FORMULATING LUBRICATING GREASE FROM CASTOR BEAN

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ABSTRACT

Lubricating grease is formulated based on certain composition of components of materials. The components are the base oil, thickener and additives. To be used as lubricant grease, it has to be tested to be sure that it is suitable to be grade as lubricant grease. This formulation of lubricant grease is generally called a bio-grease because of the raw material used to make the lubricant grease are from natural resources. To formulate this grease, it has to be certain with the composition of the components. The behavior of the grease is depends on the materials' type and its amount of composition in use. Thus, certain analysis is done towards this formulation of lubricant grease to discover or determine its characteristic. The analysis or characterizations are the penetration, dropping point test, kinematic viscosity, and density analysis. The result shows that this formulation is NLGI 0 which is soft in texture. While dropping point test within 2 hours shows that the melting point for this formulation is up to 220.67 °C.

ABSTRAK

Gris pelincir dirumuskan berdasarkan komposisi komponen bahan-bahan tertentu. Komponen-komponennya adalah minyak sebagai asas, pemekat dan aditif. Untuk digunakan sebagai minyak pelincir, ia perlu diuji untuk memastikan bahawa ia sesuai dengan gred dan sifat-sifat sebagai minyak pelincir. Formulasi gris pelincir ini secara umumnya dipanggil bio-gris kerana bahan mentah yang digunakan untuk membuat minyak pelincir adalah daripada sumber asli. Untuk menghasilkan rumusan gris, ia mempunyai peratus komposisi komponen yang sesuai. Sifat sesuatu gris adalah bergantung kepada jenis dan bahan-bahan serta jumlah komposisi bahan yang digunakan. Oleh itu, analisis tertentu haruslah dilakukan terhadap gris untuk menentukan sifat gris ini. Analisis yang dilakukan adalah penembusan kon terhadap gris, ujian penetuan takat suhu untuk gris mencair, kelikatan kinematik gris, dan analisis ketumpatan gris. Hasil kajian menunjukkan bahawa rumusan ini adalah pada gred NLGI 0 yang mana menunjukan tekstur yang lembut. Melalui ujian penentuan gris mencair, didapati bahawa suhu yang diperlukan untuk gris ini mencair adalah pada 220.67 °C.

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LIST OF SYMBOLS

ν	Kinematic Viscosity
η	Dynamic Viscosity
ρ	Density
%	Percentage
°C	Degree Celcius
τ	Torque
°F	Degree Fahrenheit
±	Plus minus

LIST OF ABBBREVIATIONS

cm^3	Centimeter Cubic
NLGI	National Lubricant Grade International
cSt	Centi Stoke
cP	Centi Poise
ppm	Part per million
rpm	Revolution per minute
CMC	Carboxyl Methyl Cellulose
g	Gram
mm	Millimetre
ml	Millilitre
mPa·s	Mega Pascal Second
mm^2/s	Millimetre Square per Second
m^2/s	Meter Square per Second
Pa·s	Pascal Second
g/cm^3	Gram per Centimetre Cubic

CHAPTER 1

INTRODUCTION

1.1 Background of Study

This work is focused on the formulating a lubricant grease, which has high potential on biodegradable lubricant grease mixed with based formula, the thickener, and additives. In general terms lubricating grease may be defined as a liquid lubricant thickened to form a solid or semi-fluid product by means of a thickening agent. The lubricant industry's toughest challenges are performance; environmental compatibility and cost concern for the environment. It has been shown by the continuous developments and research on synthetic lubricants to minimize the way our daily activities impart on the environment. Most lubricants are based on combinations of mineral oils, with the additions of synthetic fluids as appropriate for higher levels of performance.

The main difference between lubricating greases and other lubricants is their characteristic rheological behavior, frequently referred as consistency in the lubricant industry, which results from the addition of the thickener (R. Sánchez et al., 2011). Thus, the thickener used will affect and improve the lubricant grease behavior and performance. There are issues that that have to be concerned while formulating the lubricant grease; biodegradable lubricants, good solubility, resist oxidation and have good temperature viscosity characteristics. The choice made will depend upon the lubricant application, balancing cost constraints against technical and environmental targets, particularly emphasizing such features as biodegradability and recyclability.

1.2 Problem Statement

The lubricant grease that already exists in the market is high cost as the best common thickener used is lithium, for instance. The specialty of lubricating grease is needed in various fields such as ball and roller bearing and it has high demand in industry. The endurance for this special lubricating grease is at high temperature even without using the compound of lithium. In the contrast, our research will adopt new base material which easily to get from castor bean in producing this special lubricant grease which bear to extend the high temperature.

1.3 Research Objective

The objectives are based on the production cost and the eco-friendly product. The objectives as followed:

1. To make a formulation of special lubricant grease
2. To produce low cost special lubricant grease
3. To produce biodegradable lubricating grease

1.4 Scope of Study

. Based on the problems stated above, the lubricating grease which was released in the market is in expensive price as the components to formulate it are expensive to formulate since it is very necessary in industry. Thus, the objective obtained must consider the production cost which is for the component used and to signalize on the environmental issue. By formulating the kernel as base formulation and the methyl cellulose as the thickener agent; we will expect to experience the eco-friendly lubricating grease at an alternative material to reduce negative effects on the environment and at significant price or value.

1.5 Rational and Significance

Due to the general tendency to promote the replacement of non-renewable raw materials with the renewable sources, lots of research had been done in a few years. In this study, we are put a thought in using the easy way to get these component or material to formulate the special lubricating grease. Following this, the minimization of the environmental impact caused by industrial waste materials is one other factor that promotes the increasing interest in the use of natural components in a wide variety of manufactured products.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

The important formulation for the special lubricating grease is the lubricant as the base and the gelling agent or thickener to improve the properties of the lubricant grease. The improvement is kept on track by the researches all over the world to produce quality products especially on mechanical devices such ball bearing. The tested had been done to ensure and study the characteristic of each lubricant grease produce. (R. Sánchez et al., 2011) says certain biodegradable greases consist of vegetable oil, synthetic esters or glycols as base fluids; metal soaps or polyureas as thickeners, and additives to improve the behavior of the final material in specific conditions. Grease is oil suspended in a base or carrier, and when these bases are exposed to moisture or heat, they can turn into soap or carbon ash. Therefore, it may be necessary to use synthetic additives to prevent deterioration of the base. There are factors to consider certain greases to use include hardness (consistency), stability (ability to retain consistency) and water resistance (emulsification).

2.2 Material formulation

2.2.1 Lubricant / base formulation

The safe lubricant plays very important medium or compound to formulate the lubricant grease formulation. The motivation to produce eco-friendly lubricants which presenting good quality of lubricant is basically from vegetable oils. It is biodegradable indeed. (R. Sánchez et al., 2008) reported, a traditionally a mineral or synthetic oil could be replaced by a vegetable one. In common research nowadays, (R. Sánchez et al., 2011) in his journal mention that castor oil being the material used as it is quite reasonable to get. The castor seed itself contain 50% of oil and it has two parts; kernel and the shell. Kernel reaches around 70-75% of the whole seed weight. The main ingredients of kernel are oil and protein (MBI Green Farm Corp., n.d).

Other than castor oil, soybean oil is also being use as base oil to formulate the grease. (R. Sánchez et al., 2011) shows the comparison data for castor oil and soybean oil. The lack of the lubricant properties could be recovered with addition with the additives or thickener. In Ponnekanti Nagendramma et al,. (2011), there are a lot of different vegetable oils, but not all can be used for high performance lubricants. There are technical, logistic and cost barriers that might be considered. It is very difficult to anticipate that up to what extent the mineral oils will be replaced by the vegetable oils (% world consumption).

2.2.2 Thickener agent

In general formulation lubricant grease exists, there is still includes highly non-biodegradable components, particularly the thickener agents. Thus, there is an effort to find the replacement which is the biodegradable one. Thickener or gelling agents traditionally used was most commonly composition such metallic soap, phyllosilicates, silica, and betonite particles or polyurea derivatives as stated in (R. Sánchez et al., 2011).

In his research, latest thickener agent used was biopolymers which are ethyl cellulose which was being compared to the α-cellulose and methyl cellulose compounds (degree of substitution). The cellulosic derivatives is used for better satisfactory in mechanical and physical stabilities especially in formulating biolubricating greases with castor oil and blends of ethyl and methylcellulose (N. Nú˜nez et al., (2011).

The other thickener from natural resources used in research was chitin, chitosan (which is the 50% of deaceltylation of chitin in aqueous acidic media) and acylated chitosan (R. Sánchez et al., 2011). All these types of thickeners or gelling agents have their own specialization for manipulating the behaviour of the lubricant grease.

2.2.3 Additives

An additive is added as the coating material to the carrier (base). This additive brings up the characteristic as oxidation stabilizer for the lubricating grease. **Table 2.1** shows several common lubricant additives. Additive works as anti-friction for bearings that must be lubricated to prevent metal-to-metal contact between the rolling elements, raceways and retainers.

A good lubricated bearing has the best chance of reaching its maximum service life. In-Sik Rhe (n.d) mentioned that additives behave excellent in water and good storage stability, good shear and oxidation stability, good antiwear and load carrying capacity, and saltwater corrosion properties.

High index of antioxidant work to protect bearing (resistance to oxidation) from being corrode. In Matco Services Incorporation (n.d), water absorption and moisture penetration into a lubricated chamber may lead to a corrosive condition on steel components, via moisture diffusion through the lubricating thin surface film layer. The use of corrosion inhibitors (additives) in grease may be of substantial benefit. **Figure 2.1** below show the effect of corrosion in ball bearing.

Table 2.1: Common Lubricant Additives (Naval Ships' Technical Manual)

Additive Type	Compounds Used	Function
Extreme Pressure (EP)	Sulfur-Phosphorous compounds	Increase load carrying ability
Antioxidant	Hindered phenols Amines	Prolong oil life by slowing oxidation
Antifoam	Silicones	Control foaming in forcefeed systems
Anticorrosion	Zinc dithiophosphates (ZDDP)	Form anticorrosion film
Detergent or Dispersant	Calcium sulfonates	Prevent deposition of carbon
Alkalinity (TBN)	Calcium Carbonates, Magnesium, Sulfonates	Neutralize acidic engines byproducts
Antiwear	Zinc dithiophosphates (ZDDP)	Reduce wear
Pour Point Depressant	Methacrylate polymers	Lower pour point
VI Improvers	Organix polymers	Improve VI

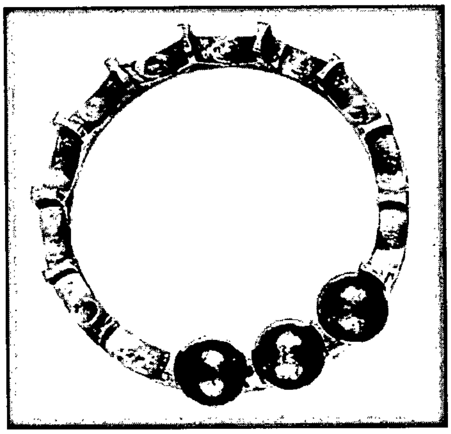

Figure 2.1: The effect of corrosion in ball bearing (E. F. Jones., 1968)

2.3 Manufacture of lubricating grease formulation

Using the same methodology in (N. Nú˜nez et al., 2011), which is to do a methylation of cellulose pulp from α-cellulose by Sigma-Aldrich. This could be the reference. The methodology to make a formulation of lubricating grease is shown by N. Nú˜nez et al., (2011) using 12-hydroxystearic acid, lithium hydroxide and naphthenic oil (it is then was replaced by castor oil as modification).

2.4 Properties or Characterization of Lubricant Grease

The test that had been done by R. Sa´nchez et al., (2011), is rheological characterization and penetration and mechanical stability tests. Rheological tests itself is the determining of rheological characterization of the formulation which is the improvement of viscosity of the thickener used. The formulations of lubricating grease's rheological characterization is studied by carrying it out by controlled-stress and controlled strain rheometers using plate-plate geometries comprises between 25 °C to 125 °C (N. Nú˜nez et al., 2011).

In rheological characterization tests in Nú˜nez et al., (2011), the experimental results seem to proof that cellulose pulp acts as the main thickener agent whereas ethyl cellulose basically modifies the rheology of the oil medium thus increasing the physical stability of dispersions. Which, this physical stability improvement does not only due to the increasing of base viscosity but also to the viscoelastic properties (depend on frequency of lower thickener concentration) of base/modified thickener agent. The mechanical stability of the formulations is simulated using the standardized roll-stability test. The stability of lubricating grease are considered to be stable to the continuous shear of rolling elements when its variation of penetration before and after performing the roll-stability test is closed to zero.

Extra with that, Nú˜nez et al., (2011) stated that other tests that had been done are thermogravimetric analysis (TGA) which is to indicates the behaviour of the comparisons of the samples, fourier transform infrared spectroscopy (FTIR) to evaluate the chemical modification of α-cellulose pulp after the methylation reaction, and morphological characterization's observations using atomic force miscroscopy (AFM) that shows the morphology of cellulose pulp is highly dependent on the methylation process.

In In-Sik Rhee (n.d), biodegradation test is one of the test which to determine the degree of aerobic aquatic biodegradation of lubricants on exposure to an inoculums under laboratory conditions. This biodegradability will test of a lubricant that is expressed as the percentage of maximum (theoretical) carbon conversion (or carbon dioxide generation) under well-controlled conditions for a period of 28 days.

2.4.1 Rigidity or Consistency (Penetration)

Greases are classified according to a hardness scale developed by the National Lubricating Grease Institute (NLGI). According to this system, softer greases are assigned a low NLGI number, and stiffer greases a high NLGI number. The penetration numbers refer to the depth, in tenths of millimeters, that a weighted cone penetrates the grease.

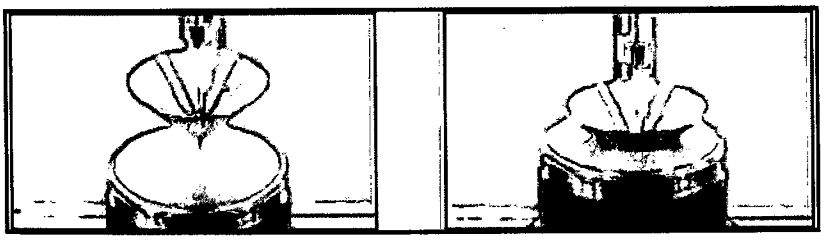

Figure 2.2: Penetrometer Cone Released In Grease

Figure 2.2 above shows the cone penetration upon a grease to figure out it rigidity and consistency. Numbers are the most important feature of grease is its rigidity and consistency. Consistency is defined as the degree to which a plastic material resists deformation under the application of force where greases consistency is easily understand by its capability or resistance to deformation by an applied force. Grease that is too fluid may leak out, while grease that is too stiff may not feed into areas requiring lubrication. Lubricant grease consistency depends on the type and amount of thickener used and the viscosity of the base oil.

In the case of lubricating greases, this is a measure of the relative hardness or softness and has some relation to flow and dispensing properties. The measure of consistency is called penetration. Penetration depends on whether the consistency has been altered by handling or working. To measure penetration, a cone of given weight is allowed to sink into a grease for 5 seconds at a standard temperature of 25 ° C. The depth, in tenth of a millimetre, to which the cone sinks into the grease, is the penetration. A penetration of 100 would represent solid grease while one of 450 would be semi fluid.

The NLGI has established consistency numbers or grade numbers, ranging from 000 to 6, corresponding to specify along with a description of the consistency of each classification. Consistency is measured by ASTM D 217 and D 1403 methods which measure penetration of unworked and worked greases. Cone Penetration of Lubricating Grease and is often reported in terms of NLGI grade (Exxon Mobil Corporation, 2009).

Such in **Figure 2.1**, cone penetration using ASTM D217 requires a large volume of sample and is not normally performed on used grease samples. A more modern alternative to cone penetration, for estimating changes in the consistency of used greases is Thermal Gravimetric Analysis (TGA). TGA analysis measures the mass of a substance in relationship to temperature and is used to determine the loss of material with increasing temperature. **Table 2.2** below shows the definition of NLGI grade from 000 to 6 ranges.

Table 2.2: Definition of NLGI Grade (Sukirno et. al,.)

NLGI Consistency Grade	Penetration Range (1/10mm)	Description
000	445 – 475	Very Soft
00	400 – 430	Very Soft
0	355 – 385	Soft
1	310 – 340	Creamy
2	265 – 295	Almost Solid
3	220 – 250	Hard
4	175 – 205	Hard
5	130 – 160	Very Hard, as soap
6	85 – 115	Very Hard, as soap

2.4.2 Dropping Point

Greases exist in an essentially semi-solid form. The temperature at which grease changes from a semi-solid to a liquid is termed its dropping point. Dropping point provides some indication of the high temperature characteristics of grease. ASTM D2265 (preferred over the older and less precise ASTM D566) is the standard method used to determine the dropping point of grease.

The drop point also understand as the temperature at which the first drop of the melting substance to be examined falls from a cup under defined conditions. The drop point for the grease is the temperature at which the thickener loses its ability to maintain the base oil within the thickener matrix. This may be due to the thickener melting or the oil becoming so thin that the surface tension and capillary action become insufficient to hold the oil within the thickener matrix.

The dropping point is one of the determinations that characterise the grease's thermal stability. However it is not an accurate prediction of the grease's upper operating temperature limit which is a function of many variables such as base oil oxidation stability, additive degradation, thickener shearing, oil separation and so forth. A high dropping point, while not a predictor of upper operating temperature, is an indicator of the maximum peak temperature that the grease may be subjected to for a short duration while not releasing oil excessively and therefore drastically reducing the life of the grease and potentially damaging the application in the long run (Exxon Mobil Corporation, 2009).

2.4.3 Viscosity

The most important physical property of a lubricant is its viscosity. The viscosity is a fluid's resistance to flow (shear stress) at a given temperature. In certain cases, viscosity is misinterpreted to as thickness (or weight). Viscosity is not a dimensional measurement, so calling highly viscous oil thick and less viscous oil thin is disorientate. Likewise, reporting viscosity for trending purposes without a reference to temperature is no sense. The temperature must be defined to interpret the viscosity reading. Typically, viscosity is reported at 40°C and/or 100°C or both if the viscosity index is required. (Machinery Lubrication, 3/2002)

Viscosity, which may be defined as a fluid's resistance to flow, is the characteristic most frequently stipulated by equipment manufacturers when making lubricant recommendations. The selection of proper lubricant viscosity is often a compromise between selecting one high enough to prevent metal to metal (wear) contact, and one low enough to allow sufficient heat dissipation. Kinematic viscosity in centistokes is obtained by measuring the time required for a specified volume of fluid to flow through a calibrated capillary tube at a specified temperature.

2.4.3.1 Kinematic Viscosity

This test method covers the determination of the Kinematic viscosity of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. This is known as an ASTM International Standard Test Method for the analysis of viscosity in used and new lubricants which ASTM D445 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity (ASTM International, May 2011). This test method is actually for a base oil viscosity but also valid for grease viscosity. Yet, the dominant procedure for performing kinematic viscosity measurements is ASTM D445, often modified in the used oil analysis lab to save time and make the test measurement more efficient.

Of all the tests employed for used oil analysis, none provides better test repeatability or consistency than viscosity. Despite that, there is no property more critical to effective component lubrication than base oil viscosity. (Machinery Lubricant, n.d). Drew Troyer in his writing said that most used oil analysis laboratories measure and reports the kinematic viscosity, yet by contrast, most onsite viscometers measure absolute viscosity, but are typically programmed to estimate and report kinematic viscosity.

The kinematic viscosity in cSt at 40°C is the basis for the ISO 3448 kinematic viscosity grading system, making it the international standard. The other common kinematic viscosity systems such as the SAE grading system and Saybolt Universal Seconds (SUS) can be related to the measurement of the viscosity in cSt at either 40°C or 100°C (Drew Toyer, n.d). Kinematic viscosity is traditionally measured by noting the time it takes oil to travel through the orifice of a capillary under the force of gravity. The orifice of the kinematic viscometer tube produces a fixed resistance to flow in different sized capillaries. The time taken for the fluid to flow through the capillary tube can be converted directly to a kinematic viscosity using a simple calibration constant provided for each tube.

2.4.3.2 Relationship between Dynamic Viscosity, cP and Kinematic Viscosity, cSt

The viscosity of a fluid is its resistance to shear or flow, and is a measure of the fluids adhesive/cohesive or frictional properties. This arises because of the internal molecular friction within the fluid producing the frictional drag effect. There are two related measures of fluid viscosity which are known as dynamic and kinematic viscosity.

Dynamic viscosity is also termed absolute viscosity and is the tangential force per unit area required to move one horizontal plane with respect to the other at unit velocity when maintained a unit distance apart by the fluid. The dynamic viscosity is the viscosity that relates shear stress τ and shear rate du/dz in a fluid. The viscous shear stress τ is proportional to the shear rate, the dynamic viscosity η being the proportionality factor. So, thicker oils have a higher viscosity value causing relatively higher shear stresses at the same shear rate.

Dynamic viscosities are usually measured under high shear conditions, for example, the cone on plate or cylinder viscometer in which the viscous shear torque is measured between two cylinders. With the viscosity known at two reference temperatures the viscosity can be calculated for intermediate temperatures with specific interpolation functions those from Reynolds or Vogel & Cameron.

The kinematic viscosity, v is the quotient of the dynamic viscosity η and the fluid density ρ, $\boldsymbol{\nu} = \eta/\rho$. The physical principle of measurement is based on the rate at which a fluid flows under gravity through a capillary tube. With the viscosity known at two reference temperatures the viscosity can be calculated for intermediate temperatures using the interpolation function of Ubbelohde-Walther, which is adopted by ASTM D341.

2.4.4 Fourier Transform Infrared Spectroscopy, FTIR

Contamination from water or other grease types can be identified by Fourier Transform Infrared Spectroscopy (FTIR) Analysis. FTIR can also measure gelling agent type and concentration, along with oxidation by-products. If cross-contamination with different types of grease is suspected, it may also be feasible to perform elemental analysis (after acid digestion) to look for common metals present in the gelling agent.

For example, grease that is supposed to be aluminum complex grease, but has become contaminated with calcium sulfonate complex grease will show both aluminum and calcium in spectroscopic analysis, indicating a contamination problem. FTIR can identify the presence of water. However, it is not sensitive to low levels. Water in the parts-per-million (ppm) range can be measured using a variation of ASTM D6304 (Machinery Lubrication, 3/2002).

2.5 Lubricating Grease Uses Or Application

2.5.1 Corrosion Protection

Corrosion prevention of a system is a second role provided by a lubricant. In environments where contamination of the system with water is likely, protection of machinery components from corrosion is of the utmost importance. Salt water is considerably more corrosive than fresh water, so machinery must be well protected from this contaminant. Water molecules may also diffuse through the lubricant and enter surface micro-cracks, causing hydrogen embrittlement and subsequent surface failure.

It is thus important that water contamination of machinery systems be minimized. To achieve corrosion protection, lubricants must form a protective barrier on machinery surfaces. Modern-day lubricants often contain corrosion inhibitors which chemically bond to the metallic surfaces of equipment components. Additive is an example of a class compound known as corrosion inhibitors.

2.5.2 Reduce Wall Friction

When dry surfaces move relative to one another, asperities may rub, lock together, and break apart. The resistance generated when these adjacent surfaces come in contact is called friction. The welding together and breaking apart of asperities is a form of adhesive wear. Another form of wear may occur when a hard contaminant particle becomes trapped