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Effect of Carbon Nanoparticles (CNPs) as an Additive on the Performance of Food-grade Lubricant Grease Formulation

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Abstract

This study aimed to formulate food-grade lubricating grease using vegetable oil as base oil, aluminium isopropoxide blended with hydroxystearic acid as a thickener, and graphene (carbon nanoparticles) as an additive. This research also aims to analyse the effect of graphene as an additive in different formulations using different characterisation methods. The grease analysis studied was the penetration test (ASTM D-217) and the drop point test (ASTM D-2265). Three formulations were made in the study consisting of formulation 1 (F1), formulation 2 (F2) and formulation 3 (F3) with 0 %, 1 % and 2 % of additive added into the formulation, respectively. The results show that the F1 consistency of the grease is in the range of the grease formulated. Graphene significantly contributed to the performance of food-grade grease formulations with higher mechanical stability, but the dropping point was lower than expected. This formulation grease might improve based on another method consisting of other components or materials.

1. Introduction

Food-grade lubricant grease is ideal for use in factories, applications, and machinery to process meat, poultry, and other types of food where there are concerns about grease contamination towards the products. These food-grade designations are classified based on the risk of contact with food [1], [2].

Three components are often found in grease: base oil, thickener and additive as depicted in Fig. 1. The grease's performance is improved by adding small amounts of additives, which can also improve the base oil's properties. The thickener separates the grease to keep the grease in a semi-solid state. Sometimes, lubricants like soft solids or highly viscous liquids are called grease [3].

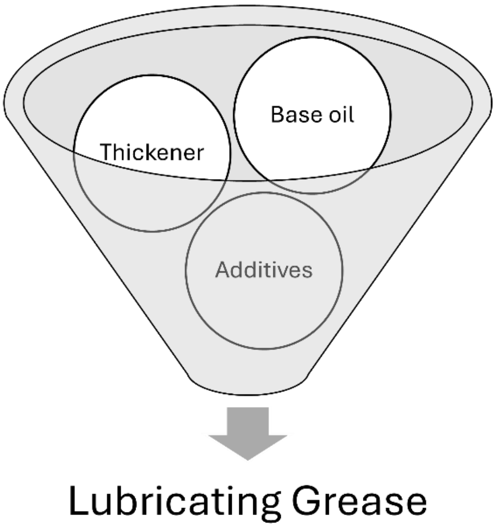

Fig. 1: General components of lubricating grease

Typically, the effectiveness of the additives has a significant impact on the lubricity of the base grease. Graphene has garnered substantial interest in various applications because of its remarkable mechanical, electrical, and thermal properties. Graphene is recognised as a nanomaterial and identified as an allotropic type of carbon made up of a monoatomic layer of carbon atoms arranged in a two-dimensional plane in a honeycomb pattern, with a 0.142 nm spacing between each atom [4], [5]. Graphene's exceptional properties make it appropriate for various technical uses, including flexible composite material, solar cells, sensors and high-frequency electronics [6]. Nanomaterial could be embedded into lubricant as an additive due to the inherent properties of the materials [7]–[9]. This work uses various characterisation techniques to examine the effects of adding carbon nanoparticles (CNPs), specifically graphene, into grease formulation.

2. Materials and Methods

2.1 Materials

The formulation utilised vegetable oil for base oil obtained from a local supermarket. Industrial-grade aluminium isopropoxide and 12-hydroxystearic acid (imported from China; unbranded) were used as a thickening agent. Graphene flake in the suspended form obtained from Universiti Malaysia Pahang Al-Sultan Abdullah (UMPSA) was used as an additive.

Prior to performing the formulation, the base oil (vegetable oil) was analysed to determine the viscosity. A viscometer (Brookfield DV-II+Pro) was used to measure the viscosity of the cooking oil samples, and the measurement was conducted at room temperature. The result shows that the viscosity of the base oil is 0.25 cP.

2.2 Grease Formulation and Preparation

The formulation of the grease is based on the range percentage of each component of aluminium (thickeners), vegetable oil (base oil) and graphene (additive). The formulation was conducted as shown in Table 1.

Table 1 *Grease formulation and composition*

Formulation	Composition (w/w)
F1	75 % base oil
	25 % thickener
	0 % without additive
F2	75 % base oil
	24 % thickener
	1 % additive
F3	75 % base oil

23 % thickener
2 % additive

There are three formulations of the grease and the composition, as shown in Table 1. Base oil and thickener are weighed based on w/w percentage in this formulation. The formulation started with measuring the appropriate weight or volume of the ingredients. Next, a weighted aluminium isopropoxide was dissolved in water to create an aqueous solution and was added to heated 12-hydroxystearic acid for a saponification reaction in a steel mixing vessel. An overhead stirrer is used to mix these components. The thickener was mixed until no water was present, and a pre-determined weight of vegetable oil was slowly added to the mixture as a base oil. Finally, graphene was added and stirred continuously until the solution was well mixed.

2.3 Grease Analysis

2.3.1 Penetration Test (ASTM D-217)

The ASTM D217 Standard Test Methods for Cone Penetration of Lubricating Grease are used to quantify grease [10]. The penetration test utilised two conditions for a particular grease. Each grease was subjected to mechanical stress using a grease worker machine (60 strokes or P60). The changes in grease hardness were compared by comparing the penetration result of grease without mechanical stress (unworked grease; P0) and grease subjected to mechanical stress (worked grease). The P0 (unworked) and P60 (worked) penetration test results were compared to determine the mechanical stability of the grease. Fig. 2 shows the equipment used for the analysis.

The penetration value is reported in mm$^{-1,}$ determining the depth of the penetration cone that penetrated the grease. The results were then compared to the National Lubricant Grease Institute, or NLGI classification. NLGI classify grease into nine different consistency grades, from fluid (NLGI 000) to semi-fluid (NLGI 6).

(a) **(b)**

Fig. 2: *Equipment used for penetration test comprising (a) Grease penetrometer; (b) Grease worker*

2.3.2 Dropping point test (ASTM D-2265)

The grease-dropping point was determined according to the ASTM D-2265 method. The apparatus used consisted of a sample cup with a hole at the bottom, a test tube and a heating block equipped with a temperature-measuring device and thermometer. The temperature in the sample cup is measured with a sample thermometer inserted inside the tube to avoid contact with the grease sample.

The dropping point temperature measurement was taken when the first drop of the grease was seen out of the sample cup. To the closest degree, the reading on the sample thermometer is noted. The aluminium block oven's temperature is likewise measured in real-time and to the nearest degree. Finally, one-third of the difference between the two values is the correction factor, added to the estimated value and noted as the grease-dropping point.

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3. Results and Discussion

The grease formulation is based on the percentage of thickening agent, base oil, and additives. The grease analysis being investigated is the penetration test (ASTM D217) and dropping point test (ASTM Method D-2265).

Table 2 shows the properties of the formulated greases comprising penetration test (unworked and worked) and dropping point. The penetration test is used to analyse the grease's hardness (consistency) and determine the grease's NLGI grade. The NLGI grade was established by the National Lubricant Grease Institute (NLGI) to evaluate and classify a grease's stiffness based on the depth of the cone penetrating the grease. The grade comprised a scale ranging from 000,00, 0, 1, 2, 3, 4, 5, and 6 (scale 000 indicates very soft grease while 6 indicates stiff grease) was used to classify the grease consistency accordingly for any application.

Table 2 *Properties of grease formulation*

Grease	Penetration Test (dmm)		Dropping point test (^{0}C)
	Unworked (P0)	Worked (P60)	
F1	161.5 (NLGI 4-5)	252.2 (NLGI 2-3)	<100
F2	165.9 (NLGI 4-5)	272.3 (NLGI 2)	<100
F3	322.7 (NLGI 1)	350.8 (NLGI 0-1)	<70

A penetration test was conducted to analyse the consistency and mechanical stability of the two greases, as shown in Table 2. The penetration value of F1, which is without additives, was 252.2 mm^{-1} for P60, while P0 was 161.5 mm^{-1}. The result for F2 with additives was 272.3 mm^{-1} for P60, while P0 was 165.9 mm^{-1}. The NLGI grade in this scale was defined as about 4 to 5 for unworked and about 2 for worked, a medium-soft grease. In the case of F1 and F2, it was a mixture of aluminium isopropoxide and 12-hydroxystearic acid. The change in the grease consistency could be observed by the shift in penetration value of the grease before and after mechanical stress was applied. This also could indicate grease degradation if the value of penetration changes for worked grease as compared to unworked grease [11].

The dropping point of all the greases was tested using the dropping point apparatus. However, the results show that all the greases have very low dropping point value, which is in the range of 70 to 100 °C. This could be attributed to the incompatibility of the base oil and the thickener, which is supposed to hold the oil. Furthermore, the incompatibility of these two components would hinder the complete reaction of the formulated grease and adversely affect the thermal properties of the grease [12], [13].

Fig. 3 shows the formulated greases appearance with different formulation. The structure and the appearance of the grease are slightly different due to the addition of the additive. F1 comprises 75% base oil and 25% thickeners without graphene. It has a buttery hard texture, white-light colour, oily, and stiff structure. F2 comprises of 75% base oil, 24% thickeners and 1% graphene. It has a buttery texture, a slightly dark colour, and a sticky structure. It was observed that grease without additives has a lighter-coloured and stiffer structure than grease with additives. The grease structure looks buttery when the graphene is included in the structure.

(a) (b) (c)

Fig. 3 *Structure of grease for (a) Lubricant grease without additive; (b) Lubricant grease with 1% additive; (c) Lubricant grease with 2% additive*

The analysis shows that graphene does not significantly influence the grease properties, as indicated by the tests. During the experiment, the thickener used, which is aluminium isopropoxide that is dissolved using distilled water, did not dissolve properly, the volume temperature used was uncontrolled, and there was an excessively long heating time for the grease. It may be the reason for the damage caused by grease.

4. Conclusion

In this work, the effect of graphene used as an additive in standard lubricant grease has been investigated. The aim is to compare the formulation of grease, which has the addition of additives and without additives, which is graphene. It shows the effect, which is slightly different in its appearance in terms of colour and texture. Hence, from all the formulation grease analysis test results, it does not exactly achieve the standard lubricant grease for the food industry used. To improve the performance of the formulation grease until it becomes the industry standard lubricant grease, we will conduct a temperature volume, mix the grease consistently over a set period of time, and use a homogeniser to improve homogeneous grease that may contain new or different types of components.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Syubaikah Badrul, Siti Maisurah Maudoode, Mohd Khairul Nizam Mohd Zuhan, Najmuddin Mohd Ramli, Mohd Najib Razali;* ***data collection:*** *Syubaikah Badrul, Siti Maisurah Maudoode, Mohd Khairul Nizam Mohd Zuhan, Najmuddin Mohd Ramli, Mohd Najib Razali;* ***analysis and interpretation of results:*** *Syubaikah Badrul, Siti Maisurah Maudoode, Mohd Khairul Nizam Mohd Zuhan, Najmuddin Mohd Ramli, Mohd Najib Razali;* ***draft manuscript preparation:*** *Syubaikah Badrul, Siti Maisurah Maudoode, Mohd Khairul Nizam Mohd Zuhan, Najmuddin Mohd Ramli, Mohd Najib Razali. All authors reviewed the results and approved the final version of the manuscript.*

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