STUDYING THE EFFECT ON THE PROPERTIES OF FUME SILICA PERCENT FORMULATED GREASE

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I declare that this thesis entitled "Studying the effect on the properties of fume silica percent formulated grease" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree."

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Date	: 30 April 2009

Special Dedication to lovely parent, my family members,
my friends, my fellow colleague
and all faculty members

For all your care, support and believe in me.

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ABSTRACT

Vegetable oil has significant potential as a base fluid and substitute for mineral oil for grease formulation. Vegetable oils are promising candidates as base fluid for eco-friendly lubricants because of their excellent lubricity, biodegradability, viscosity-temperature characteristic and low volatility. This paper presents the effective and ability of grease by using natural material. Performance characteristics of greases used for industrial and automotive application are largely depending on the hardness. Grease hardness was determined using standard test methods. Results indicate that the fume silica percent in formulated grease are directly proportional to the viscosity and drop point. There is no drop point at 310°C occur more than 10%wt of fume silica. Also, the grease didn't show negative effect on the metal surface. Therefore, developed greases can be a good substitute for mineral oil-based greases in industrial, agriculture, forestry and marine application.

ABSTRAK

Minyak sayuran mempunyai potensi untuk menggantikan mineral galian untuk pembentukan gris. Minyak sayuran menjanjikan pengguna sebagai pelincir yang mesra alam kerana tahap pelinciran yang tinggi, biodegradasi, tahap kualiti kelikatan-suhu dan sukar meruap. Kajian ini membentangkan keberkesanan and kebolehan gris dengan menggunakan bahan semula jadi. Kebiasaanyer, gris yang digunakan didalam industri dan bidang automotif adalah bergantung kepada kekuatan gris trsebut. Kekuatan gris boleh ditentukan dengan kaedah ujian piawai. Keputusan menunjukkan, peratus “fume silica” didalam pengahasilan gris adalah berkadar langsung dengan kelikatan dan tahap titisan gris tersebut. Tiada tahap titisan pada suhu 310°C berlaku yang mengandungi 10% berat bersih “fume silica”. Oleh itu, kemajuan gris ini adalah sangat bersesuain bagi menggantikan minyak galian gris didalam industri, pertanian, penghutanan dan marin.

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CHAPTER 1

INTRODUCTION

1.1 Background of study

The search for environmentally friendly materials that have the potential to substitute for mineral oil in various industrial applications is currently being considered a top priority in the fuel and energy sector. This emphasis is largely due to the rapid depletion of world fossil fuel reserves and increasing concern for environmental pollution from excessive mineral oil use and its disposal. Renewable resources such as seed oils and their derivatives are being considered as potential replacement for mineral oil base stock in certain lubricant application where immediate contact with the environment is anticipated. The nontoxic and readily biodegradable characteristics of vegetable oil-based lubricant pose less danger to soil, water, flora and fauna in the case of accidental spillage or during disposal. (Atanu Adhvaryu *et al.*, 2004)

1.1.1 Lubricating grease

Lubricating greases are semisolid colloid dispersions of a thickening agent in a liquid lubricant matrix. They owe their consistency to a gel forming network where the thickening agent is dispersed in the lubricating base fluid. Grease may include various chemical additives for specific property enhancement. A typical grease composition contains 60-95% base fluid (mineral, synthetic, or vegetable oil), 5-25% thickener (fatty acid soaps of alkali or alkaline metals), and 0-10% additives (antioxidants, corrosion inhibitors, anti-wear/ extreme pressure, antifoam, tackiness agents, etc.). The base fluid imparts lubricating properties to the grease, whereas the thickener, essentially the gelling agent, holds the matrix together. (Atanu Adhvaryu *et al*., 2004)

Grease is the preferred form of lubrication in hard-to-reach places in a mechanically rubbing or dynamic systems. Grease acts as reservoir for lubricant-based fluids and additive molecules. Much of its functional properties are dependent on their ability to flow under force, have shear stability, resist viscosity changes with temperature and pressure, water stable, seal out contaminants, decrease dripping and spattering, etc. The dependability of lubricating grease depends on their physical properties that are structurally related, which is obtained by the proper selection of ingredients and processing. Thus, it is pertinent to understand the grease microstructure as it contributes significantly to various functional properties of grease. (A. Adhvaryu *et el*., 2003)

1.1.2 Function

The function of grease is to remain in contact with and lubricate moving surfaces without leaking out under the force of gravity, centrifugal action or being squeezed out under pressure. Its major practical requirement is that it retains its properties under shear forces at all temperatures it experiences during use. The others function of greases are keep moving parts apart, reduce friction, transfer heat, carry away contaminants & debris, protect against wear and prevent corrosion.

1.1.2.1 Applications Suitable for Grease

Grease and oil are not interchangeable. Grease is used when it is not practical or convenient to use oil. The lubricant choice for a specific application is determined by matching the machinery design and operating conditions with desired lubricant characteristics (Jeremy Wright, 2008). Grease is generally used for machinery that runs intermittently or in storage for an extended period of time. Because grease remains in place, a lubricating film can instantly form. Machinery that is not easily accessible for frequent lubrication. High-quality greases can lubricate isolated or relatively inaccessible components for extended periods of time without frequent replenishing. These greases are also used in sealed-for-life applications such as some electrical motors and gearboxes. The other application is in machinery operating under extreme conditions such as high temperatures and pressures, shock loads or slow speed under heavy load because of grease maintains thicker films in clearances enlarged by wear and can extend the life of damaged parts that were previously lubricated by oil.

1.1.2.2 Functional Properties of Grease

Grease functions as a sealant to minimize leakage and to keep out contaminants. Because of its consistency, grease acts as a sealant to prevent lubricant leakage and also to prevent entrance of corrosive contaminants and foreign materials. It also acts to keep deteriorated seals effective.

Grease is easier to contain than oil. Oil lubrication can require an expensive system of circulating equipment and complex retention devices. In comparison, grease, by virtue of its rigidity, is easily confined with simplified, less costly retention devices.

Grease holds solid lubricants in suspension. Finely ground solid lubricants, such as molybdenum disulfide (moly) and graphite, are mixed with grease in high-temperature service or in extreme high-pressure applications. Grease holds solids in suspension while solids will settle out of oils.

1.1.3 Grease formulation

The American Society for Testing and Materials (ASTM) defines lubricating grease as: "A solid to semi fluid product of dispersion of a thickening agent in liquid lubricant. Other ingredients imparting special properties may be included" (ASTM D 288, Standard Definitions of Terms Relating to Petroleum). (V. V. Sinitsyn and A. B. Kofman ,2004)

As this definition indicates, there are three components that form lubricating grease. These components are oil, thickener and additives. The base oil and additive package are the major components in grease formulations, and as such, exert considerable influence on the behavior of the grease. The thickener is often referred to as a sponge that holds the lubricant (base oil plus additives).

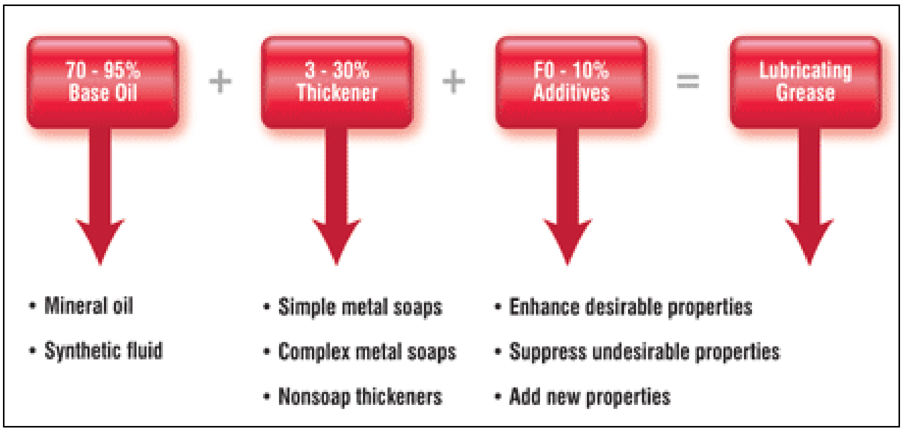

Figure1.0: Formulated grease

1.1.3.1 Base Oil

Most grease produced today use mineral oil as their fluid components. These mineral oil-based greases typically provide satisfactory performance in most industrial applications. In temperature extremes (low or high), a grease that utilizes a synthetic base oil will provide better stability. (Jeremy Wright, 2008)

Grease is a preferred form of lubrication in certain applications because it gives low friction, is easily confined, and has a long lubricating life at low cost. (Brajendra K. Sharma *et al*., 2006)

1.1.3.2 Thickener

The thickener essentially the gelling agent, holds the matrix together. (Atanu Adhvaryu et. el, 2004). The primary type of thickener used in current grease is metallic soap. These soaps include lithium, aluminum, clay, polyurea, sodium and calcium. Lately, complex thickener-type greases are gaining popularity. They are being selected because of their high dropping points and excellent load-carrying abilities. (Jeremy Wright, 2008)

Complex greases are made by combining the conventional metallic soap with a complexion agent. The most widely used complex grease is lithium based. These are made with a combination of conventional lithium soap and a low- molecular-weight organic acid as the complexion agent.

Non-soap thickeners are also gaining popularity in special applications such as high-temperature environments. Bentonite and silica aerogel are two examples of thickeners that do not melt at high temperatures. There is a misconception, however, that even though the thickener may be able to withstand the high temperatures, the base oil will oxidize quickly at elevated temperatures, thus requiring a frequent relube interval.

1.1.3.3 Additives

Grease additives (Silverstein, R. S.; Rudnick, L.R. 2003) are specialized compound that improve the existing physical and chemical performance properties of grease. These compounds just required a small amount, are evenly dispersed in the grease matrix, and undergo physical and/or chemical interaction with the moving mechanical metal part during use. (Hunter, M. E.;Baker, R.F. 2000). Additives can play several roles in lubricating grease. These primarily include enhancing the existing desirable properties, suppressing the existing undesirable properties, and imparting new properties. (Jeremy Wright, 2008).

The most common additives are:

- Anti-corrosion :-

Grease must prevent or reduce processes which lead to rust or corrosion.

- Anti-wear:-

Grease must prevent wear by forming a strong surface film to reduce contact between surfaces to prevent heating as an outcome of extreme pressure.

- Anti-oxidant:-

Grease must prevent oxidation which will lead to damage which in turn will admit impurities or gives rise to acidity.

- Viscosity index improver :-

Additives will retain the viscosity of grease. Grease will break down at high engine temperatures or when the engine has run too long.

1.1.4 Categories / types

Grease can be divided into 6 general categories or types; i.e., mixtures of mineral oils and solid materials, heavy, asphaltic-type oils blended with lighter oils, extreme-pressure greases, roll-neck greases, soap-thickened mineral oils and multi-purpose grease (Ron Hughes, 2007)

- Mineral of oils mixed solids

These types of greases are very heavy lubricants for specialized applications. Such greases lubricate rough-fitting machine parts operating under heavy pressures or loads at relatively slow speeds. Examples of equipment that will typically use this type of grease include concrete mixers, bearings and rollers on conveyors and heavy construction equipment. (Ron Hughes, 2007)

- Heavy-Asphaltic-type oils blended with lighter oils

These types of lubricants are classified as greases but are actually thick, heavy oils used to lubricate open-type gearing and wire rope. A primary advantage of these oils is that they form a heavy protective film when heated or painted on surfaces and then allowed to cool. Lighter oil is typically blended with the heavy oils in order to improve the pour point of the oil. (Ron Hughes, 2007)

- Extreme-pressure greases

The unique characteristic of this type of grease is that it contains additives to improve firm strength under various applications. In essence, film strength provides the resistance of the lubricant to being torn apart, thus preventing metal-to-metal contact of the equipment being lubricated. A film is formed by a chemical reaction of the metal to the additives in the grease. The chemical reaction is usually brought about (or accelerated) by pressure exerted on the grease, creating heat. (Ron Hughes, 2007)

A few of the additives used in EP greases include compounds containing parts of chlorine, phosphorus, active and/or passive sulfur, chlorinated waxes and phosphates. Zinc and lead may also be added, as well as asbestos in some lubricants as a filler to cushion the shock loading on gear drives. The specific additive being used will always depend on the application for use. Factors to be considered for types of additives include specific equipment operating conditions such as load, speed, surface condition and inherent design characteristics (Ron Hughes, 2007)

- Roll Neck Greases

Roll neck greases are specialized lubricants used almost exclusively for lubricating plain bearings in rolling equipment. For example, it’s fairly common to use a block of NLGI No. 6 grease, which has the consistency of a bar of soap, carved to mate with the shape needed to accommodate the bearing of heavily loaded equipment. (Ron Hughes, 2007)

- Soap Thicked Mineral Oils

This is by far the most widely used category of grease in industry today. This type of grease varies by the additive that forms the soap in the lubricants chemical makeup. (Ron Hughes, 2007)

Sodium-base greases are also general-purpose greases. Because they have a higher dropping point (approximately 300^{o} to 350ºF), they are often used to lubricate machine parts operating near heat. Sodium greases made with lighter oils are used for ball and roller bearing lubrications, as are combinations (mixed base) of calcium and sodium grease. Sodium-soap greases have a spongy or fibrous texture and are yellow or green in color. Because of their working stability and intermediate melting point, they are used for lubricating wheel bearings (other than disc brakes) and for general-purpose industrial applications. Typical examples include rough, heavy bearings operating at low speeds, as well as skids, track curves and heavy-duty conveyors. (Ron Hughes, 2007)

Barium-soap greases are general-purpose types, valued for their ability to work over a wide temperature range. Their dropping point is approximately 350ºF or higher, although they are not intended to be used in continuous operation at temperatures above 275ºF. Barium-soap greases are chosen for a variety of jobs, especially for nearly all types of bearing lubrications. They have high-soap content. However, this makes this type of lubricant less suitable for use at low temperatures and in very high-speed applications. They have a buttery of fibrous texture and are reddish-yellow or green in color. (Ron Hughes, 2007)

Lithium-soap grease handles extremes of temperature quite well, which makes them highly suitable for both high and low temperature application. They have a dropping point of approximately 350ºF, and can be used in continuous temperatures of 300ºF. One reason for their successful low-temperature performance is that they are made with oil having a low pour point. In fact, lithium-soap greases have been used successfully at temperatures of -60ºF. Use of lithium-soap grease at higher temperatures requires a different formula; however the same grease can't be used at both extremes of high and low temperatures because the change would be in the viscosity of the oil used in the grease. Basically, lithium-soap greases have very good stability; good water resistance, and are also readily pumpable. They have a buttery texture and a brownish-red color. (Ron Hughes, 2007)

Calcium-soap, also called lime-soap greases, are probably the best known and most often used of all greases. Depending on the method of manufacture they are usually relatively inexpensive. Uses include axle grease, water pump grease and general machinery applications. Because its water content begins to dry out, and the soap and oil separate, calcium-soap grease isn’t suited to applications where the temperature will get above 160ºF. The major advantage of calcium-soap grease is that they don’t dissolve in water. However, it is not suited to use in lubricating high-speed bearings. Ordinary general-purpose calcium-soap greases have a dropping point of approximately 175^{o} to 200ºF. Calcium-complex grease has unusually high heat resistance making it of considerable value in extreme-pressure applications. The dropping point of this type of grease is 500ºF or even higher. This means that this type of lubricant will maintain its stability while running continuously at high temperatures. However, this type of grease has not replaced lithium-soap greases because they are not as mechanically stable.

Calcium-soap greases are yellow or reddish in color, and have a smooth buttery texture. (Ron Hughes, 2007)

Aluminum-soap greases are special-purpose lubricants. Their particular advantage is that they are very sticky making them perfect for applications requiring surface lubrication. (Ron Hughes, 2007)

- Multi- Purpose Grease

Multi-purpose greases combine the properties of two or more specialized greases. This permits the use of a single type of grease for a variety of applications. It is possible to replace as many as six specialized greases with single multi-purpose grease and get better results all at the same time. Most of the multi-purpose greases have a soap base of barium, lithium, or calcium complex. For example, the lithium-soap greases discussed earlier. They are not only water-resistant and corrosion inhibiting, but they have very good mechanical and oxidation stability as well.

By reducing the number of lubricants, which a company keeps in stock, the lubricator's job becomes much easier. Another advantage is that it helps reduce the chances of error in application.

Good multi-purpose lubricating grease has to perform well in a number of applications. It should have a high melting point, and operate well at continuous temperatures of 250ºF or more. Such grease should also have good resistance to water, and exceptional stability.

1.1.5 Characteristic

As with oil, grease displays its own set of characteristics that must be considered when being chosen for an application. The characteristics commonly found on product data sheets include the following: (Jeremy Wright, 2008)

1.1.5.1 Water resistance.

This is the ability of grease to withstand the effects of water with no change in its ability to lubricate. Soap/water lather may suspend the oil in the grease, forming an emulsion that can wash away or, to a lesser extent, reduce lubricity by diluting and changing grease consistency and texture.

1.1.5.2 Consistency.

Grease consistency depends on the type and amount of thickener used and the viscosity of its base oil. Grease's consistency is its resistance to deformation by an applied force. The measure of consistency is called penetration. Penetration depends on whether the consistency has been altered by handling or working. ASTM D 217 and D 1403 methods measure penetration of not worked and worked greases. To measure penetration, a cone of given weight is allowed to sink into a grease for five seconds at a standard temperature of 25°C (77°F).

The depth, in tenths of a millimeter, to which the cone sinks into the grease, is the penetration. A penetration of 100 would represent solid grease while a penetration of 450 would be semi fluid. The NLGI has established consistency numbers or grade numbers, ranging from 000 to 6, corresponding to specified ranges of penetration numbers. Table 1 lists the NLGI grease classifications along with a description of the consistency of how it relates to common semi fluids.

1.1.5.3 Dropping point:

Dropping point is an indicator of the heat resistance of grease. As grease temperature increases, penetration increases until the grease liquefies and the desired consistency is lost. The dropping point is the temperature at which grease becomes fluid enough to drip. The dropping point indicates the upper temperature limit at which grease retains its structure, not the maximum temperature at which grease may be used.

1.1.5.4 Oxidation stability.

This is the ability of grease to resist a chemical union with oxygen. The reaction of grease with oxygen produces insoluble gum, sludge and lacquer-like deposits that cause sluggish operation, increased wear and reduction of clearances. Prolonged exposure to high temperatures accelerates oxidation in greases.

1.1.5.5 High-temperature effects.

High temperatures harm greases more than they harm oils. Grease, by its nature, cannot dissipate heat by convection like circulating oil. Consequently, without the ability to transfer away heat, excessive temperatures result in accelerated oxidation or even carbonization where grease hardens or forms a crust.

Effective grease lubrication depends on the grease's consistency. High temperatures induce softening and bleeding, causing grease to flow away from needed areas. The mineral oil in grease can flash, burn or evaporate at temperatures greater than 177°C (350°F).

1.1.5.6 Low-temperature effects.

If the temperature of grease is lowered enough, it will become so viscous that it can be classified as a hard

1.1 Problem Statement

Mineral oils are the most commonly used and they consist predominantly of hydrocarbons but also contain some sulfur and nitrogen compound with traces of a number of metals

1.2 Objective

To determine the effect on the properties of fume silica percent formulated grease by several type of testing will be involve such as:

- Penetration (ASTM D-127)
- Corrosive substance (ASTM D-130)
- Drop Point (ASTM D-2265)
- Viscosity

1.3 Scope of study

The variables that involve in this experiment are the different percentage of fume silica, constant percentage (%wt) of additive and constant percentage (%wt) of based oil

- The percentages of thickening agent (fume silica) are less than 1 - 15% by weight of total formulation.
- Constant percentage (%wt) of additive at 0.5%wt of iron octoate and 5%wt of molybdenum di-sulfide
- Constant percentage (%wt) of based oil (cooking oil) at 100g

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Grease consists of oil and/or other fluid lubricant that is mixed with another thickener substance, a *soap*, to form a solid. Grease-lubricated bearings have greater frictional characteristics at the beginning of operation. Under shear, the viscosity drops to give the effect of an oil-lubricated bearing of approximately the same viscosity as the base oil used in the grease. Lithium-based greases are the most commonly used; sodium and lithium based greases have higher melting point than calcium-based greases but are not resistant to the action of water. (Mr. M.E. Spaght and Nav Tec Miseu, 1945)

Grease is the preferred form of lubrication in hard-to-reach places in a mechanically rubbing or dynamic systems. Grease acts as reservoir for lubricant-based fluids and additive molecules. Much of its functional properties are dependent on their ability to flow under force, have shear stability, resist viscosity changes with temperature and pressure, water stable, seal out contaminants, decrease dripping and spattering, etc. The dependability of lubricating grease depends on their physical properties that are structurally related, which is obtained by the proper selection of ingredients and processing. Thus, it is pertinent to understand the grease microstructure as it contributes significantly to various functional properties of grease. Grease consistency (or National Lubricating Grease Institute (NLGI)