleeding test that has been done, it found that all the organic greases show negative percentage results. This situation occurs as the used greases have undergone ageing at temperature of 50°C. Comparing the fresh and aged organic greases, it seems clear that the ageing of grease significantly affects the oil bleeding. The amount of oil bleeding is reduced because at the ageing stage, oil evaporation has occurred for all of the organic greases, where they have lost significant amount of oil. This result also accordance with Goncalves et al. [2] which stated that the used greases show much lower oil bleed rates as the thermal ageing procedure applied in the oil bleeding test has lead oil loss by evaporation. Hence, that is why when, the diameter of the used grease is getting reduced when the oil bleeding rate for the used grease is been tested. Besides, the organic grease is also prone to oxidation as there is no additive which being added in this greases formulation. Hence, the decline of oil bleeding rate of organic grease gives the fact that grease formulated by organic thickener is limited to be use at high in-service temperatures [3].

Fumed silica grease shows positive percentage result on oil bleeding rate. Thus, it shows that the fume silica grease is still holding the oil in the grease even after going through ageing. Therefore, it is suitable and to be used without any required re-lubrication as the consistency between the oil and the thickener of the used fume silica grease is still maintained.

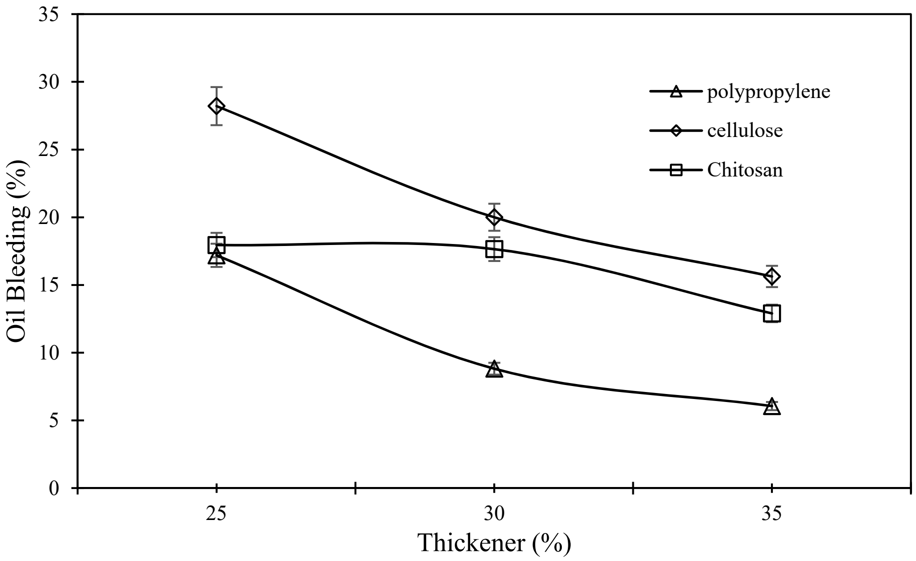

Fig. 1. Effect of thickener percentage on oil bleeding.

Fig. 1. shows that the percentage of oil bleeding significantly reduced as the percentage of the grease is increasing. This situation occurs because as the amount of thickener content is increase, the network of the thickener becomes stronger, thus decreasing the oil bleeding rate.

3.3. Oil bleeding test

Table 4. Oil separation from grease.

Grease	Base oil to thickener percentage	NLGI number
Polypropylene	65/35	0.0
	70/30	0.4
	75/25	0.8
Cellulose	65/35	0.8
	70/30	2.0
	75/25	2.8
Chitosan	65/35	0.8
	70/30	1.2
	75/25	1.6
Fumed silica (inorganic thickener) – as comparison reference		0.0

By referring to Table 4, all formulated greases are stable with < 4% oil separation. These results shows that as the thickener percentage was increased, the oil separation from grease were found to decrease along with the percentage of thickener. The small amount of oil separation from all formulated grease shows that this situation of a very thin layer of separated oil was due to a certain degree of particles sedimentation.

Comparing all organic greases to fumed silica grease, it can be seen that fume silica grease found to have no oil separation occurred even after 2 months of storage. This is due to the fact that fumed silica consists of microscopic droplets of amorphous silica which has an extremely large surface area and smooth nano-porous surface which could promote strong physical contact between the filler and the polymer matrix [11]. Hence, due to its fractal structure and its high specific area, it is prone to self-aggregation and can consequently form a very tight three dimensional network [12]. That is why the condition of fumed silica grease is still maintained even it stored for a long time.

4. Conclusion

This study was done to formulate grease by using an organic thickener. Based on the result of analysis, it was found that grease formulated with polypropylene thickener is the most stable grease compared to cellulose and chitosan with low oil separation, high consistency of NLGI no. 1, and in range oil bleeding percentage. The best formulation of polypropylene grease was at 65 - 70% base oil with 30 - 35% of thickener. However, all of these formulated organic greases can be used for low temperature application as at high temperature the greases started to lose their stability and liquefied but these are reversible.

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