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**JUDUL:** PRODUCTION OF HIGH TEMPERATURE GREASE FROM WASTE LUBRICANT

SLUDGE AND SILICONE OIL

**SESI PENGAJIAN:** 2010/2011

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CHERYL YEUNG SUET LING

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Taman Klang Jaya,  
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\_\_\_\_\_  
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Signature:

Supervisor: Siti Kholijah Binti Abdul Mudalip

Date:

PRODUCTION OF HIGH TEMPERATURE GREASE FROM WASTE LUBRICANT  
SLUDGE AND SILICONE OIL

CHERYL YEUNG SUET LING

A report submitted in partial fulfillment of the  
requirements for the award of the degree of  
Bachelor of Engineering (Chemical Engineering)

Faculty of Chemical and Natural Resources Engineering  
Universiti Malaysia Pahang

NOVEMBER 2010

I declare that this thesis entitled “ *Production of High Temperature Grease From Waste Lubricant Sludge and Silicone Oil* “ is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name : Cheryl Yeung Suet Ling

Date : November 2010

To my beloved mother and father

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## ABSTRACT

This research was carried out to study the production of high temperature grease from waste lubricant sludge and silicone oil. The effects of different ratios of waste lubricant sludge to fumed silica and mixing time on grease characteristics were investigated. The ratios of waste lubricant sludge and fumed silica used were 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 and 40:60. In terms of mixing time, it was varied at 1, 2, 3, 4 and 5 hours. Throughout the experiment, the grease was prepared by using the heating and mixing technique. The grease produced was analysed according to the ASTM and NLGI standard via penetration test, dropping point test, copper corrosion test, FTIR and AAS. . Fourier transform infra red (FTIR) analysis was carried out to study on the functional groups present in the grease. Atomic absorption spectroscopy analysis was also carried out to determine the concentration of a specific metal element in the grease. The results showed that the grease produced with a minimum ratio of fumed silica to sludge (10:90) was able to produce high temperature grease with a dropping point of 272.5°C. Further analysis verified that the grease is a high temperature grease, has worked penetration of 270-290 which makes the grease fall into the common grease category, has a low tendency to corrode copper and very little metal element was present in the grease. It can be concluded that the ratio of fumed silica to sludge and the mixing time of the grease produced significant effect on worked penetration and dropping point of the grease.

## ABSTRAK

Kajian ini dijalankan dengan tujuan mengkaji penghasilan gris bersuhu tinggi daripada sisa pelumas lumpur dan minyak silikon. Kesan daripada penggunaan nisbah yang berlainan bagi sisa pelumas lumpur kepada silika kesal dan masa campuran ke atas ciri-ciri gris telah dikaji. Nisbah sisa pelumas lumpur dan silika kesal yang digunakan adalah 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 dan 40:60. Masa campuran yang digunakan adalah 1, 2, 3, 4 dan 5 jam. Sepanjang eksperimen dijalankan, gris telah dihasilkan dengan menggunakan teknik memanaskan dan mencampur. Gris yang dihasilkan telah dianalisis mengikut standard ASTM dan NLGI dengan menggunakan ujian penetrasi, ujian titik jatuhan, ujian penghakisan kuprum, FTIR dan AAS. Analisis infra merah merubah fourier (FTIR) telah dijalankan untuk mengkaji kehadiran kumpulan berfungsi di dalam gris. Spektroskopi penyerapan atom (AAS) dijalankan untuk mengkaji konsentrasi unsur logam spesifik di dalam gris. Keputusan menunjukkan bahawa gris yang dihasilkan dengan nisbah minima silika kesal kepada sisa pelumas lumpur (10:90) dapat menghasilkan gris bersuhu tinggi dengan titik jatuhan pada 272.5°C. Analisis lanjut memastikan bahawa gris itu merupakan gris bersuhu tinggi, mempunyai penetrasi bekerja sebanyak 270-290 dan memasukkan gris itu ke dalam kategori gris umum, mempunyai kecenderungan yang rendah untuk menghakis kuprum dan konsentrasi unsur logam adalah rendah di dalam gris tersebut. Sehubungan dengan itu, boleh disimpulkan bahawa nisbah silika kesal kepada sisa pelumas lumpur dan masa campuran gris mempunyai kesan signifikan ke atas penetrasi bekerja dan titik jatuhan gris



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**LIST OF ABBREVIATIONS**

AAS	-	Atomic Adsorption Spectroscopy
ASTM	-	American Society For Testing and Materials
BC	-	Before Century
DSC	-	Differential Scanning Calorimetry
EP	-	Extreme Pressure
FTIR	-	Fourier Transform Infrared
KGF	-	Kilogram Force
NLGI	-	National Lubricating Grease Insitute
PSI	-	Pound Per Square Inch
RPM	-	Radium Per Minute
RUL	-	Remaining Useful Life

**LIST OF SYMBOLS**

°C	-	Degree Celcius
F	-	Fahrenheit
ml	-	Milliliter
wt. %	-	Weight percent
%	-	Percentage



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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background of Study**

Greases are one of the oldest forms of lubricating material and in the early days, greases are made from environmentally friendly and biodegradable materials such as mutton fat, beef fat and lime. Ancient Egyptians about 1400 B.C made crude greases to lubricate the wheels of their chariots (Rudnick, 2003). Grease is a solid to semi-solid lubricant consisting of a thickening agent dispersed in a liquid lubricant such as mineral oil (petroleum oil), synthetic oil (silicone oil) or vegetable oil. The most important factors affecting the properties and characteristics of a grease are firstly, the amount and type of thickener used, secondly, the viscosity and physical characteristics of the oil and last but not least, the additives (Bloch, 2005). Simply put, grease is made up of three essential components which are the base oil, thickener and additives.

Different types and combinations of base fluids with the thickener and additives and the way in which the grease is made will produce greases with different lubricating properties. For example, low-viscosity oil is chosen as the base oil in situations where light loads, fast speeds and low temperatures are applied whereas high viscosity base oils are for differing conditions. The thickener will determine grease properties such as water resistance, high-temperature limit, resistance to permanent structural breakdown, "stay-put" properties, and cost.

Currently, many researches were being done to search for alternative sources for grease production due to the increasing of crude oil prices in the world market and the depletion of the source. Thus, this will definitely affect the price of base oil as main component in grease. In this research, instead of using mineral oil as the base oil, silicone oil which is a more economic choice and is more abundantly available was used. To date, there is no specific research on the production of grease using waste lubricant sludge as part of its component. That is why, this research would have a significant impact in the production of grease as it proves that sludge, which is a waste could be turned into wealth and thus reducing the pollution problem and at the same time reducing the cost of the grease produced.

## **1.2 Problem Statement**

The NLGI Grease Production Survey Report for the year 2000 reports a grand total of approximately 1.5 billion pounds of lubricating grease manufactured worldwide (Rudnick, 2003). Petroleum derived base oils currently account for about 97% of the total lubricant production (Bhaskar *et.al.*, 2003). However the supply of petroleum is diminishing and thus the prices of the fuel are leaping at a significant rate. Besides

being a major contributing factor to the current economy crisis , petroleum generate harmful acids which fall to earth as acid rain ,contains radioactive material and not to mention emits large amount of carbon monoxide and carbon dioxide when being combusted .In economic terms, pollution from petroleum is regarded as a negative externality. Many alternatives are being discovered to replace this limited resource. In order to overcome this problem, action should be taken to explore the utilisation of new raw materials for grease production. Therefore attempt were made in this research to study on the potential of utilising waste lubricant sludge to produce grease.

In the year 2004, 37.4 million tons of lubricants were consumed worldwide. About 50% of the lubricants sold worldwide end in and thus pollute the environment (Mang and Dresel, 2007). These wastes which were not properly disposed are toxic to the environment as it contains toxic chemicals and are not biodegradable. Waste lubricant oil contain significant amount of base oil and it is worth recovering it using solvent extraction process. However, in this study, waste lubricant sludge was recovered instead as till today; the research on the recovery of sludge is still very limited. Sludge was proven to have potential as thickener in the production of grease in this study. This step will not only reduce the disposal cost but will also produce grease at a low cost. As mentioned, using sludge as the thickener is also an incentive in this research. The reason saying so is because the thickening additive that is being used which is the fumed silica is a very good thickener but it is also very expensive. So in order to decrease the amount used in the production of grease, sludge was used to replace a significant amount of fumed silica .It is found that sludge may also have the thickening property that is needed in producing grease.

The recycling of these wastes is not only beneficial to the animals and the environment but is also important in keeping the society free from health hazards. The severity of this waste generation might not be seen now but it will definitely impact the society some time later. That is why, it is important to take the first step in recycling

and turning waste to wealth no matter how small the step is. The recycling of the waste lubricating oil will stop these wastes from being improperly discarded into the environment. Besides that, this study will also be very economical when comparing to producing grease from fresh base oil. This study is also done in the hopes that society will come to realise the importance of recycling and at the same time turning waste to wealth as the treatment of wastes has become one of the most important concerns of modern society to protect the environment.

### **1.3 Objective**

To study on the production of high temperature grease from waste lubricant sludge.

### **1.4 Scope of Study**

In order to achieve the objective of the research study, several scopes have been identified:

- 1) To produce Calcium Fluoride a thermal resistant additive for grease preparation.
- 2) To study on the effects of using different ratios of waste lubricant sludge and fumed silica on grease characteristics. The ratios used are 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 and 40:60.

- 3) To study on the effects of using different mixing time on grease characteristics. The mixing times used are 1, 2, 3, 4 and 5 hours.
- 4) To characterize the grease produced by using penetration test and dropping point test.
- 5) To compare the characteristics of the grease produced.

### **1.5 Rationale and Significance**

The production of high temperature grease from silicone oil and waste lubricant sludge contributes to the community and the environment in more than one way. For starters, this research will be a stepping stone for other researches who will be doing researches similar to this and who intend to curb pollution. It will be a guide for them to what's to do and what's not to do in the future.

In the aspect of the society, this research will protect them from more toxic pollution of waste lubricant sludge. The protection of the environment is crucial as the cycle of life depends on it. Thus through this research, it will not only tackle the pollution problem but will also lower the chances of wildlife being exposed to these dangerous substances. It is hoped that the concept of 'zero waste' and 'waste to wealth' can be cultivated in our society for the betterment of the future.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Introduction**

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 (Wright, 2001). Figure 2.1 shows the general composition of base oil, thickener and additive to produce grease.

There are a few reasons why greases are more favourable than oil as a lubricant. Due to its consistency, grease acts as a sealant to prevent lubricant leakage and also to prevent entrance of corrosive contaminants whereas oil would simply seep away. Besides that, 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. Another advantage of grease is that it holds solid lubricants in suspension. Finely ground solid lubricants, such as molybdenum disulfide are mixed with grease in high temperature service (over 315°C or 599°F) or in extreme high-pressure applications. Grease holds solids in suspension while solids will settle out of oils. Lastly the fluid level of grease does not have to be controlled and monitored.

However, there are some disadvantages when using grease. Due to its consistency, grease cannot dissipate heat by convection like circulating oil. Grease has more resistance to motion at start-up than oil, so it is not appropriate for low torque/high speed operation. It is also more difficult to handle than oil for dispensing, draining and refilling. Also, exact amounts of grease cannot be as easily metered (Engineering Edge, 2010).

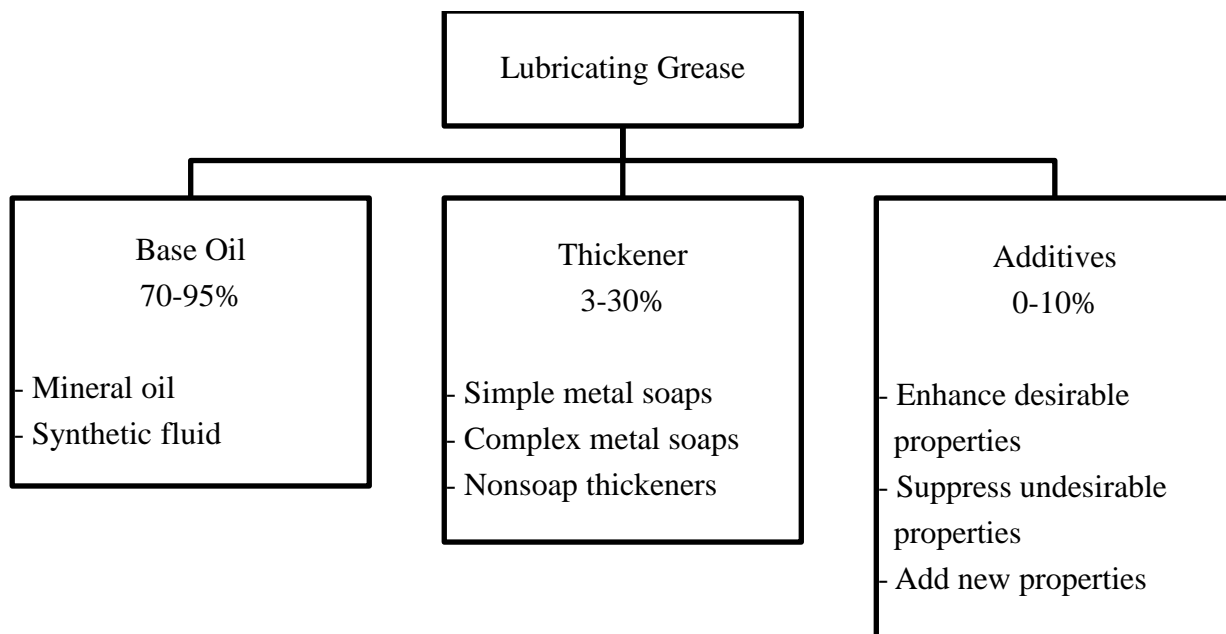
The function of grease is to remain in contact with and lubricate moving surfaces without leaking out under gravity or centrifugal action, or be squeezed out under pressure. Its major practical requirement is that it retain its properties under shear at all temperatures that it is subjected to during use. At the same time, grease must be able to flow into the bearing through grease guns and from spot to spot in the lubricated machinery as needed, but must not add significantly to the power required to operate the machine, particularly at startup (Boehringer, 1992).

Generally, a satisfactory grease for a given application must reduce friction and wear in the machine element being lubricated under various operating conditions. Besides that, a grease function as a rust and corrosion protector. Greases also prevent dirt, water and other contaminants from entering the part being lubricated and at the same time, tolerate and repel some degree of moisture so that contamination does not occur. All this happen without significant loss of performance. Many types of grease are used in an extended period of time thus it is expected to maintain its structure and



consistency during long periods of use. At low temperature, a good grease permits free motion of moving parts. Furthermore, different greases have different characteristics for different methods of application. A satisfactory grease should retain those characteristics during storage and not change in chemical properties or physical appearance.

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. Grease is generally used for machinery that runs intermittently or is 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 also requires grease. 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. Machinery operating under extreme conditions such as high temperatures and pressures, shock loads or slow speed under heavy load prefers the usage of greases compared to oils. Worn components is another suitable application for grease as grease maintains thicker films in clearances enlarged by wear and can extend the life of worn parts that were previously lubricated by oil (Engineering Edge, 2010).



**Figure 2.1** Grease Anatomies (Wright, 2001)

## 2.2 Base Oils

In terms of volume, base oils are the most important components of lubricants. As a weighted average of all lubricants, they account for about +70-95% of lubricant formulations (Wright, 2003). Many different types of base oils may be used in the manufacture of grease, including mineral (petroleum), vegetable (palm oil) and synthetic oil (esters, silicones, and glycols). As mentioned before, the viscosity of the base oil is the most significant property. Lighter, lower viscosity base oil is used to formulate low temperature greases or greases suitable for high rotational speeds, while heavier, higher viscosity base oils are used to formulate greases used in applications where high loading is encountered, high temperature or low rotational speeds are seen. In this research silicone oil was used as the base oil. Silicone oil is used to produce

silicone grease which is an amorphous fumed, silica-thickened, polysiloxane-based compound. Silicone greases provide lubrication, corrosion resistance and also maintain stability under high temperatures which was one of the reasons why silicone oil was chosen as the base oil in this research (Rudnick, 2006).

### **2.3 Thickener**

Thickener is the term describing the ingredients added to the base oil in order to thicken it to a grease structure. Thickeners are, in fact what make grease grease and it is the component that differentiates grease from conventional lubricating fluid. The thickening agent may be an inorganic or an organic substance. Organic thickeners can be either soap-based or non-soap based, while inorganic thickeners are non-soap based. They are designed to reduce migration and to prevent leakage under gravity. The thickening agent makes up about 3-30% of the entire grease. Fumed silica was chosen as the thickener in this research as fumed silica has a very strong thickening effect. Waste lubricant sludge was also used as part of the thickener in this research in the effort to reduce the amount of fumed silica being used so that the cost of grease production could be lowered.

## **2.4 Additives**

Additives are added to some grease to improve their lubricating properties. Besides enhancing performances, additive also plays a role in protecting the grease and lubricated surfaces. The most common additives found in greases are for example; antioxidants (prolong the life of a grease), anti-corrosion agent (protect metal against attack from water, sulphides or corrosive elements), Extreme Pressure agents (guard against scoring and galling) and anti-wear agents (.prevent abrasion and metal to metal contact). There are about +0-10% of additives in grease. Molybdenum disulfide and calcium fluoride (EP & high thermal resilience additives) were the additives used in this research. (Takadoum, 2008)

## **2.5 Types of Grease**

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.

### **2.5.1 Mineral Oils Mixed with 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 (Hughes, 2010).

### **2.5.2 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 (Hughes, 2010).

### **2.5.3 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.

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 (Hughes, 2010).

#### **2.5.4 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 (Hughes, 2010).

### 2.5.5 Soap Thicken 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.

Sodium-base greases are also general-purpose greases. Because they have a higher dropping point (approximately 300° to 350°F), 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.

Barium-soap greases are general-purpose types, valued for their ability to work over a wide temperature range. Their dropping point is approximately 350°F or higher, although they are not intended to be used in continuous operation at temperatures above 275°F. Barium-soap greases are chosen for a variety of jobs, especially for nearly all types of bearing lubrications. They have a 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 or fibrous texture and are reddish-yellow or green in color.

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 350°F, and can be used in continuous temperatures of 300°F. 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 -60°F. Use of a 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.

Calcium-soap, also called lime-soap greases, is 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 160°F. 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 175o to 200°F. 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 500°F 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. Aluminum-soap greases are special-purpose lubricants. Their particular advantage is that they are very sticky making them perfect for applications requiring surface lubrication (Hughes, 2010).



### **2.5.6 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 250°F or more. Such grease should also have good resistance to water, and exceptional stability (Hughes, 2010).

## **2.6 Grease Manufacturing Process**

### **2.6.1 Batch Process**

Greases can be made in either a batch or continuous process. Batch production is the most common manufacturing method. The steps of manufacturing include the following. Bulk ingredients are metered or weighed into the processing reactor. For

soap-based greases made by saponification (the process of forming soap by splitting a fat with an alkali), the fatty ingredient, alkali and a portion of the oil are added to the reactor.

By heating (300 - 450°F) and mixing, the fat is converted to soap, and the soap is dispersed throughout the mixture. This may be done in open kettles or in closed pressure kettles. After completion of saponification and dehydration (removal of water), the remaining oil is added to the batch to lower the temperature. Next, the grease is milled or homogenized. This step of homogenization or milling is very important, because it will produce a uniform crystal and gel structure that will not change when the grease is used. Homogenizing the grease will break down the solid particles or fibers and will disperse the resultant small particles in the liquid. It also breaks up lumps, eliminates graininess and produces a smooth product. Homogenization of certain types of greases will stiffen the grease producing lower penetration value. Homogenization can improve texture and “brighten” a grease’s appearance.

In many cases this homogenization process is carried out at temperatures greater than 200°F (93°C). After homogenization, the grease is further cooled, deaerated and packaged. Of course, it is understood that there are many different grease manufacturing methods depending on the type of grease and the manufacturer. The single-stage homogenizer with wear-resistant parts may be operated at up to 10,000 psi. The homogenizer is the preferred piece of equipment for the solid-additive type greases, because high energy is needed to break up and delaminate the particles such as with clay dispersions. Although a colloid mill can be used to process grease, there are advantages to using the homogenizer. First, the homogenizer is a constant-displacement pump, and its capacity does not vary with different grades of greases. This makes it possible to tie into filling equipment, if desired. The colloid mill must be pump fed and the capacity will be significantly decreased on the stiffer grades of greases. The homogenizer has sufficient pressure to deliver the grease to any point in the process

after it has been homogenized; but a second pump must pick up the colloid mill product, if the grease is to be delivered to another location (SPX Corporation, 2008).

### **2.6.2 Continuous Process**

In the continuous production, the feed materials and lubricating oil were introduced into selected locations of a screw process unit which contains a series of adjacent, longitudinally connected barrel sections for performing different operative steps and houses a rotating screw device traversing the interior of the barrel sections and having separate elements along its length to perform desired operations. The feed materials were then mixed and conveyed along said process unit through adjacent barrel sections by continuous operation of said rotation screw. The temperature of said material was controlled while it is being conveyed through said process unit by use of various heat exchanger means which are located in or adjacent each barrel to aid in carrying out the operative steps of dispersion, reaction, dehydration and/or homogenization. The resulted water from the dehydration of the feed mixture was vented out at selected barrel discharge points in process unit. Additional oil and additives were introduced at downstream barrel sections following the dehydration step. The complete grease formulation was homogenized by continued rotation of screw device and the finished lubricating grease were removed from the end barrel section of screw process unit (Alexander, 1981).

## **2.7 Grease Characteristics**

### **2.7.1 Apparent Viscosity**

As grease has a high viscosity, it will have a resistance towards motion during start up. However, with higher motion and with shearing between wearing surfaces, the grease's resistance to flow reduces. Its viscosity decreases as the rate of shear increases. By contrast, oil at constant temperature would have the same viscosity at start-up as it has when it is moving. To distinguish between the viscosity of oil and grease, the viscosity of grease is referred to as "apparent viscosity." Apparent viscosity is the viscosity of grease that holds only for the shear rate and temperature at which the viscosity is determined (Engineering Edge, 2010). The ratio of stress to rate of strain, calculated from measurements of forces and velocities as though the liquid were Newtonian. If the liquid is actually non-Newtonian, the apparent viscosity depends on the type and dimensions of the apparatus used (IUPAC, 1997).

### **2.7.2 Bleeding, Migration and Syneresis of Grease**

Bleeding occurs when the liquid lubricant separates from the thickener. High temperature and long storage periods are factors that causes bleeding to happen. Migration is a form of bleeding that occurs when oil in a grease seeps out of the thickener network under certain circumstances. For example, when grease is pumped through a pipe in a centralized lubrication system, it may encounter a resistance to the flow and form a plug. The oil continues to flow, migrating out of the thickener network. As the oil separates from the grease, the thickener concentration increases, and plugging gets worse. If two different greases are in contact, the oils may migrate from one grease to the other and change the structure of the grease. Therefore, it is unwise to mix two

greases. Syneresis is a special form of bleeding caused by shrinking or rearrangement of the structure due to physical or chemical changes in the thickener (Engineering Edge, 2010).

### **2.7.3 Consistency, Penetration, and National Lubricating Grease Institute (NLGI) Numbers**

The rigidity and consistency of grease is an important feature of the grease. A grease that is too viscous may not be able to seep into areas requiring lubrication, while a grease that is too fluid may leak out. The consistency of the grease depends heavily 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 unworked 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 a solid grease while a penetration of 450 would be semifluid. The NLGI has established consistency numbers or grade numbers, ranging from 000 to 6, corresponding to specified ranges of penetration numbers. Table 2.1 lists the NLGI grease classifications along with a description of the consistency of how it relates to common semifluids (Engineering Edge, 2010).

**Table 2.1: NLGI Grease Classification System**

NLGI Grade	Worked penetration after 60 Strokes at 25 °C (0.1 mm)	Appearance	Consistency food analog
000	445-475	Fluid	cooking oil
00	400-430	semi-fluid	applesauce
0	355-385	very soft	brown mustard
1	310-340	Soft	tomato paste
2	265-295	"normal" grease	peanut butter
3	220-250	Firm	vegetable shortening
4	175-205	very firm	frozen yogurt
5	130-160	Hard	smooth pate
6	85-115	very hard	cheddar cheese

#### 2.7.4 Contaminants in Grease

Many bearings fail prematurely due to contamination. Grease contamination can come not only from common environmental contaminants such as dirt and water, but also cross-contamination from other grease types. This is a major issue with greases because many gelling agents are incompatible, resulting in either a significant change in consistency (either thicker or thinner), or a separation of the oil from the gelling agent (Herguth, 2002).

### **2.7.5 Corrosion and Rust Resistance of Grease**

Corrosion and rust resistance of grease is the ability of grease to protect the metal parts that it is lubricating from being corroded and getting rusty or in short chemical attack. The natural resistance of a grease depends upon the type of thickener that is being used. Corrosion-resistance can be enhanced by corrosion and rust inhibitors (Engineering Edge, 2010).

### **2.7.6 Dropping Point of Grease**

Dropping point indicates the ability of a grease to resist heat. As grease temperature increases, penetration increases until the grease liquefies and the desired consistency is lost. The dropping point of a grease is the temperature at which the grease changes from semi-solid to a liquid. The dropping point establishes the maximum useable temperature of the grease, which is typically set at 50°C to 100 °C below the experimentally determined dropping point. Dropping point can help to establish if the correct grease was supplied or is in use, and to determine if a used grease is good for continued service (Herguth, 2002).

### **2.7.7 Evaporation of Grease**

The evaporation of grease here refers to the evaporation of the base oil. Excessive oil evaporation causes the grease to harden as the thickener concentration increases. Therefore, higher evaporation rate for the base oil requires more frequent relubrication (Engineering Edge, 2010).

### **2.7.8 Fretting Wear and False Brinelling**

Fretting refers to wear and sometimes corrosion damage at the contact points. This damage is induced under load and in the presence of repeated relative surface motion such as vibration or some other force. Sometimes, the oscillation is so minute that the grease is displaced from between parts but is not allowed to flow back in. False brinelling is damage caused by fretting that causes the imprints to look like brinelling but is actually caused by other factors. Material wear or removal indicates false brinelling and this usually occurs over an extended period from minute oscillation and light loads. Brinell damage can be defined as permanent material deformation and it occurs during one load event. False brinelling is basically caused by the pushing out of the lubricant from the loaded region causing the wear to increase (Engineering Edge, 2010).



### **2.7.9 Oxidation Stability - Grease**

A grease's ability to withstand a chemical fusion with oxygen is defined as oxygen stability. The reaction of grease with oxygen produces insoluble gum, sludges, and lacquerlike deposits that cause sluggish operation, increased wear, and reduction of clearances. Prolonged high-temperature exposure accelerates oxidation in greases (Engineering Edge, 2010).

### **2.7.10 Pumpability and Feedability of Grease**

Pumpability is the ability of a grease to be pumped or pushed through a system. More practically, pumpability is the ease with which a pressurized grease can flow through lines, nozzles and fittings of grease-dispensing systems. Feedability is the ability of the grease to be fed into a pump (Engineering Edge, 2010).

### **2.7.11 Water Resistance**

This is the ability of a grease to withstand the effects of water with no change in its ability to lubricate. A 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 (Engineering Edge, 2010).

### **2.7.12 Shear Stability of Grease**

The consistency of a grease may be changed as it is continuously being mechanically worked and sheared between wearing surfaces. The ability of a grease to maintain its consistency when being worked is called its shear stability or mechanical stability. A grease that softens as it is worked is called thixotropic. Greases that harden when worked are called rheopatic (Engineering Edge, 2010).

### **2.7.13 High Temperature Effects**

High temperatures harm greases more than they harm oils. Grease, by its nature, cannot dissipate heat by convection like a 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) (Engineering Edge, 2010).

#### **2.5.14 Low Temperature Effects**

If the temperature of a grease is lowered enough, it will become so viscous that it can be classified as a hard grease. Pumpability suffers and machinery operation may become impossible due to torque limitations and power requirements. As a guideline, the base oil's pour point is considered the low-temperature limit of a grease (Engineering Edge, 2010).

#### **2.7.15 Texture of Grease**

Texture is observed when a small sample of grease is pressed between thumb and index finger and slowly drawn apart. Texture can be described as:

- Brittle: the grease crumbles when being pressed or compressed.
- Buttery: the grease separates in short peaks with no visible fibers.
- Long fiber: the grease stretches or strings out into a single bundle of fibers.
- Resilient: the grease can withstand moderate compression without permanent deformation or rupture.
- Short fiber: the grease shows short break-off with evidence of fibers.
- Stringy: the grease stretches or strings out into long, fine threads, but with no visible evidence of fiber structure (Engineering Edge, 2010).

## 2.8 Grease Selection

Most users are aware of the importance of selecting the right lubricant for a given application. When it comes to selecting lubricating oils for manufactured equipment, it's easy to determine which products meet the original equipment manufacturer (OEM) requirements. OEM specifications for a lubricating oil normally include viscosity at operating or ambient temperature, additive requirements, base oil type and even special considerations for different environmental conditions. Grease specifications, on the other hand, often lack the detail necessary to make a proper selection, leaving it up to the lubrication engineer to create the specification.

A common OEM grease specification might be to use an NLGI (National Lubrication Grease Institute) No. 2 lithium grease of good quality. Using this information alone, one could select the right consistency and thickener type.

Due to the lack of specificity in most grease recommendations, it is important to learn how to properly select greases for each application in the plant. Proper grease specification requires all of the components of oil selection and more. Other special considerations for grease selection include thickener type and concentration, consistency, dropping point and operating temperature range, worked stability, oxidation stability, wear resistance, etc. Understanding the need and the methods for appropriate grease selection will go a long way toward improving lubrication programs and the reliability of lubricated machinery (Noria Corporation, 2005).

### **2.8.1 Base Oil Viscosity**

The most important property of any lubricant is viscosity. A common mistake when selecting a grease is to confuse the grease consistency with the base oil viscosity. Because the majority of grease-lubricated applications are element bearings, one should consider viscosity selection for those applications. There are several common methods for determining minimum and optimum viscosity requirements for element bearings, most of which use speed factors, commonly denoted as DN or NDm. Speed factors account for the surface speed of the bearing elements (Noria Corporation, 2005).

### **2.8.2 Additives and Base Oil Type**

Once the appropriate viscosity has been determined, it's time to consider additives. The additive and base oil types are other components of grease that should be selected in a fashion similar to that used for oil-lubricated applications. For instance, a lightly loaded high-speed element bearing does not require extreme pressure (EP) additives or tackifying agents, while a heavily loaded open gear set does (Noria Corporation, 2005).

### 2.8.3 Grease Consistency and Thickener Type

The consistency of grease is controlled by the thickener concentration, thickener type and the viscosity of the base oil. Even though base oil viscosity affects consistency, it is important to note that a grease can have a high consistency and a low base oil viscosity or vice versa. The NLGI has established a scale to indicate grease consistency which ranges from grades 000 (semifluid) to 6 (block grease). The most common NLGI grade is two and is recommended for most applications.

Numerous types of grease thickeners are currently in use, each with its own pros and cons. The most common types are simple lithium soaps, lithium complex and polyurea. Simple lithium soaps are often used in low-cost general-purpose greases and perform relatively well in most performance categories at moderate temperatures. Complex greases such as lithium complex provide improved performance particularly at higher operating temperatures. A common upper operating temperature limit for a simple lithium grease might be 250°F, while that for a lithium complex grease might be 350°F. Another thickener type that is becoming more popular is polyurea. Like lithium complex, polyurea has good high-temperature performance as well as high oxidation stability and bleed resistance. Thickener type should be selected based on performance requirements as well as compatibility when considering changing product types (Noria Corporation, 2005).

#### **2.8.4 Performance Properties**

Once the appropriate base oil viscosity, additive requirements and consistency have been determined, the remaining criteria to consider are the performance properties. This is where the grease quality factors in. Grease performance properties include many of the same properties used for lubricating oils, as well as others exclusive to grease. Properties exclusive to grease include dropping point, mechanical stability, water washout, bleed characteristics and pumpability. The most important performance properties are determined by the application. If an application operates continuously at room temperature, properties like dropping and upper operating temperature limits are not as important. If an application operates under heavy loads at low speeds, load carrying tests such as four-ball EP should be considered. It is important to remember that greases, like oils, have a careful balance of properties. A product may excel in one category and perform poorly in another. For this reason, it is important to weigh each property's significance relative to the intended applications to select the best overall fit.

Sometimes while trying to consolidate lubricants, it is easy to overconsolidate. Over time, this can lead to inaccurate lube specifications. A generic oil spec, including performance properties, should be configured for each lubricated point in the plant. Armed with this generic specification, it is easy to select the right product no matter what brand is preferred. It is also important to review these specifications on a periodic basis to guard against specification creep. While improving a lubrication program can be a tough job, lubricant specification is relatively easy (Noria Corporation, 2005).

## **2.9 Typical Property and Bench Tests for Grease**

### **2.9.1 Cone Penetration Test (ASTM D217)**

Consistency correlates to the firmness of the grease. Consistency is measured with the ASTM Cone Penetration Test D 217. Under specific conditions, a standardized cone is allowed to drop into the grease for 5 seconds. The level of penetration is measured to determine its NLGI consistency number, ranging from 000 to 6. The higher the penetration number, the lower the consistency number (ASTM D217-02, 2007). The standard for penetration from NLGI is available in Table 2.1.

### **2.9.2 Antioxidant Levels in Grease**

Greases, like oils, contain a variety of additives. Antioxidant levels are of particular interest in identifying the useful life of the grease. Differential Scanning Calorimetry (DSC) is a modern analytical method for measuring the onset of oxidation in used grease (ASTM D5483). When compared to the new reference grease, the test can be used to determine the remaining useful life (RUL) of a grease. This test is analogous in the information it seeks, if not in methodology, to the RPVOT test commonly used to determine the RUL of turbine oils and other lubricating oils.

DSC works by placing a sample of used grease into a test cell. The cell is heated and pressurized with oxygen. When the grease starts to oxidize, an exothermic reaction occurs, which liberates heat. By measuring the onset of the reaction in the used grease



(commonly called the induction point) in reference to the new grease, an estimate of the oxidation stability of the grease can be made (Herguth, 2002).

### **2.9.3 Viscosity of Grease**

The viscosity of grease is often misunderstood. The viscosity typically listed on a new oil data sheet is usually the kinematic viscosity of the oil used in blending the grease measured, using the standard ASTM D445 method. The kinematic viscosity of the base oil is important in ensuring the correct grease, containing the correct grade of oil is used for lubrication purposes. However, we can also measure the viscosity of the grease itself. Since a grease is non-Newtonian, we can only measure the apparent viscosity because the viscosity of a non-Newtonian fluid changes with shear stress (see “Understanding Absolute and Kinematic Viscosity” by Drew Troyer). The apparent viscosity of a grease is determined using ASTM D1092. This test measures the force required to force the grease through an orifice under pressure. As such, this test is an ideal way of determining the flow characteristics of grease through pipes, lines, dispensing equipment as well as its pumpability.

Rheology measurements of grease may soon replace both the cone penetration and the apparent viscosity measurements. Rheology is the study of the deformation and/or flow of matter when it is subjected to strain, temperature and time. A rheometer only requires a few grams of sample to perform the analysis, yielding much more information than the cone penetration or the apparent viscosity measurements. This makes the rheology measurement an ideal test for small amounts of used grease (Herguth, 2002).

#### **2.9.4 Contaminants in Used Grease**

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, a grease that is supposed to be an aluminum complex grease, but has become contaminated with a calcium sulfonate complex grease will show both aluminum and calcium in spectroscopic analysis, indicating a contamination problem (Herguth, 2002).

#### **2.9.5 Dropping Point (ASTM D566 and D2265)**

Temperature at which a drop of grease will fall from the bottom orifice of heated test cup, commonly reflects the change from a semisolid to a liquid state. As a general rule, the maximum usable temperature of a grease is at least 25 to 50°F below this drop point (Booser and Khonsari, 2007).

### **2.9.6 Evaporation Loss (ASTM D972 and D2595)**

Oil loss from grease at high temperatures can lead to premature hardening and loss of lubricating properties as the higher volatility (lower viscosity) portion of the mineral oil in a grease evaporates. This high-temperature evaporation increases progressively for mineral oil components as their viscosity drops below approximately 75 cSt viscosity at 40°C. Evaporation of 2 percent in 22 hours at 100°C (212°F) is a common specification limit for premium mineral oil greases and ranges down to 0.4 percent for synthetics (Booser and Khonsari, 2007).

### **2.9.7 Bleeding (Federal Test Method 321.2)**

Percentage of oil separating from a grease sample held in a wire cone is commonly measured after 30 hours at 100°C. A 2 to 5 percent range is often desirable. Lack of bleeding characterizes a grease which fails to supply adequate lubrication to rolling-element bearing surfaces and may result in noisy operation. Excessive bleeding leads to leakage and shortened grease life (Booser and Khonsari 2007).

### **2.9.8 Oxidation Stability (ASTM D942)**

Grease is exposed to pure oxygen at 110 psi at 99°C in a "bomb" for 100 to 500 hours. A decrease in oxygen pressure provides a preliminary indication of grease life in extended storage, prelubricated bearings and in high-temperature service. Maximum pressure drop in the bomb may be specified in the range of 3 to 25 psi in 100 hours, and 25 to 50 psi in 500 hours (Booser and Khonsari, 2007).

### **2.9.9 Wear and Extreme Pressure Properties (ASTM D2509, D2266 and D2596)**

Both wear and extreme pressure properties can be evaluated in similar four-ball tests using a half-inch rotating top steel bearing ball loaded on a cradled nest of three stationary balls immersed in the test grease. ASTM D2266 wear test measures scar diameter on the stationary balls after running one hour with 40 kg load. Wear scars range from approximately 0.4 to 0.6 mm at 1,200 rpm at 75°C (167°F). ASTM D2596 evaluates extreme pressure additive qualities by increasing load steps to seizure at the weld point, which ranges up to 400 to 500 kgf.

The linear oscillatory friction tester oscillates a steel ball on a disk while continuously recording the friction coefficient. Test frequency, stroke, temperature and specimen material and shape can vary to simulate field conditions. Limiting load at seizure is a useful measure of the EP and antiscuffing property of the grease (Booser and Khonsari, 2007).

### **2.9.10 Ball Bearing Grease Life (ASTM D3336 and D1741)**

Various customized ball bearing life tests at elevated temperatures have been developed to simulate automotive wheel bearings, electric motor bearings, military units and other equipment. ASTM D3336 runs a greased 204 (20 mm bore) ball bearings at 10,000 rpm at an elevated temperature either to failure or to a specified running time. Results at these high speeds and temperatures can be misleading: insufficient oil bleeding or excessive channeling could result in early high-speed failure of a NLGI Grade 3 long-life grease that would otherwise be ideal for sealing and for low-speed bearings.

ASTM D1741 uses a 306 (30 mm bore) ball bearing at 3,600 rpm to evaluate grease life at 125°C (257°F) for electric motor bearings and similar industrial uses. A lifespan of 10,000 hours can be expected with premium Grade 2 lithium and polyurea greases (Booser and Khonsari, 2007).

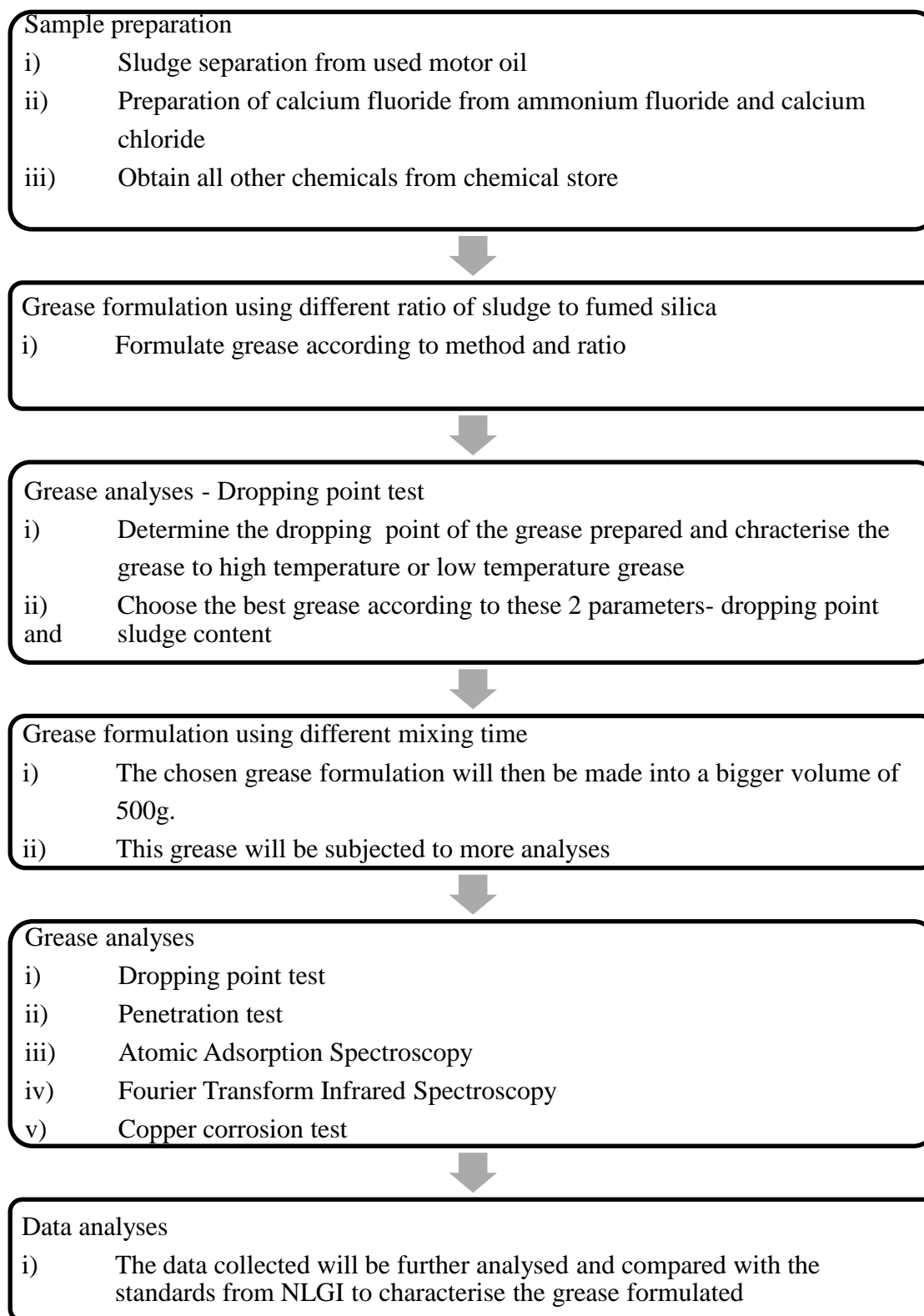
## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 Introduction**

In this study, the experimental works were classified into 2 parts. The first part is regarding the preparation samples which involve the separation of sludge from motor oil and the preparation of calcium fluoride from ammonium fluoride and calcium chloride. While the second part of the experiment involves the formulation of grease using different ratio of sludge to fumed silica and formulation of grease using different mixing time.

The greases obtained were then analysed and characterised using dropping point analysis, penetration test, copper corrosion test, Fourier Transform Infrared Spectroscopy and Atomic Adsorption Spectroscopy. Figure 3.1 shows the overall process flow for the experiment.



**Figure 3.1** Overall Process Flow

### 3.2 Materials

The materials that were used for sludge separations from motor oil were hexane 96% (Merck), isopropyl alcohol 99.7% (R & M Chemicals), potassium hydroxide 85% (R & M Chemicals) and used motor oil. The preparation of calcium fluoride needed calcium chloride 80% (R & M Chemicals), ammonium fluoride 98% (R & M Chemicals), distilled water and ethanol 96% (Merck). The materials used for grease preparation were sludge, silicone oil (R & M Chemicals), fumed silica (SIGMA-Aldrich), molybdenum disulphate 98.5% (ACROS Organics) and calcium fluoride. The materials used in the AAS analysis were standard sample solutions from copper, nickel, cadmium, chromium, lead and zinc. The material needed for the dropping point analysis was thermal oil (Shell Thermia B Oil).

### 3.3 Apparatus

The experiments were carried out with equipments such as the mechanical stirrer and heater. For grease analysis, the Fourier Transform Infrared Spectroscopy (FTIR), Atomic Absorption Spectroscopy (AAS), copper strip, dropping point apparatus and penetrometer were used



### **3.4 Experimental Procedures**

There are several steps involved in the formulation of grease. The steps involved are as below:

- i. Sludge separation from used motor oil
- ii. Preparation of calcium fluoride
- iii. Grease formulation using different ratios of sludge to fumed silica
- iv. Grease formulation using different mixing time

#### **3.4.1 Sludge Separation from Used Motor Oil**

In a 2 litre beaker, 360 ml of used motor oil were added. Next, 576 ml of hexane, 864 ml of isopropyl alcohol and 2.592 grams of potassium hydroxide were added. The mixed solution was stirred for 1 hour using a mechanical stirrer until the oil was completely dissolved in the solvents. The beaker of solution was then covered with an aluminium foil and set aside for at least 1 day to let the sludge settle to the bottom of the beaker. After a day, a layer of sludge could be seen at the bottom of the beaker. All the solvent were poured out and the sludge was filtered using a vacuum filter so that the sludge can be separated from the remaining solvent. The collected sludge was stored in a plastic container to be used later in the grease formulation. The weight of sludge for one mixing is about 65 grams. The steps above were repeated for a few more times to obtain the amount of sludge required.

### 3.4.2 Preparation of Calcium Fluoride

In a 500 ml flask, 0.01 mol of  $\text{CaCl}_2$  was dissolved in 100 ml distilled water. Then, 0.02 mol of  $\text{NH}_4\text{F}$  was dissolved in 100 ml of distilled water and added into the flask under vigorous stirring. The mixed solution was stirred for another 2 hour, with the gradual transformation of the reaction mixture from transparent solution into opaque white suspension. The solid was separated via centrifugation and was washed three times with ethanol to remove the residual chloride and the ammonium ions to produce a white solid. The yield of the reaction was around 90% which means that if 1 mol of  $\text{CaCl}_2$  reacts with 2 mol of  $\text{NH}_4\text{F}$ , it can only produce 0.9 mol of  $\text{CaF}_2$ . It can be assumed that some materials were lost during the centrifugation process because the particles are very small and the rotational speed of the centrifuge was limited to 10,000 rpm. (Wang *et al.*, 2007). The calcium fluoride prepared in this experiment was in crystal form unlike the calcium fluoride found in the lab which is in powder form. Calcium fluoride in the form of crystal will be better than calcium fluoride in the form of powder.

### 3.4.3 Grease Formulation Using Different Ratios of Sludge to Fumed Silica

25 grams of silicone oil were heated until  $80^\circ\text{C}$  to  $100^\circ\text{C}$ . Fumed silica and sludge were added according to the ratio given in Table 3.1. For the first experiment, the ratio used was the first ratio which was 0 grams of fumed silica and 25.0 grams of sludge. The mixture was heated and stirred at a temperature more than  $100^\circ\text{C}$  for one hour. After that, molybdenum disulfide and calcium fluoride were added and heated at a temperature between  $90^\circ\text{C}$  to  $100^\circ\text{C}$  while stirring continuously for 1 hour. The grease formulated was cooled to room temperature before keeping it in a suitable container.

The steps above were repeated but with ratio 2, 3 until ratio 11. From the grease formulated, dropping point analysis was conducted to determine the dropping point of each grease.

**Table 3.1:** Ratio of fumed silica to sludge in grease formulation

No	Fumed Silica		Sludge	
	Wt %	g	Wt %	g
1	0	0	0	25.0
2	10	2.5	10	22.50
3	20	5.0	20	20.0
4	30	7.5	30	17.5
5	40	10.0	40	15.0
6	50	12.5	50	12.5
7	60	15.0	60	10.0
8	70	17.5	70	7.5
9	80	20.0	80	5.0
10	90	22.5	90	2.5
11	100	25.0	0	0

The grease with the best formulation were chosen based on 2 factors which were the dropping point temperature of the grease and fumed silica and sludge ratio, which means the grease with the best dropping point (above the standard temperature for high temperature grease) with the highest sludge content or the lowest fumed silica content. The chosen grease formulation was used to formulate grease weighing 500 grams so that it can be used to run several other analyses.

### **3.4.4 Grease Formulation Using Different Mixing Time**

From the chosen grease formulation, 500 grams were divided with the weight of the grease produced. The value obtained from the division is the number of times each material used in the chosen grease needs to be multiplied to obtain a grease with a weight of 500 grams in the end. After obtaining the weight needed for each material, the similar steps used in grease formulation previously were repeated. The grease formulated was cooled to room temperature and stored in a suitable container for further analyses.

## **3.5 Grease Property Analyses**

### **3.5.1 Dropping Point Test**

Dropping point is the temperature at which the grease passes from semi-solid to liquid. It is not the maximum temperature at which a grease can be used but it is the upper temperature limit at which grease retains its structure. In this test, a cup with a hole was used. The cup was filled with grease and was heated until the grease falls through the hole. The dropping temperature is the temperature at which the drop breaks. It is an indicator of what type of grease is produced.

To run the dropping point test, all the materials and apparatus needed for this test were prepared beforehand. First of all, the cork was placed on one of the thermometers so that the tip of the thermometer bulb will be about 1/8" above the

bottom of the grease cup when the apparatus is assembled. The other cork was placed equidistant between the lower end of upper cork and the grease cup - this cork will have to be worked and cut to fit inside the test tube. The other thermometer was suspended into the oil bath so that its bulb will be about the same level as the test thermometer. The grease cup was filled with a sample of grease and was gently pressed into the cup until a small amount is extruded at the small opening. Using the metal rod, a wedge of grease was cut from the cup by revolving the cup against the rod and simultaneously pushing the rod upward. A smooth film of grease shall remain in the cup.

After assembling the apparatus, the stirrer was started and the bath was heated at a rate of 8-12°F per minute until the bath reaches a temperature approximately 30°F below the expected dropping point. The rate of heating was reduced so that the temperature in the test tube will be within 4°F or less of the bath temperature. As the temperature increases the grease will slowly protrude through the orifice of the cup. The temperature at which a drop of grease falls to the bottom of the cup is the dropping point. If the drop has a tailing thread, the test is continued until it breaks completely. Record all the data for each grease tested (ASTM D566 – 02, 2009).



**Figure 3. 2** Dropping point test apparatus

### 3.5.2 Penetration Test

In this test, the grease is heated to 25°C (77°F) and placed below the tip of the test cone, as shown in Figure 2.2



**Figure 3.3** Standard Penetrometer Cone

The cone is allowed to drop into the grease; the amount of penetration is measured by the penetrometer in tenths of a millimeter. The greater the penetration, the thinner the grease consistency. The test is often repeated after “working” the grease to replicate the effects of mechanical shearing. In fact, worked penetration of new greases is the property upon which the NLGI (National Lubricating Grease Institute) grease consistency classification system is based, as shown in Table 2.1.

Cone penetration using ASTM D217 requires a large volume of sample and is not normally performed on used grease samples. An alternative method, ASTM D1403 uses  $\frac{1}{4}$  or  $\frac{1}{2}$  the sample volume of ASTM D217 making it more amenable to used grease analysis (Herguth, 2002).

### 3.5.3 Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. FTIR technique has made dispersive infrared spectrometers all but obsolete (except sometimes in the near infrared) and opened up new applications of infrared spectroscopy (Griffiths *et.al.*, 2007).

FTIR is most useful for identifying chemicals that are either organic or inorganic. It can be utilized to quantitate some components of an unknown mixture. It can be applied to the analysis of solids, liquids, and gasses. FTIR can be used to identify chemicals from spills, paints, polymers, coatings, drugs, and contaminants. FTIR is perhaps the most powerful tool for identifying types of chemical bonds (functional groups). The wavelength of light absorbed is characteristic of the chemical bond as can be seen in this annotated spectrum. By interpreting the infrared absorption spectrum, the chemical bonds in a molecule can be determined. FTIR spectra of pure compounds are generally so unique that they are like a molecular "fingerprint". While organic compounds have very rich, detailed spectra, inorganic compounds are usually much

simpler. For most common materials, the spectrum of an unknown can be identified by comparison to a library of known compounds. We have several infrared spectral libraries including on-line computer libraries. To identify less common materials, IR will need to be combined with nuclear magnetic resonance, mass spectrometry, emission spectroscopy, X-ray diffraction, and/or other techniques (Exova, 2010).

In this experiment, the grease sample produced can be readily used. It does not need to undergo extraction or dilution. The procedure on how to use the FTIR has already been briefed by the analytical lab owner and the FTIR manual is readily available in the lab for reference. After analyses by FTIR, the result would then be compared with the wavelengths of organic and inorganic material to find out what component is in the grease.

#### **3.5.4 Atomic Absorption Spectroscopy**

“In analytical chemistry, atomic absorption spectroscopy is a technique used to determine the concentration of a specific metal element in a sample as described by Sperling *et. al*, (1999).” Atomic-absorption spectroscopy (AAS) uses the absorption of light to measure the concentration of gas-phase atoms. Since samples are usually liquids or solids, the analyte atoms or ions must be vaporized in a flame or graphite furnace. The atoms absorb ultraviolet or visible light and make transitions to higher electronic energy levels. The analyte concentration is determined from the amount of absorption. Applying the Beer-Lambert law directly in AAS is difficult due to variations in the atomization efficiency from the sample matrix, and nonuniformity of concentration and path length of analyte atoms (in graphite furnace AAS).



Concentration measurements are usually determined from a working curve after calibrating the instrument with standards of known concentration.

To make sure this research was conducted with minimum errors; the experiment was done following a well planned sequence;

- i) Preparation of the samples and apparatus
- ii) Standard calibration curve
- iii) Sample analysis

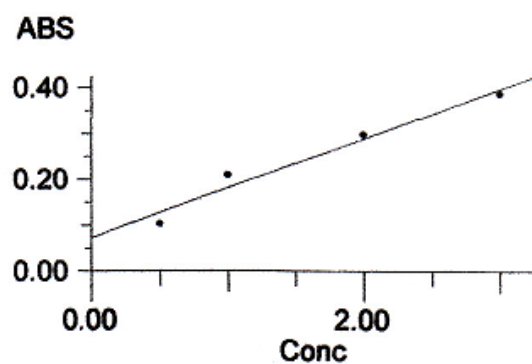
#### **3.5.4.1 Preparation of the Samples and Apparatus**

The metal elements tested in these analyses were copper, nickel, cadmium, chromium, lead and zinc. The standard solutions for all these elements were prepared. The main sample grease was extracted from its components using a microwave extractor. After that, the grease was diluted with ultra pure water and acidified with nitric acid before it was ready to be used in the AAS.

#### **3.4.4.2 Standard Calibration Curve**

The standard calibration curve for each metal element was obtained through the AAS before analyzing the sample. Figure 3.1 shows the standard calibration curve for

the standard zinc solution at concentration of 0.5, 1.0, 2.0 and 3.0 ppm versus absorbance.



**Figure 3. 4** Standard calibration curve for zinc

#### 3.4.4.3 Sample Analyses

After testing the standard sample to obtain a calibration curve, the grease sample was analysed. From the data obtained, a comparison can be made between the calibration curve and the grease sample data to determine the type and the concentration of metal element present in the grease sample.

### 3.4.5 Copper Corrosion Test

This test method measures the tendency of lubricating grease to corrode copper under specific static conditions. It can help in predicting possible chemical attack on lubricating parts such as bearings containing copper or copper alloys. Such corrosion can cause premature bearing failures. However, no corrosion with actual field service has been established. The test does not measure either the ability of the lubricant to inhibit copper corrosion caused by factors other than the lubricant itself nor does it measure the stability of the grease in the presence of copper.

A prepared copper strip is totally immersed in a sample of grease and heated in an oven or liquid bath at a specific temperature for a definite period of time. Commonly used conditions are 100°C for 24 hours. At the end of this heating period, the strip is removed, washed, and compared with the ASTM copper strip corrosion standards. Due to the nature of this test, the precision of this test method was not obtained in accordance with standard ASTM protocol. The test has no bias (Nadkarni, 2007). The ASTM copper strip classification is in Table 3.2.

**Table 3. 2:** ASTM Copper Strip Classification (Andersen *et al.*, 2003 )

Classification	Designation	Description
Freshly polished strip	-	-
1	Slight tarnish	a. Light orange, almost the same as a freshly polished strip b. Dark orange
2	Moderate tarnish	a. Claret red b. B. lavender c. C. multicoloured with lavender blue or

		silver, or both, overlaid on claret red d. Silvery e. Brassy or gold
3	Dark tarnish	a. Magenta overcast on brassy strip b. Multicoloured with red and green showing (peacock), but no gray
4	Corrosion	a. Transparent black, dark gray or brown with peacock green barely showing b. Graphite black or lusterless black c. Glossy or jet black

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Overview**

In this work, there are two parameters considered in the making of the grease as mentioned in the scope of study. The first parameter is the ratio of fumed silica to sludge and the second parameter is the mixing time of the grease. Both of the parameters mentioned were used to produce grease with different characteristics. For the first parameter, only dropping point test was conducted to analyse the grease. Based on the first parameter, the grease formulation with the best dropping point was chosen before proceeding with the second parameter. As for the second parameter, the analyses carried out were the dropping point test, penetration test, copper strip corrosion test, Fourier Transform Infrared Spectroscopy test and the Atomic Absorption Spectroscopy test. All the analyses done are further discussed in this chapter.

## **4.2 Effects of Fumed Silica to Sludge Ratio on Grease Characteristics**

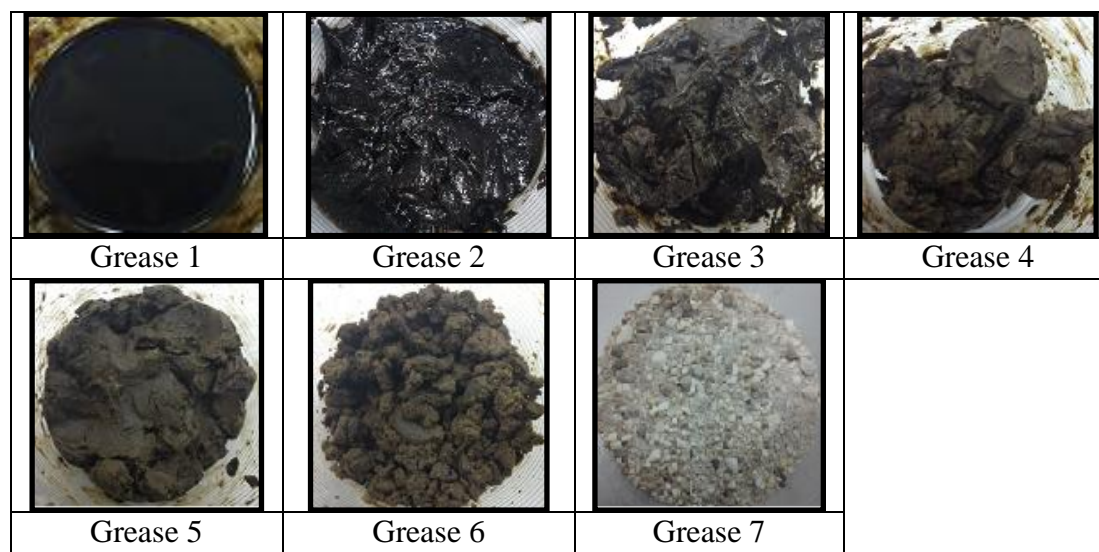
### **4.2.1 Physical Appearance Analysis**

Table 4.1 describes the physical appearance for grease 1 to grease 7 while Figure 4.1 shows the physical appearance for each of the said grease. Normally, the colour of grease is determined by the type of chemicals used. In this work, waste lubricant sludge and molybdenum disulfide was used and therefore they had contributed to the grease's dark colour. The application of grease depends heavily on the type of chemicals used and other characteristics such as the dropping point and the worked penetration and less on its physical appearance.

High temperature grease are mainly used in high temperature bearings, kiln car bearings, pellet mills, oven conveyors, lime kilns, exhaust fans, soot blower, brick/ceramic kilns, plastics, asphalt plants, construction, agricultural and mining equipment, chassis and other similar applications (Lubrication Engineers, 2010). The lubricant choice for a specific application is determined by matching the machinery design and operating conditions with desired lubricant characteristics (Engineering Edge, 2010). From Table 4.1, the specific application cannot be determined solely from the physical appearance. Other characteristics such as the dropping point and the worked penetration has to be determined before deciding the suitable application.

**Table 4. 1:** Physical Appearance Analysis

No	Sample	Description	Appearance
1	Grease 1	Semi fluid	Shiny, black liquid-like
2	Grease 2	Very soft	Shiny, black
3	Grease 3	Soft	Shiny, black brown
4	Grease 4	Semi firm	Not shiny, like hardened mud, dark brown
5	Grease 5	Firm	Not shiny, like hardened mud, dark brown
6	Grease 6	Very firm	Lumpy, brittle
7	Grease 7	Sandy	White, greyish brown like sand

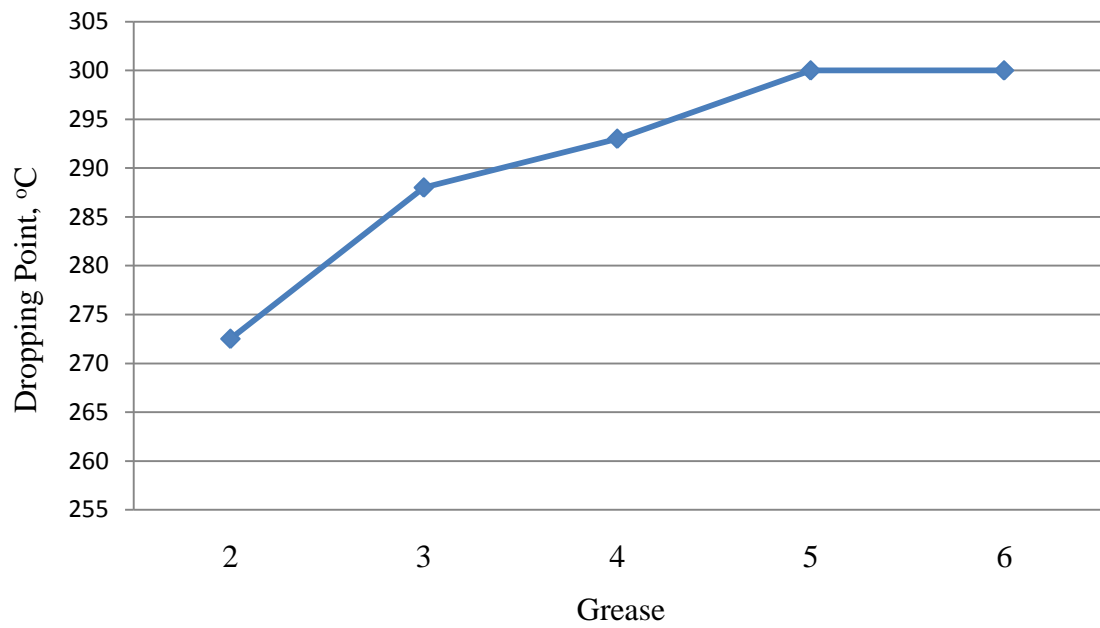
**Figure 4. 1** Physical appearance of the greases formulated

### 4.2.2 Dropping Point

Figure 4.2 shows that the dropping point increases from grease 2 to grease 6. From grease sample 1 to 7, the composition of fumed silica increases while the composition of sludge decreases. Due to the mechanical stability (Jiang Ding and Wunder, 2010), thickening property, combined with the non melting characteristic of the fumed silica, the dropping point of the grease may have been affected (Machinery Lubrication, 2002). Besides that, fumed silica has a dropping point of more than 1500°C which may also have contributed to the grease's high dropping point. This means that, the higher amount of fumed silica in the grease, the higher the dropping point. As explained in Chapter 1, sludge was used to replace fumed silica as sludge cost much lower than fumed silica. Although, sludge has a thickening property but it is not as efficient as fumed silica. Thus, this explains the trend of dropping point in the figure below.

The dropping point for grease 5 and 6 is higher than 300°C and due to the constraint of the thermal oil (liquid bath for dropping point test) which has a boiling point of 320°C, the test cannot be run higher than a temperature of 300°C. So, the exact dropping point for grease 5 and 6 cannot be determined. The dropping point for grease 1 and 7 were not being tested as the physical condition of both of these greases was unsuitable for the testing.





**Figure 4. 2** Graph of dropping point of grease versus the type of grease

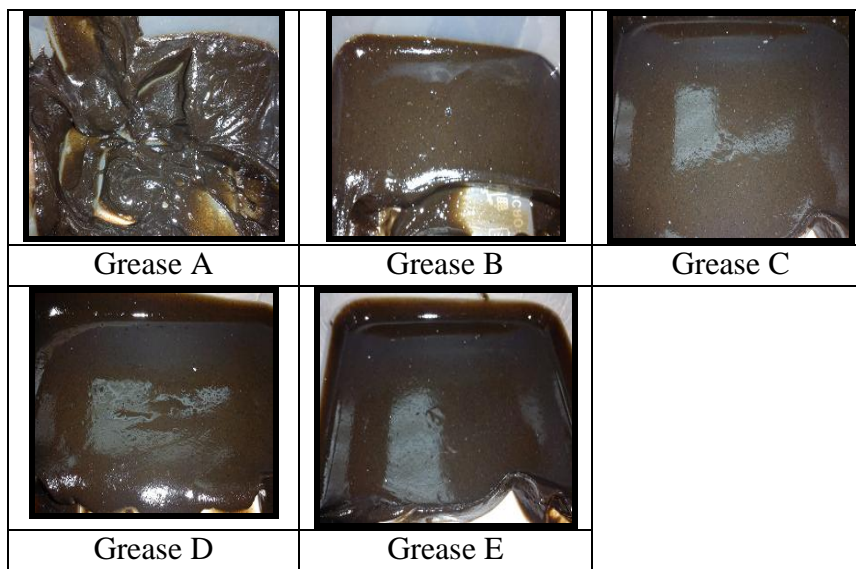
### 4.3 Effect of Mixing Time on Grease Characteristics

#### 4.3.1 Physical Appearance Analysis

From Table 4.2 and Figure 4.3, it can be seen that the mixing time does not affect the physical appearance of the grease. Grease A, B, C, D and E has the same appearance which is shiny, dark-brown-black grease. The penetration value of the grease increases with the mixing time. This means that, as the mixing time goes from grease A to grease E, the penetration increases gradually but the physical appearance is not affected (Abul Bari *et al.*, 2008).

**Table 4. 2:** Physical Appearance Analysis

No	Sample	Mixing Time (hours)	Food Analogy	Appearance
1	Grease A	1	Peanut Butter	Shiny, dark brown-black
2	Grease B	2	Peanut Butter	Shiny, dark brown-black
3	Grease C	3	Peanut Butter	Shiny, dark brown-black
4	Grease D	4	Peanut Butter	Shiny, dark brown-black
5	Grease E	5	Peanut Butter	Shiny, dark brown-black



**Figure 4.3** Physical appearance of the greases formulated

### 4.3.2 Dropping Point

From Table 4.3, it can be seen that the dropping point for grease 1 is 237.5°C while the dropping point for the other greases were more than 300°C. Technically, the dropping point for grease A should be more than 272.5°C as this was the temperature obtained in the first parameter for greases with 10:90 ratios between fumed silica and sludge and a mixing time of 1 hour. However, in this study, results show that the grease A with the same ratio and mixing time as in the first parameter has a dropping point difference of 35°C. The difference in the dropping point may have been caused by human error such as the sudden rise in the thermal oil bath temperature for the dropping point test or the uneven mixing during the production of grease.

Table 4.3 also shows that the dropping point for grease B, C, D and E is higher than 300°C and due to the constraint of the thermal oil (liquid bath for dropping point test) which has a boiling point of 320°C, the test cannot be run higher than a temperature of 300°C. So, the exact dropping point for these greases cannot be determined. From this analysis, grease A, B, C, D and E are proven to be high temperature greases.

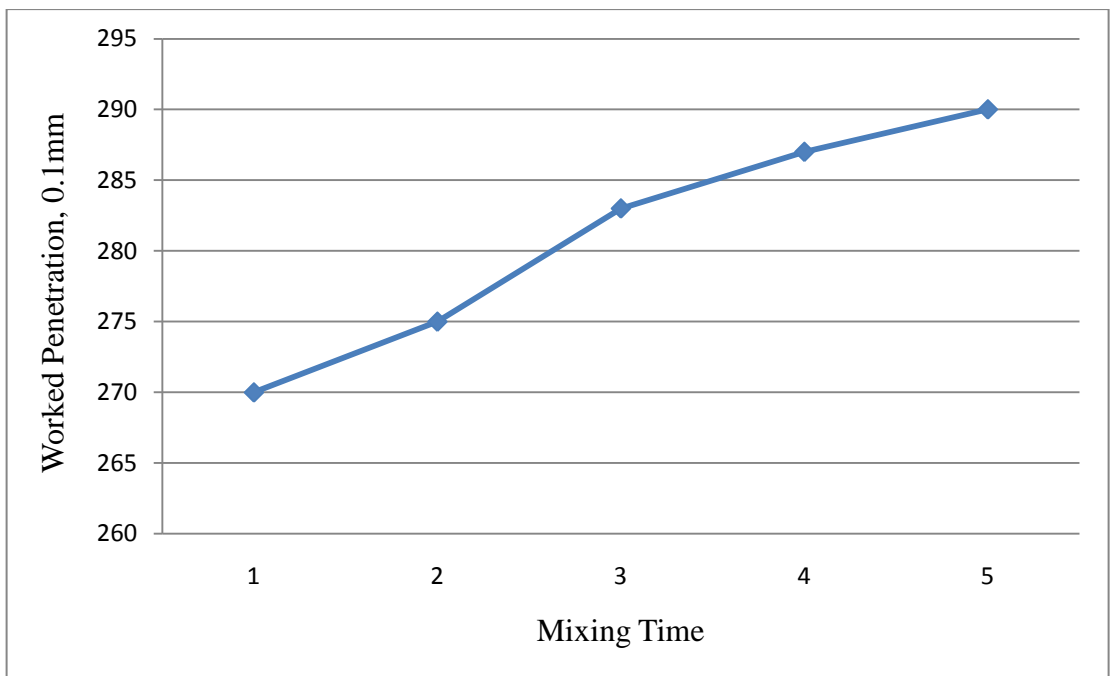
**Table 4. 3:** Dropping point of grease

No	Sample	Mixing Time	Dropping Point (°C)
1	Grease A	1	237.5
2	Grease B	2	>300°C
3	Grease C	3	>300°C
4	Grease D	4	>300°C
5	Grease E	5	>300°C

### 4.3.3 Penetration Test

From Figure 4.4, it is observed that the worked penetration of the grease increases with the mixing time. This is because the longer the grease is heated the lower the consistency of the grease. Thus, this will cause the worked penetration of the grease to increase with the mixing point.

The penetration value of the grease increases with the mixing time. This means that, as the mixing time goes from grease A to grease E, the penetration increases gradually but the physical appearance is not affected (Abul Bari *et al.*, 2008). “In the article ‘Fume Silica Base Grease’ by Abul Bari *et al.*, (2008) they stated that at 10% addition of fume silica, the penetration value increases gradually with the mixing time and the penetration reaches the maximum value at 1.5 hours.”












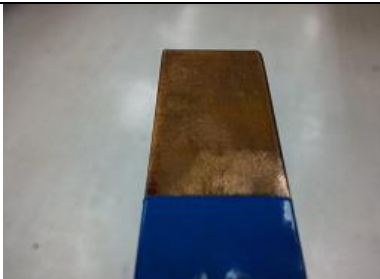
**Figure 4. 4** Graph of worked penetration versus the type of grease

#### 4.3.4 Copper Strip Corrosion Test

The copper strip corrosion tests that were performed in this research indicate that the tendency of the grease samples to corrode under specific static conditions was low. This can be seen from the results in Figure 4.5. Figure 4.5 shows that the copper strip before and after the corrosion strip differs very slightly in colour. According to the ASTM Copper Strip Classification, the copper strip immersed in greases A, B, C, D, and E is only slightly tarnished and can be classified under 1b which is a passing ASTM rating as shown in Table 4.4. This means that the greases produced in this study can be used in its suitable application as the test indicates that the presence of certain corrosive substances which may corrode equipment, such as acidic compounds or sulfur compounds is not present or is in a very low concentration.

**Table 4. 4:** Copper strip classification

<b>No</b>	<b>Sample</b>	<b>ASTM Rating</b>	<b>Result</b>
1	Grease A	1b	Pass
2	Grease B	1b	Pass
3	Grease C	1b	Pass
4	Grease D	1b	Pass
5	Grease E	1b	Pass

No	Sample	Before	After
1	Grease 1		
2	Grease 2		
3	Grease 3		
4	Grease 4		
5	Grease 5		

**Figure 4. 5** Comparison of copper strip before and after corrosion

### 4.3.5 Fourier Transform Infrared Spectroscopy

In order to identify chemicals that are either organic or inorganic in the grease, Fourier Transform Infrared Spectroscopy was used in the present study. It can be utilized to quantitate some components of an unknown mixture. The FTIR spectrum for grease A, B, C, D and E is almost the same. This is because; the chemicals used in each of the grease are the same, only the amount varies. Therefore, only one FTIR spectra will be discussed in this chapter. Figure 4.6 shows the FTIR spectra of grease A.

The absorption peak at  $2972.02\text{ cm}^{-1}$  can be assigned to the symmetric stretching vibration of the  $\text{CH}_3$  groups of the grease. The absorption peak due to silica is located at  $1152.48\text{ cm}^{-1}$  while the absorption peak at  $3764.07\text{ cm}^{-1}$  can be assigned to isopropanol and potassium hydroxide. The peak from the range of  $400\text{ cm}^{-1}$  to  $500\text{ cm}^{-1}$  is due to the presence of halogens in the grease. This halogen can be attributed to the fluoride in the additive calcium fluoride (Socrates, 1994). These characteristic absorptions proof the presence of hexane, isopropanol, potassium hydroxide, silicone oil, molybdenum disulfide and calcium fluoride.



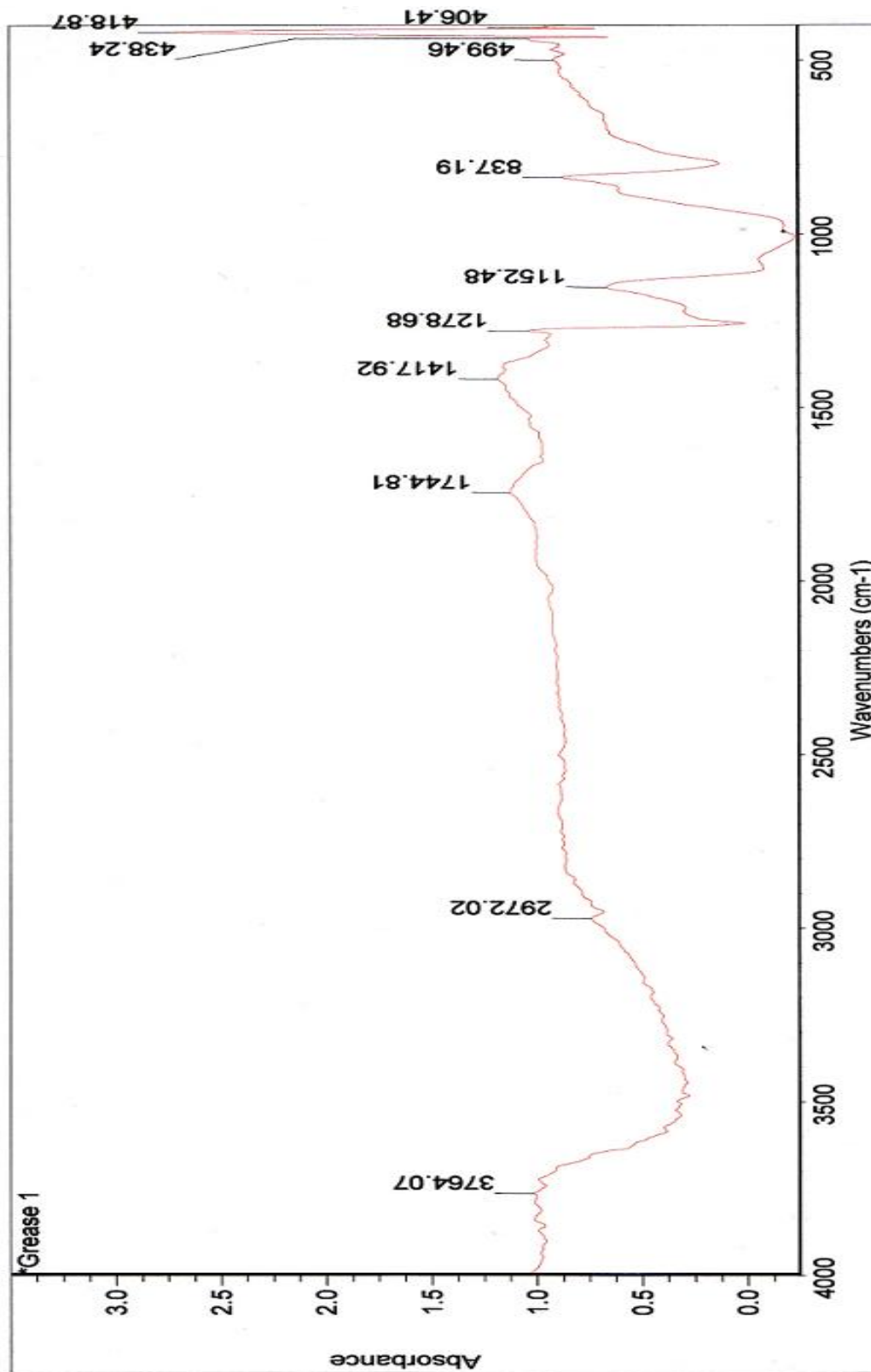
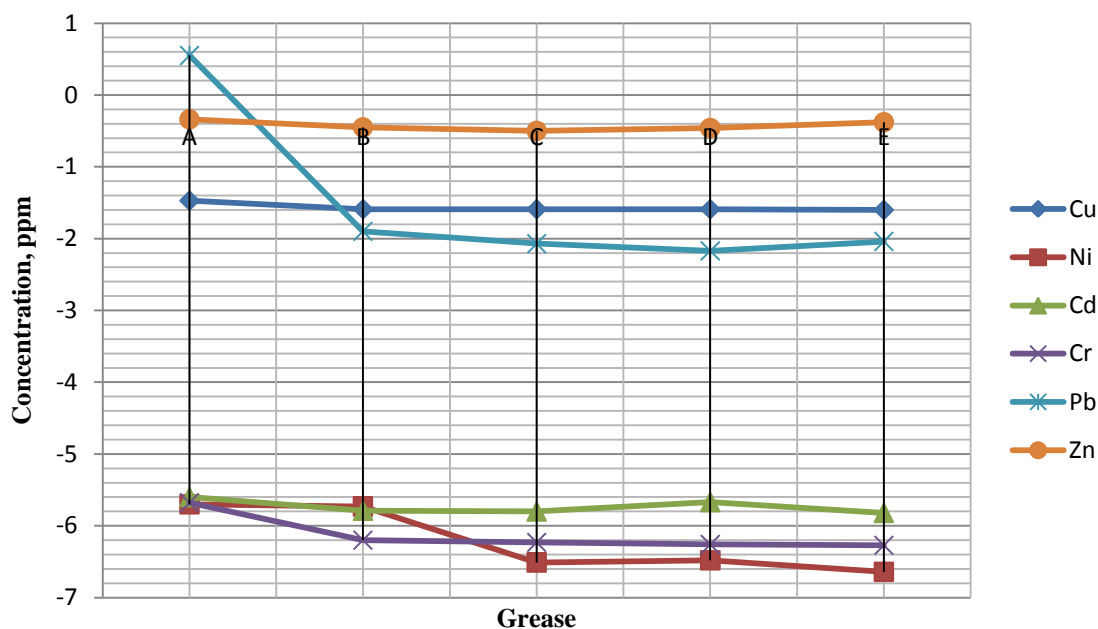


Figure 4.6 FTIR spectra of grease A

### 4.3.6 Atomic Absorption Spectroscopy

The metal elements tested in these analyses were copper, nickel, cadmium, chromium, lead and zinc. From the calibration curve obtained, the AAS was able to make a comparison between the calibration curve and the grease sample data to determine the type of metal element present in the grease sample.

Figure 4.7 shows the concentration for the type of metal element present in the grease sample. Most of the values obtained were of negative value. This is because, the concentration of the standard solutions prepared for the calibration curve were at from range 0.5 ppm to 100 ppm. Thus, any concentration of metal element lower than the standard solution concentration cannot be read accurately. From the graph, we can see that the presence of the copper, nickel, cadmium, chromium, iron and zinc is very low in grease sample A, B, C, D and E.



**Figure 4.7** Graph of concentration of metal element, ppm versus grease

## **CHAPTER 5**

### **CONCLUSION & RECOMMENDATION**

#### **5.1 Conclusion**

This study on the title of “Production of High Temperature Grease from Waste Lubricant Sludge and Silicone Oil” has been successfully done. From the result obtained and analysis that have been done, it can be concluded that the purpose of this study has been achieved. This study is to produce high temperature grease from waste lubricant sludge and silicone oil.

The production of high temperature grease from silicone oil and waste lubricant sludge contributes to the community and the environment in more than one way. For starters, this research will be a stepping stone for other researches who will be doing researches similar to this and who intend to curb pollution. It will be a guide for them to what's to do and what's not to do in the future.

In the aspect of the society, this research will protect them from more toxic pollution of waste lubricant sludge. The protection of the environment is crucial as the cycle of life depends on it. Thus through this research, it will not only tackle the pollution problem but will also lower the chances of wildlife being exposed to these dangerous substances. It is hoped that the concept of 'zero waste' and 'waste to wealth' can be cultivated in our society for the betterment of the future.

The results of this study indicated that it is possible to produce high temperature grease from waste lubricant sludge and silicone oil. Factors such as the ratios of waste lubricant sludge to silicone oil and mixing time will have a significant effect on the grease's dropping point and worked penetration value. The study shows that the dropping point of the grease increases with the increase in the amount of fumed silica added into the grease. The physical appearance of the grease also changes from semi-fluid to sandy. Thus, from the first parameter grease formulation, grease 2 with the ratio of 10:90 (fumed silica: sludge) having a dropping point of 272.5 °C was chosen to be used in the parameter 2 grease formulation.

From the second parameter results, analysis shows that grease with higher mixing time will have higher worked penetration value but the physical appearance is not affected. The copper strip corrosion tests that were performed in this research indicate that the tendency of the grease samples to corrode under specific static conditions was low. This means that the greases produced in this study can be used in its suitable application as the test indicates that the presence of certain corrosive substances which may corrode equipment, such as acidic compounds or sulfur compounds is not present or is in a very low concentration. FTIR analysis shows that the compound in the grease produced were the compounds used to produce the grease. The AAS analysis that was done verified that the presence of metal element copper, cadmium, chromium, iron and zinc is very low in the greases produced. Overall, these

results indicate that high temperature grease can be produced from waste lubricant sludge and silicone oil.

## **5.2 Recommendation**

The production of high temperature grease from waste lubricant sludge and silicone has proven to be feasible. However, more tests need to be conducted in order to determine its working performance. Tests such as the oxidation stability test, bleeding test, wear and extreme pressure properties test and other related tests should be performed in order to further characterize and determine its specific application. It is also discovered that the viscometer in the lab is not able to read materials as viscous as grease. This can be done by sending the grease to a different university that has the mentioned analysis or to an established grease analysis lab.

For the AAS analysis, a lower concentration range for the standard solution should be prepared so that the exact concentration of the metal element in the grease could be specifically determined. Instead of using a range from 0.5 to 100 ppm, a range from 0.01 to 1.0 ppm should be used instead.

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**APPENDIX A**

**ANALYSIS RESULTS**



## Dropping point of grease (Parameter 1)

No	Sample	Composition (g)		Dropping Point (°C)
		Fumed Silica	Sludge	
1	Grease 1	0	25.0	-
2	Grease 2	2.5	22.5	272.5
3	Grease 3	5.0	20.0	288
4	Grease 4	7.5	17.5	293
5	Grease 5	10.0	15.0	>300
6	Grease 6	12.5	12.5	>300
7	Grease 7	15.0	10.0	-

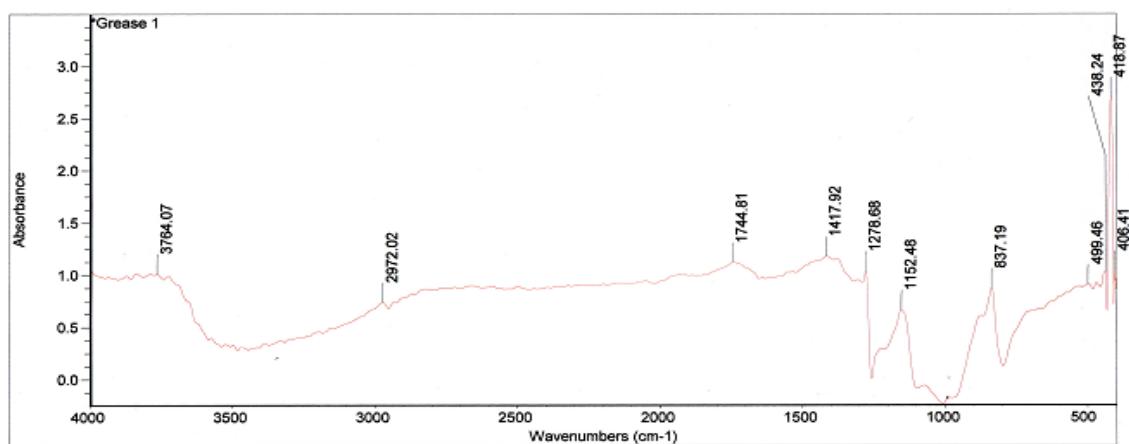
## Worked penetration (Parameter 2)

No	Mixing Time	ASTM Worked Penetration 0.1mm at 25°C
1	1	270
2	2	275
3	3	283
4	4	287
5	5	290

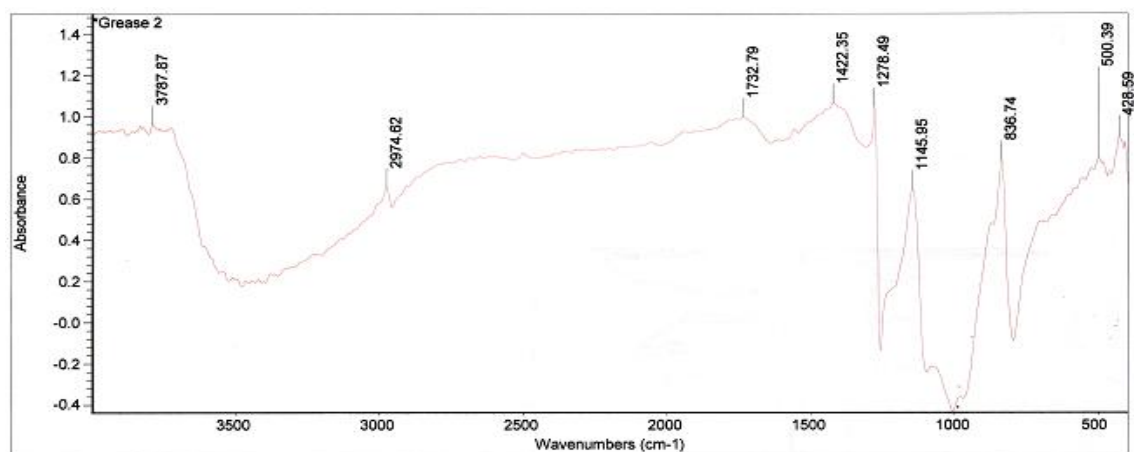
## Concentration of metal element of grease (Parameter 2)

Sample/Metal	Copper	Nickel	Cadmium	Chromium	Lead	Zinc
<b>A</b>	-1.47	26.86	-5.60	-5.68	0.55	-0.34
<b>B</b>	-1.59	-5.73	-5.79	-6.20	-1.90	-0.45
<b>C</b>	-1.59	-6.51	-5.80	-6.23	-2.07	-0.50
<b>D</b>	-1.59	-6.48	-5.67	-6.26	-2.17	-0.46
<b>E</b>	-1.60	-6.64	-5.82	-6.27	-2.04	-0.38

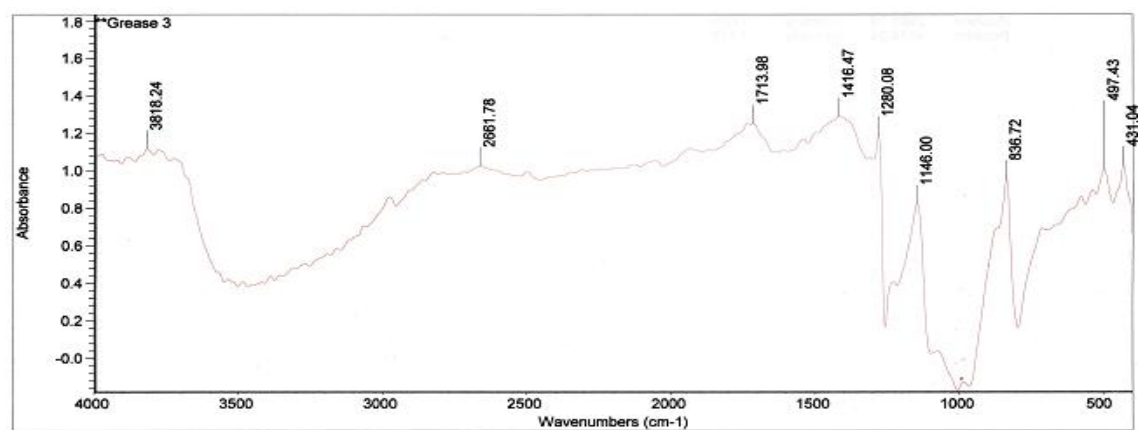
## FTIR Analysis



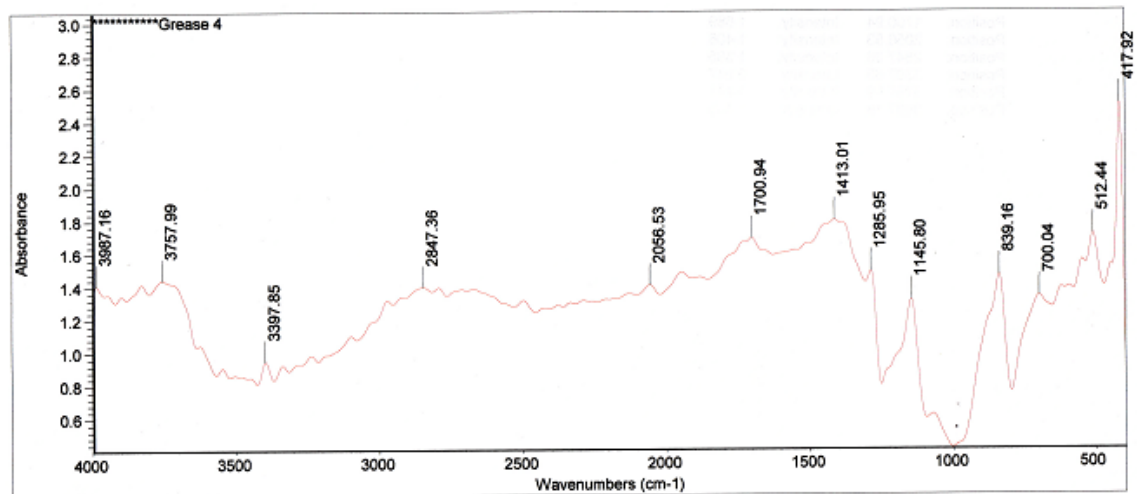
Grease A



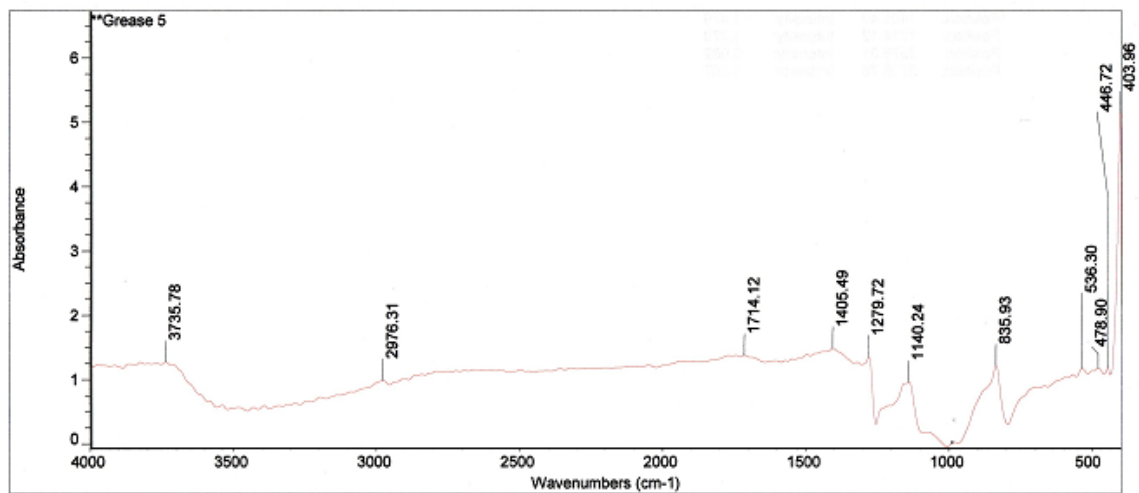
Grease B



Grease C



Grease D



Grease E

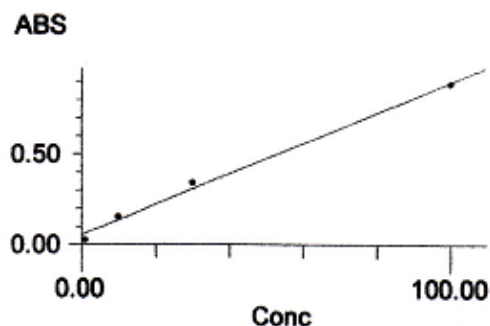
## AAS Analysis

Meas. Date : 11/2/2010 4:27 PM  
 Element : Cr STD Unit :

UNK Unit :

Sample ID	Sample Name	Conc.	RSD(%)	ABS	REF
STD1		1.00	0.00	0.0232	-0.0081
STD2		10.00	0.00	0.1555	-0.0079
STD3		30.00	0.00	0.3379	-0.0055
STD4		100.00	0.00	0.8850	0.0072

Coefficient : K3=---  
 K2=---  
 K1=8.424955E-003  
 K0=5.342032E-002  
 Corr.Coef. : 0.9966



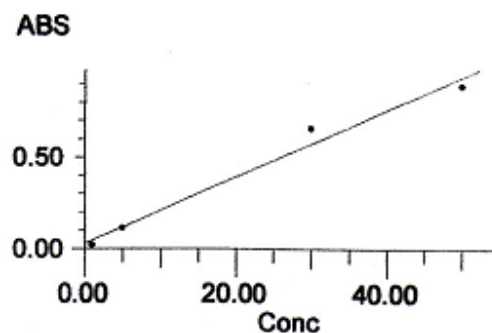
UNK-001	G1	-5.68	0.00	0.0056	-0.0177
UNK-002	G2	-6.20	0.00	0.0012	-0.0187
UNK-003	G3	-6.23	0.00	0.0009	-0.0201
UNK-004	G4	-6.26	0.00	0.0007	-0.0211
UNK-005	G5	-6.27	0.00	0.0006	-0.0220

Meas. Date : 11/2/2010 4:39 PM  
 Element : Cu STD Unit :

UNK Unit :

Sample ID	Sample Name	Conc.	RSD(%)	ABS	REF
STD1		1.00	0.00	0.0192	0.0110
STD2		5.00	0.00	0.1127	0.0884
STD3		30.00	0.00	0.6578	0.4954
STD4		50.00	0.00	0.8894	0.7194

Coefficient : K3=---  
 K2=---  
 K1=1.819027E-002  
 K0=2.868428E-002  
 Corr.Coef. : 0.9904



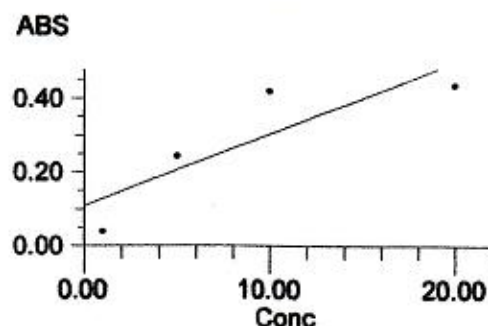
UNK-001	G1	-1.47	0.00	0.0019	-0.0173
UNK-002	G2	-1.59	0.00	-0.0003	-0.0204
UNK-003	G3	-1.59	0.00	-0.0002	-0.0219
UNK-004	G4	-1.59	0.00	-0.0003	-0.0226
UNK-005	G5	-1.60	0.00	-0.0004	-0.0233

Meas. Date : 11/2/2010 12:51 PM  
 Element : Cd STD Unit :

UNK Unit :

Sample ID	Sample Name	Conc.	RSD(%)	ABS	REF
STD1		1.00	0.00	0.0387	0.0066
STD2		5.00	0.00	0.2426	0.0482
STD3		10.00	0.00	0.4182	0.0981
STD4		20.00	0.00	0.4343	0.1162

Coefficient : K3=  
 K2=  
 K1=1.938366E-002  
 K0=1.089970E-001  
 Corr.Coeff. : 0.8606



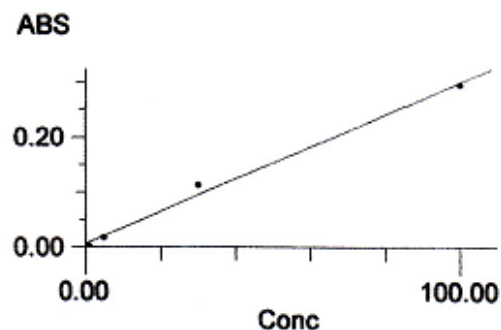
UNK-001	G1	-5.60	0.00	0.0005	0.0012
UNK-002	G2	-5.79	0.00	-0.0032	0.0010
UNK-003	G3	-5.80	0.00	-0.0034	0.0009
UNK-004	G4	-5.67	0.00	-0.0009	0.0013
UNK-005	G5	-5.82	0.00	-0.0039	0.0010

Meas. Date : 11/2/2010 1:12 PM  
 Element : Pb STD Unit :

UNK Unit :

Sample ID	Sample Name	Conc.	RSD(%)	ABS	REF
STD1		1.00	0.00	0.0004	-0.0099
STD2		5.00	0.00	0.0170	-0.0115
STD3		30.00	0.00	0.1111	-0.0043
STD4		100.00	0.00	0.2948	0.0156

Coefficient : K3=  
 K2=  
 K1=2.936560E-003  
 K0=5.981966E-003  
 Corr.Coeff. : 0.9963



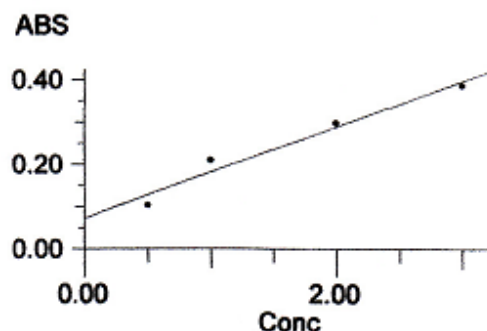
UNK-001	G1	0.55	0.00	0.0076	-0.0199
UNK-002	G2	-1.90	0.00	0.0004	-0.0236
UNK-003	G3	-2.07	0.00	-0.0001	-0.0272
UNK-004	G4	-2.17	0.00	-0.0004	-0.0298
UNK-005	G5	-2.04	0.00	0.0000	-0.0327

Meas. Date : 11/2/2010 1:58 PM  
 Element : Zn STD Unit :

UNK Unit :

Sample ID	Sample Name	Conc.	RSD(%)	ABS	REF
STD1		0.50	0.00	0.1027	0.0183
STD2		1.00	0.00	0.2080	0.0364
STD3		2.00	0.00	0.2972	0.0532
STD4		3.00	0.00	0.3863	0.0695

Coefficient : K3=  
 K2=  
 K1=1.076814E-001  
 K0=7.356780E-002  
 Corr.Coeff. : 0.9829



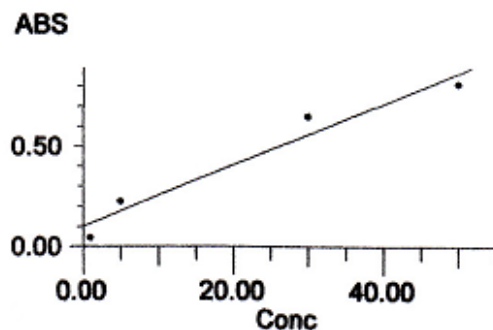
UNK-001	G1	-0.34	0.00	0.0374	0.0079
UNK-002	G2	-0.45	0.00	0.0246	0.0057
UNK-003	G3	-0.50	0.00	0.0193	0.0061
UNK-004	G4	-0.46	0.00	0.0245	0.0060
UNK-005	G5	-0.38	0.00	0.0330	0.0073

Meas. Date : 11/2/2010 12:09 PM  
 Element : Ni STD Unit :

UNK Unit :

Sample ID	Sample Name	Conc.	RSD(%)	ABS	REF
STD1		1.00	0.00	0.0413	0.0543
STD2		5.00	0.00	0.2234	0.0697
STD3		30.00	0.00	0.6445	0.1207
STD4		50.00	0.00	0.8094	0.1904

Coefficient : K3=  
 K2=  
 K1=1.522727E-002  
 K0=1.022638E-001  
 Corr.Coeff. : 0.9762



UNK-001	G1	26.86	0.00	0.5112	0.0979
UNK-002	G2	-5.73	0.00	0.0150	0.0531
UNK-003	G3	-6.51	0.00	0.0032	0.0527
UNK-004	G4	-6.48	0.00	0.0036	0.0527
UNK-005		-6.64	0.00	0.0011	0.0527

**APPENDIX B**

**PICTURES OF EQUIPMENTS**



**Microwave Extractor**

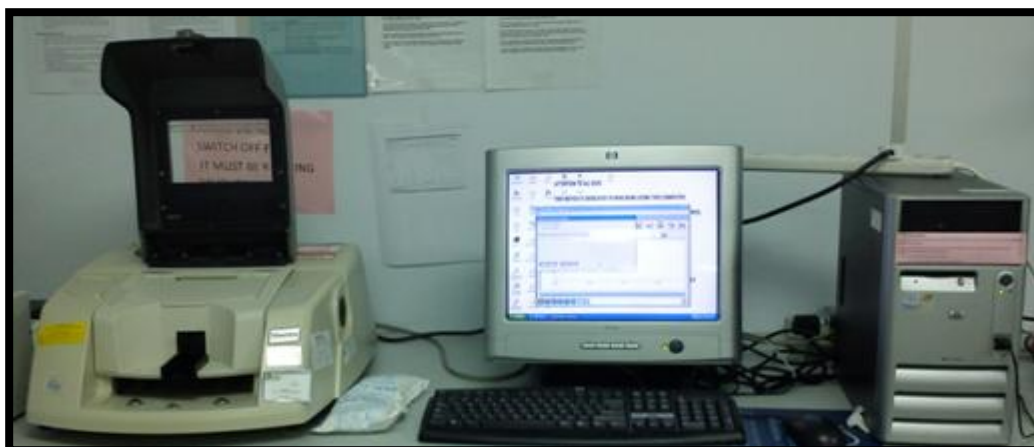


**Mechanical Stirrer and Heater**





**Thermal Oil Bath**



**Fourier Transform Infrared Spectroscopy**



**Atomic Adsorption Spectroscopy**