PRODUCTION OF GREASE FROM SPENT LUBRICANT

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PRODUCTION OF GREASE FROM SPENT LUBRICANT

AWANG OMAR FIRDAUS B AWANG ISMAIL

**A thesis submitted in fulfillment
of the requirements for the award of the degree of
Bachelor of Chemical Engineering**

**Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang**

MAY, 2009

I declare that this thesis entitled "*Production of Grease from Spent Lubricant*" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature	:………………………………
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Special Dedication of This Grateful Feeling to My…

Beloved parent;

Mr. Awang Ismail b Awang Rambli & Mrs. Dara bt Sahari

Loving brother and sisters;

Awang Morshidi and Dayang Nur Fitrah

Understanding and helpful friends;

For Their Love, Support and Best Wishes.

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ABSTRACT

This study was undertaken to examine the potential of spent lubricant as a medium to produce grease. Grease is a semi-solid colloidal dispersion of a thickener in a liquid lubricant matrix. Spent lubricant oils have significant potential as a base fluid and a substitute for mineral oil in grease formulations. The advantages of using products based on lubricant oils are their physical and chemical properties are close to that common grease production. Besides, this research allowing as optimizing the oil waste usage and reduce the environmental problem towards waste disposal. This work focuses on the preparation methods, optimization of thickener components, and antioxidant additive for producing spent lube oil based grease. In greases, Li-fatty acid thickener having C_{12} to C_{18} chain lengths and different metal to fatty acid ratios were synthesized. The percentages of thickener were varied at 5%, 10%, 15% and 20%. The research also focuses on varying the mixing temperature during the saponification of grease. Four sets of reaction temperature were 150◦C, 170◦C, 200◦C, and 230◦C. Four types of grease properties or characteristic to be tested in this research were grease oxidation stability, penetration, dynamic viscosity, and dropping point. Results indicate that Li-thickener composition, fatty acid types and base oil content and reaction temperature significantly affect grease hardness, dynamic viscosity, oxidation, and dropping point. It has been observed that the physical and chemical properties of grease are largely based on the thickener fibre micro-structure.

ABSTRAK

Kajian ini dijalankan untuk mengkaji keupayaan atau potensi minyak hitam (pelincir) terpakai sebagai medium untuk menghasilkan gris. Gris merupakan bahan separa pejal yang mengandungi serakan bahan pemekat yang dipanggil thickener. Minyak hitam terpakai mempunyai potensi yang tersendiri sebagai minyak asas bagi mengantikan penggunaan minyak mineral komersil dalam pembuatan gris. Minyak hitam terpakai mempamerkan beberapa kelebihan yang tersendiri berasaskan fizikal dan kimianya yang menghampiri sifat-sifat gris. Kajian ini memberikan peluang untuk kita mengoptimumkan penggunaan bahan terpakai sekaligus mengurangkan masalah terhadap alam sekitar. Kajian memfokus kepada metod pembuatan gris, penggunaan optimum cecair pemekat dan penggunaan bahan tambahbaikan untuk menghasilkan gris berasaskan minyak hitam terpakai. Dengan ini, peratus cecair pemekat akan dipelbagaikan iaitu 5%, 10%, 15%, dan 20% daripada keseluruhan gris. Di samping itu, kajian in juga memfokuskan kepelbagaian penggunaan suhu ketika proses saponifikasi. Untuk itu, empat set suhu akan digunapakai iaitu 150°C, 170°C, 200°C, dan 230°C. Keputusan kajian menunjukkan beberapa impak penting terhadap cirri-ciri gris berdasarkan kaedah mempelbagaikan peratus cecair pemekat serta suhu untuk proses saponifikasi. Antara cirri-ciri gris yang dikaji ialah kekerasan gris, kelikatan gris, pengoksidaaan dan suhu pencairan gris. Pemerhatian menunjukkan sifat-sifat gris banyak dipengaruhi oleh cecair pemekat yang akan membentuk mikrostuktur gris tersebut

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CP	-	Centi Poise
$NaS_2CN\ (C_2H_5)_2$	-	Sodium Diethyldithiocarbamate
LiOH	-	Lithium Hydroxide
TFMO	-	Thin Film Micro Oxidation
◦C	-	Degree Celsius
◦F	-	Degree Fahrenheit

APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Grease, as it is looked upon today, is a relatively new science. In 1400 B.C., mutton or beef fat was sometimes mixed with lime to reduce the friction in chariot wheels. Under the correct conditions, heating lime and fat in oil will form grease. Modern greases, however, were not commercially available until more than 3,300 years later. The first grease produced in volume was calcium soap grease. Lithium, barium and calcium complex greases were introduced in the 1930s and 1940s. Aluminum complex greases followed in the early 1950s, but modern lithium complex greases did not enter the market until the early 1960s. (Sandra Cowan, 2007)

Conversely, the worldwide production reports higher percentages of hydrated calcium, conventional lithium and sodium soap grease. This could be due to a difference in equipment lubrication demands in various parts of the world. In general, high-speed or heavily loaded equipment can generate more heat, which creates an increased need for greases with higher dropping points. In addition, higher labor costs in North America factor into the need to extend relubrication intervals and therefore increase the need for grease that can function for longer periods of time. (NLGI Annual Grease Survey, 2005)

As industrial growth rapidly, the grease production industries become wider. More technologies being develop to produce a high-end quality of grease. Currently, many researchers have been carried out to search alternative sources for grease production due to increasing of crude oil price in the world market, thus affecting the price of base oil as main component in grease. Instead of using commercial oil as based, other potential sources which are more economic and abundantly available are soybean oil, synthetic oil and waste lubricant.

1.2 Problem of Statement

Crude oil or mineral oil is one of the most valuable compounds in the world. Crude oil products contribute major application within industries such as automotive, machinery, building and medicine. Lubricant oil is one of the application products from crude oil. In machinery and automotive industries, the lubricant oil is applied onto the surface of moving part such as rotor and ball bearing. It also acts as a surface seal to inhibit corrosion (Cavengros, J. *et al.,* 2002). Lubricant oil has several characteristics which contribute for its long life spent inside the machine. But, after certain years, the lubricant oil will loss it characteristic and become vulnerable. This is due to contaminant or metal waste trap inside the lubricant oil. As lube oil become harder, it will scratch the performance of the machine and need to replace. Thus, this lubricant is claim as waste lubricant. Several techniques are available to treat this waste lubricant so that it can be reused in other application.

From the research by Prakash K Ramdoss *et al.*, those wastes were not manage appropriately and it just disposes through land filling and this scenario become worst. As record recently in 2002 worldwide, this number soars to about 5.3 billion gallons. Of the amount generated in the United States each year, over 400 million gallons are largely not recovered and presumed to be disposed of improperly, creating significant environmental problems. Of the approximately 900 million gallons collected, only about 140 million are reprocessed (Lundberg J. e*t al.*, 2003). Recently, vast researches of grease production claim to use vegetable oil such as waste cooking oil or soybean oil to prepare the grease (Brajendra K. Sharma *et al.*, 2006). However, there are no specific researches on the grease production that use spent lubricant as the base oil component of the grease. It is due to the high viscosity of the lubricant itself, thus it is hard to handle. Since the lubricant base is come from mineral oil, it is not impossible to make grease from spent lubricant with a high quality and economically viable.

1.3 Objectives

To produce high quality grease from spent lubricant and comparing the quality with commercial grease.

1.4 Scope of Research

In attempt to understand the characteristic of lubricating grease, there are some influence factors that must be study to ensure the grease meet the quality of industrial standard and long life performance. In order to achieve the objectives, several scopes has been identified.

i. To investigate the effect of thickener percentage composition towards the oxidation stability of the grease. Thickener compositions were varied 5%, 10%, 15%, and 20% w/w.
ii. To study the effect of thickener towards consistency of grease
iii. To study the effect of thickener towards apparent viscosity of grease.
iv. To investigate the effect of thickener towards grease dropping point.
v. To study the effect of mixing temperature towards the formation of grease. Mixing temperatures were varied 150°C, 170°C, 200°C, and 230°C.

CHAPTER 2

LITERATURE REVIEW

2.1.1 Grease Anatomy

Grease is a semi fluid combination of a fluid lubricant, a thickener, and additives. The fluid lubricant or the based oil that performs the actual lubrication can be petroleum (mineral) oil, synthetic oil, or vegetable oil (Lundberg J *et al.,* 2003). The thickener gives grease its characteristic consistency and is sometimes thought of as a “three-dimensional fibrous network” or “sponge” that holds the oil in place (Crone I. *et al.,* 2003). Common thickeners are soaps and organic or inorganic nonsoap thickeners. The majority of greases on the market are composed of mineral oil blended with a soap thickener. Additives enhance performance and protect the grease and lubricated surfaces. Figure 2.1 shows the general composition of base oil, thickener and additive to produce grease. The composition can be manipulated to get different properties and function of grease. From the figure, it shows that the base oil can be divided into mineral oil or synthetic oil. Thickener or soap can be classified as simple soap, complex, and non soap. Moreover, the existence of additive can create new properties as well as enhance the performance of grease (Aihara C. *et al.,* 1995).

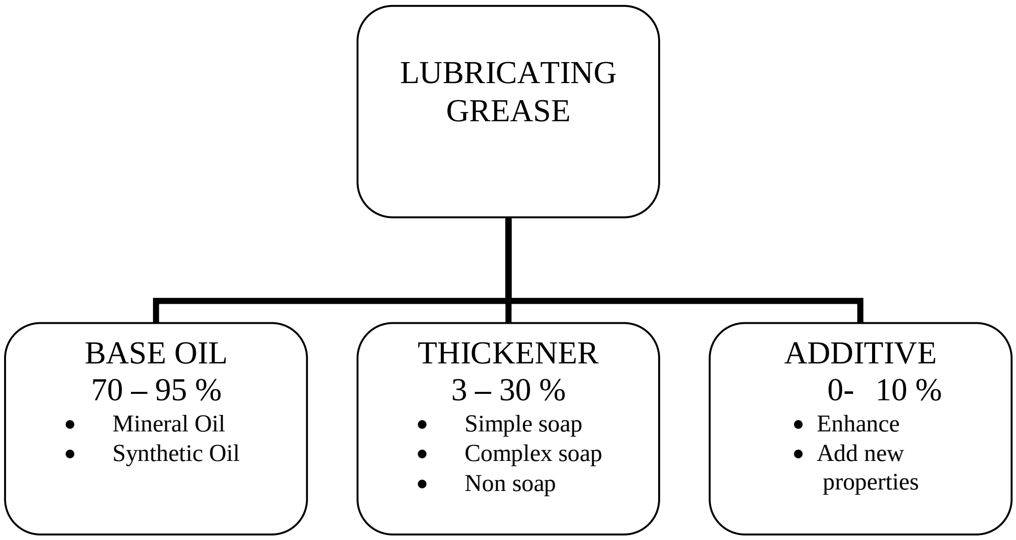

Figure 2.1 Grease Anatomies (After (Jeremy Wright, 2001))

2.2 Base Oil

Most grease produced today use mineral oil as their fluid components. These mineral oil-based greases typically provide satisfactory performance in most industrial applications. In this research spent lubricant is utilized as base oil. There are five specific categories of base oils. These categories define the type of base stock the oil is formulated from. The categories are as follows.

2.2.1 **Group I - Solvent Freezing** (M. A. Delgado *et al*., 2006)

Group 1 base oils are the least refined of all the groups. They are usually a mix of different hydrocarbon chains with little or no uniformity. While some automotive oils on the market use Group I stocks, it's generally used in less demanding applications.

2.2.2 **Group II - Hydro processing and Refining** (Mazuyer D. *et al.*, 2003)

Group II base oils are common in mineral based motor oils currently available on the market. It have fair to good performance in lubricating properties such as volatility, oxidative stability and flash/fire points. It gives fair performance in areas such as pour point, cold crank viscosity and extreme pressure wear.

2.2.3 **Group – III Hydro processing and Refining** (Chin –Hsian Kuo *et al.*, (1996)

Group III base oils are subjected to the highest level of mineral oil refining of the base oil groups. Although it is not chemically engineered, it offers good performance in a wide range of attributes as well as good molecular uniformity and stability. They are commonly mixed with additives and marketed as synthetic or semi-synthetic products. Group III base oils have become more common in America in the last decade.

2.2.4 Group IV -Chemical Reactions (Sevim Z. Erhan *et al.*, 2006)

Group IV base oils are chemically engineered synthetic base stocks. Polyalphaolefins (PAO's) are a common example of a synthetic base stock. Synthetics, when combined with additives, offer excellent performance over a wide range of lubricating properties. They have very stable chemical compositions and highly uniform molecular chains. Group IV base oils are becoming more common in synthetic and synthetic-blend products for automotive and industrial applications.

2.2.5 Group V (Giordin D. *et al.*, 2003)

Group V base oils are used mainly in the creation of oil additives. Esters and polyolesters are both common Group V base oils used in the formulation of oil additives. Group V oils are generally not used as base oils themselves, but add beneficial properties to other base oils.

Most grease is formulated using Group I and II mineral oil base stocks, which are appropriate for most applications. However, there are applications that might benefit from the use of synthetic base oil. Such applications include high or low operating temperatures, a wide ambient temperature range, or any application where extended relubrication intervals are desired.

2.3 Lubricant oil

Motor oil is a lubricant used in internal combustion engines. These include motor or road vehicles such as cars and motorcycles, heavier vehicles such as buses and commercial vehicles, non-road vehicles such as go-karts, snowmobiles, boats (fixed engine installations and outboards), lawn mowers, large agricultural and construction equipment, trains and aircraft, and static engines such as electrical generators. In engines there are parts which move very closely against each other causing friction which wastes otherwise useful power by converting the energy to heat. Contact between moving surfaces also wears away those parts, which could lead to lower efficiency and degradation of the motor. This increases fuel consumption and decreases power output and can, in extreme cases, lead to total engine failure (Cavengros, J. *et al.*, 2002).

Lubricating oil creates a separating film between surfaces of adjacent moving parts to minimize direct contact between them, decreasing friction, wear, and production of excessive heat, thus protecting the engine. Motor oil being a good conductor of heat, it is brought into contact with a hot surface, thereby absorbing some of the heat from said surface so the oil can then transfer the heat elsewhere, typically to the air or a heat sink of some variety.

Coating metal parts with oil keeps them from being exposed to oxygen, inhibiting oxidation at elevated operating temperatures preventing rust or corrosion. Corrosion inhibitors may also be added to the motor oil. Many motor oils also have detergent and dispersant additives to help keep the engine clean and minimize oil sludge build-up.

Rubbing of metal engine parts inevitably produces some microscopic metallic particles from the wearing of the surfaces. Such particles could circulate in the oil and grind against the moving parts, causing erosion and wear. Because particles inevitably build up in the oil, it is typically circulated through an oil filter to remove harmful particles. An oil pump, a vane or gear pump powered by the vehicle engine, pumps the oil throughout the engine, including the oil filter. Oil filters can be a full flow or bypass type (Cavengros, J. *et al.*, 2002).

In the crankcase of a vehicle engine, motor oil lubricates rotating or sliding surfaces between the crankshaft journal bearings (main bearings and big-end bearings), and rods connecting the pistons to the crankshaft. The oil collects in an oil pan, or sump at the bottom of the crankcase. In some small engines such as lawn mower engines, dippers on the bottoms of connecting rods dip into the oil at the bottom and splash it around the crankcase as needed to lubricate parts inside. In modern vehicle engines, the oil pump takes oil from the oil pan and sends it through the oil filter into oil galleries, from which the oil lubricates the main bearings holding the crankshaft up at the main journals and camshaft bearings operating the valves. In typical modern vehicles, oil pressure-fed from the oil galleries to the main bearings enters holes in the main journals of the crankshaft. From these holes in the main journals, the oil moves through passageways inside the crankshaft to exit holes in the rod journals to lubricate the rod bearings and connecting rods. Some simpler designs relied on these rapidly moving parts to splash and lubricate the contacting surfaces between the piston rings and interior surfaces of the cylinders. However, in modern designs, there are also passageways through the rods which carry oil from the rod bearings to the rod-piston connections and lubricate the contacting surfaces between the piston rings and interior surfaces of the cylinders. This oil film also serves as a seal between the piston rings and cylinder walls to separate the combustion chamber in the cylinder head from the crankcase.

2.4 Thickener

Thickener is also known as soap. Numerous types of grease thickeners are currently in use, each will give different properties towards the grease. The most common types are simple lithium soaps, lithium complex and polyurea (Gallegos C. *et al.*, 2005). 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 simple lithium grease might be 250°F, while that for 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. (R.L Marker, 1945) General types of thickener are discussed below:

2.4.1 Calcium Grease. (Atanu Adharvu *et al.*, 2005)

Calcium or lime grease, the first of the modern production greases, is prepared by reacting mineral oil with fats, fatty acids, a small amount of water, and calcium hydroxide (also known as hydrated lime). The water modifies the soap structure to absorb mineral oil. Because of water evaporation, calcium grease is sensitive to elevated temperatures. It dehydrates at temperatures around 79 °C (175 °F) at which its structure collapses, resulting in softening and, eventually, phase separation. Greases with soft consistencies can dehydrate at lower temperatures while greases with firm consistencies can lubricate satisfactorily to temperatures around 93 °C (200 °F). In spite of the temperature limitations, lime grease does not emulsify in water and is excellent at resisting “wash out.” Also, its manufacturing cost is relatively low. Calcium complex grease is prepared by adding the salt calcium acetate. The salt provides the grease with extreme pressure characteristics without using an additive. Dropping points greater than 260 °C (500 °F) can be obtained and the maximum usable temperature increases to approximately 177 °C (350 °F). With the exception of poor pumpability in high-pressure centralized systems, where caking and hardening sometimes occur calcium complex greases have good all-around characteristics that make them desirable multipurpose greases.

2.4.2 Sodium Grease.

Sodium grease was developed for use at higher operating temperatures than the early hydrated calcium greases. Sodium grease can be used at temperatures up to 121 °C (250 °F), but it is soluble in water and readily washes out. Sodium is sometimes mixed with other metal soaps, especially calcium, to improve water resistance. Although it has better adhesive properties than calcium grease, the use of sodium grease is declining due to its lack of versatility. It cannot compete with water-resistant, more heat-resistant multipurpose greases. It is, however, still recommended for certain heavy-duty applications and well-sealed electric motors. (R.L Merker *et al.*, 1945)

2.4.3 Aluminum Grease.

Aluminum grease is normally clear and has a somewhat stringy texture, more so when produced from high-viscosity oils. When heated above 79°C (175°F), this stringiness increases and produces a rubber like substance that pulls away from metal surfaces, reducing lubrication and increasing power consumption. Aluminum grease has good water resistance, good adhesive properties, and inhibits rust without additives, but it tends to be short-lived. It has excellent inherent oxidation stability but relatively poor shear stability and pumpability. Aluminum complex grease has a maximum usable temperature of almost 100°C (212°F) higher than aluminum-soap greases. It has good water-and-chemical resistance but tends to have shorter life in high-temperature, high-speed applications.

2.4.4 Lithium Grease.

Smooth, buttery-textured lithium grease is by far the most popular when compared to all others. The normal grease contains lithium 12-hydroxystearate soap. It has a dropping point around 204 °C (400 °F) and can be used at temperatures up to about 135 °C (275 °F). It can also be used at temperatures as low as -35°C (-31°F). It has good shear stability and a relatively low coefficient of friction, which permits higher machine operating speeds. It has good water-resistance, but not as well as that of calcium or aluminum. Pumpability and resistance to oil separation are good to excellent. It does not naturally inhibit rust, but additives can provide rust resistance. Anti-oxidants and extreme pressure additives are also responsive in lithium greases. (R.L Merker *et al.*, 1945)

2.4.5 Polyurea Grease.

Polyurea is the most important organic nonsoap thickener. It is a low-molecular-weight organic polymer produced by reacting amines (an ammonia derivative) with isocyanates, which results in an oil soluble chemical thickener. Polyurea grease has outstanding resistance to oxidation because it contains no metal soaps (which tend to invite oxidation). It effectively lubricates over a wide temperature range of -20 to 177 °C (-4 to 350 °F) and has long life. Water-resistance is good to excellent, depending on the grade. It works well with many elastomer seal materials. It is used with all types of bearings but has been particularly effective in ball bearings. Its durability makes it well suited for sealed-for-life bearing applications. (C.L.Knoff *et al.,* 1984). Polyurea complex grease is produced when a complexing agent, most commonly calcium acetate or calcium phosphate, is incorporated into the polymer chain. In addition to the excellent properties of normal polyurea grease, these agents add inherent extreme pressure and wear protection properties that increase the multipurpose capabilities of polyurea greases.

2.4.6 Organo-Clay.

Organo-clay is the most commonly used inorganic thickener. Its thickener is modified clay, insoluble in oil in its normal form, but through complex chemical processes, converts to platelets that attract and hold oil. Organo-clay thickener structures are amorphous and gel-like rather than the fibrous, crystalline structures of soap thickeners. This grease has excellent heat-resistance since clay does not melt. Maximum operating temperature is limited by the evaporation temperature of its mineral oil, which is around 177 °C (350 °F). However, with frequent grease changes, this multipurpose grease can operate for short periods at temperatures up to its dropping point, which is about 260 °C (500 °F). A disadvantage is that greases made with higher-viscosity oils for high thermal stability will have poor low temperature performance.. Pumpability and resistance to oil separation are good for this buttery textured grease. (F.H.Thomas *et al.*, 1986)

2.5 Additives

Additive is components that can advance the performance of grease. These primarily include enhancing the existing desirable properties, suppressing the existing undesirable properties, and imparting new properties. The most common additives are oxidation and rust inhibitors, extreme pressure, antiwear, and friction-reducing agents. The additive types are other components of grease that should be selected to be used for oil-lubricated applications and grease formulation. (Jarrod Potteiger *et al.*, 2005) 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. Most performance-enhancing additives found in lubricating oils are also used in grease formulation and should be chosen according to the demands of the application. Figure 2.2 shows some common additive requirements by application.

Additive	**Journal Bearings**	**Ball Bearings**	**Thrust Bearings**	**Roller Bearings**	**Needle Bearings**
Antioxidants	•	•	•	•	•
Antifoam Agents	•	•	•	•	•
Antiwear/EP	•	•	•	•	•
Rust Inhibitors	•	•	•	•	•
Extreme Pressure	•	•	•	•	•
Demulsibility	•	•	•	•	•
VI Improvers	•	•	•	•	•
Corrosion Inhibitors	•	•	•	•	•
• Required, Depend On Application					

Figure 2.2 Common Additive (After (Jarrod Potteiger, 2005)

Example of additive used in industrial application.

Source from Shenzhen Shuhang Industrial Development Co.Ltd.

Table 2.1 Example of Additive

ADDITIVE	FUNCTION
ZINC NAPHTHENATE	Antirust, emulsifying agent
BARIUM DINONYLNAPHTHALENE	Rust resistance for various metals
DODECYLENE SUCCINIC ACID	Preventing metal surface from corrosion

2.6 Applications Suitable For Grease.

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 *et al.,* 1992). 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:

i. Machinery that runs intermittently or in keep in storage for extended period of time. As grease remains in place, a lubricating film can instantly form.

ii. Machinery that is not easily accessible for frequent lubrication. High-quality greases can lubricate isolated or relatively inaccessible components for extended periods of time without frequent replenishing. These greases are also used in sealed-for-life applications such as some electrical motors and gearboxes.

iii. Machinery operating under extreme conditions such as high temperatures and pressures, shock loads, or slow speed under heavy load. Under these circumstances, grease provides thicker film cushions that are required to protect and adequately lubricate, whereas oil films can be too thin and can rupture.

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2.7 Functional Properties of Grease (David R.Merill *et al.*, 1975)

Greases perform their lubrication function by gradually releasing oil into the working areas of the contacting machine surfaces. This function can be compared to that a sponge gradually releasing its liquid over a period of time. A more practical image would be of a concentration of millions of microscopic sponges held inside the machine, close to the working machine components. Each one will gradually releasing oil into the work zone. This criterion of grease often contributes to several functions towards the machinery as well as follow:

i. Functions as a sealant to minimize leakage and to keep out contaminants. Because of its consistency, grease acts as a sealant to prevent lubricant leakage and also to prevent entrance of corrosive contaminants and foreign materials. It also acts to keep deteriorated seals effective (whereas oil would simply seep away).

ii. Holds solid lubricants in suspension. Finely ground solid lubricants, such as molybdenum disulfide (moly) and graphite, are mixed with grease in high temperature service (over 315 °C [599 °F]) or in extreme high-pressure applications. Grease holds solids in suspension while solids will settle out of oils.

2.8 Grease Characteristics

2.8.2 Dynamic Viscosity.

Grease has a resistance to motion, due to its high viscosity. However, as grease is sheared between wearing surfaces and moves faster, its 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 "dynamic viscosity." Dynamic viscosity is the viscosity of a grease that holds only for the shear rate and temperature at which the viscosity is determined. (Prakash K. Ramdoss *et al.*, 1996)

2.8.3 Bleeding, Migration, Syneresis.

Bleeding is a condition when the liquid lubricant separates from the thickener. It is induced by high temperatures and also occurs during long storage periods. Migration is a form of bleeding that occurs when oil in grease migrates out of the thickener network under certain circumstances. For example, when grease is pumped though 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, 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. (Prakash K. Ramdoss *et al.*, 1996)

2.8.4 Consistency, Penetration

Most important feature of grease is its rigidity or consistency. Grease that is too stiff may not feed into areas requiring lubrication, while grease that is too fluid may leak out. Grease consistency depends on the type and amount of thickener used and the viscosity of its base oil (Birova A *et al.*, 2002). 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 5 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. The penetration number of grease and its consistency classification are measured by NLGI classification shown at table 2.2.

Table 2.2 NLGI Grease Classifications

NLGI Grease Classification		
NLGI Number	**ASTM Worked Penetration 0.1mm at 25°C**	**Consistency**
0000	445-475	Semifluid
00	400-430	Semifluid
0	355-385	Very Soft
1	310-340	Soft
2	265-295	Common Grease
3	220-250	Semihard
4	175-205	Hard
5	130-160	Very Hard
6	85-115	Solid

2.8.5 Dropping point. (Truong-Dinh *et al.*, 2003)

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

2.8.6 Oxidation stability.

This is the ability of grease to resist a chemical union with oxygen. The reaction of grease with oxygen produces insoluble gum, sludges, and lacquer-like deposits that cause sluggish operation, increased wear, and reduction of clearances (Giordin D. *et al.,* (2003). Prolonged high temperature exposure accelerates oxidation in greases.

2.8.7 High-temperature effects. (Hoglund E. *et al.*, 2003)

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

2.8.8 Low-temperature effects.

If the temperature of grease is lowered enough, it will become so viscous that it can be classified as hard grease. Pumpability suffers and machinery operation may become impossible due to torque limitations and power requirements. The temperature at which this occurs depends on the shape of the lubricated part and the power being supplied to it (C.Valencia *et al.*, 2006). As a guideline, the base oil's pour point is considered the low-temperature limit of grease.

2.9 Current Research and Trend of Lubricating Grease

Expanded production of new types of lubricating greases and additives has been responsible for the initiation and completion of a series of research studies on more efficient mechanical design of the manufacturing processes. In the production of the major types of grease that are used on a large scale, these studies have been directed mainly towards the commercial use of semicontinuous and continuous processes, along with improvements in the mechanical design for batch processes used in low-volume production. Considerable experience has been accumulated in the commercial Production of Litol-24, Uuiol, and other greases in a semicontinuous process; extensive experimental data have been obtained in continuous units on a pilot-plant and experimental production scale (A. K. Maskaev *et al.*, 2004)

The effectiveness of commercial use of semicoutinuous and continuous process can be judged from the following data: By converting only one of the Litol-24 grease production units at the Berdyansk Experimental Petroleum Lubricating Oil Plant from batch operation to semicontinuous operation, the capacity of the unit has been increased by 60% and the grease cost reduced by 8%, without any substantial capital investment. On the basis of design data, full operation of the semicontinuous process for lithium grease manufacture in a unit with a capacity of 12,000-15,000 metric tons per year will give a saving on the order of 1 million rubles (A. K. Maskaev, *et al.*, 2004).

The results obtained in the research and development work underlying the design of new processes for grease manufacture have been reported extensively in the pages of the journal "Chemistry and Technology of Fuels and Oils." In transporting greases through process lines, certain difficulties arise because of specific properties of the grease, in particular its high viscosity. Studies of the relationships and laws governing the flow of grease have provided a basis for a sufficiently accurate engineering method for engineering calculations on hydraulic systems; the validity of this method has been confirmed by experimental data obtained directly in commercial operation (G. B. Froishteter, 2006).

An important stage in grease manufacture is homogenization, which gives significant improvements in grease service properties. In an article devoted to the homogenization of lithium greases in valve homogenizers, based on extensive experimental material obtained directly under commercial grease production conditions, feasibility has been demonstrated for substantial improvements in the bulk-mechanical properties of lithium greases, even with moderate homogenization pressures. These data have furnished a base for the widespread introduction of USSR-built homogenizers in commercial grease manufacture. Elimination of the early operating difficulties such as plugging of the slots was the subject of an article giving results from a study of this phenomenon in homogenizer valves of different designs.

CHAPTER 3

METHODOLOGY

The research work was carried out within University Malaysia Pahang (UMP) with well equipped to fulfill the requirements. In this chapter, the methodologies of the experiment are discussed.

3.1. Materials

The materials used in this research are discussed as follows.

3.1.1. Lithium Hydroxide (LiOH)

LiOH; $LiOH \cdot H_2O$ Colorless crystals is used as a storage-battery electrolyte, a carbon dioxide absorbent, and in lubricating greases and ceramics. It's also a corrosive alkali hydroxide, white hygroscopic crystalline material and soluble in water, slightly soluble in ethanol. A material safety data sheet (MSDS) of lithium hydroxide can be obtained through appendix A.2.

3.1.2. Spent Lubricant (waste lubricant)

A lubricant is a substance applied between two moving surfaces to reduce the friction and wear between them. After certain limit, the lubricant loss it's effective due to contaminant and treat as waste lubricant. The spent lubricant was gathered from workplace around Gambang's area. The spent lubricant mainly from vehicle part and in this research work, it was filtered to remove some solid sludges and impurities.

3.1.3. Stearic Acid

Stearic Acid is a typical example of a fatty acid, which are essentially long hydrocarbon chains containing a carboxyl group at one end and a methyl group at the other. The majority of fatty acids found in hydrogenated vegetable or animal oils. 12-hydroxy stearic acid, in its refined fatty acid form, is the most commonly used anion. These fats are functions either as the major anion or as modifiers for the desired soap structure. This structure will hold the oil molecule inside the grease like a sponge.
The -carboxyl functional group consists of a carbon atom joined by covalent bonds to two oxygen atoms, one of which in turn is covalently bonded to a hydrogen atom.
Some of stearic properties can be obtained through MSDS at appendix A.1.

3.1.4. Additive

Sodium diethyldithiocarbamate is the chemical compound with the formula $NaS_2CN(C_2H_5)_2$. This salt is obtained by treating carbon disulfide with diethylamine in the presence of sodium hydroxide. Diethyldithiocarbamate is used in spin trapping of nitric oxide radicals, in cancer and as an antioxidant.

3.2. Experimental Preparation And Procedure

3.2.1. Preparation of grease

Spent lubricant was added into mixer with set ratio of 80 %wt. Mixtures of $LiOH{\cdot}H_2O$ and stearic acid taken as 1:1 ratio to produce thickener. Compositions of thickener are set as follow: 5, 10, 15, and 20%wt. Homogeneous stirring mixture condition of 190°C was applied for about 3hrs. After the heating period, the mixture was allowed to cool, at 150 ◦C, an additional amount of spent lubricant (60–80% of the total reaction mixture) and additive (0–10 wt. % of the total reaction mixture) were added. The summary preparation of grease is showed at figure 3.1.

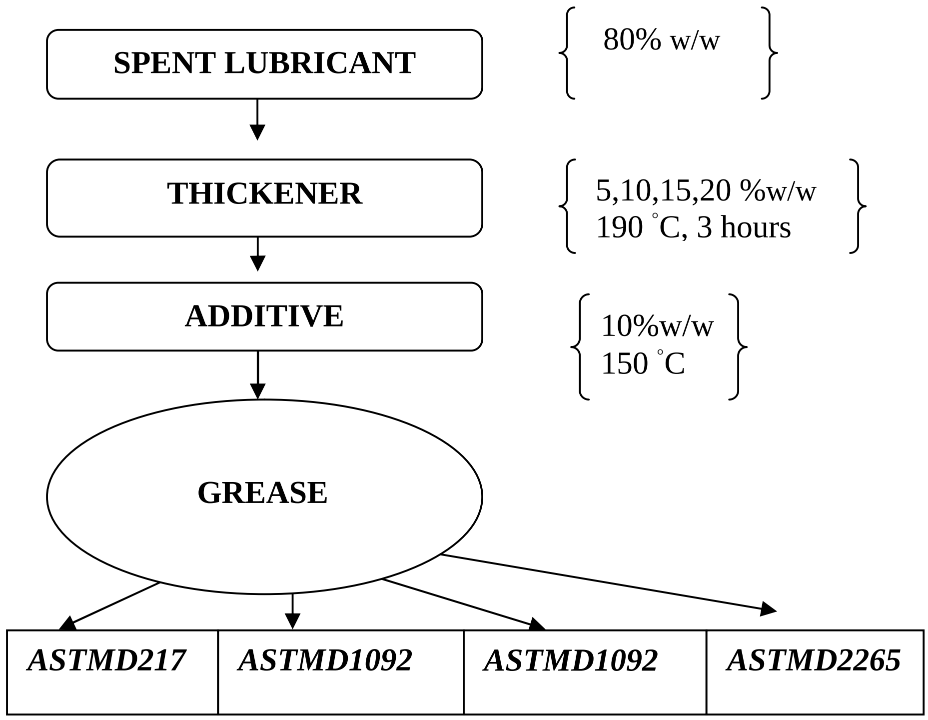


Figure 3.1 Schematic Diagrams for Grease Preparation Composition of SpentLubricant Oil-Based Grease

Table 3.1 Compositions of Thickener, Spent Lubricant And Additive.

Greases	**Thickener (Wt %)**	**Spent lubricant (Wt %)**	**Antioxidant (Wt %)**
GR1	5.0	80	15
GR2	10.0	80	10
GR3	15.0	80	5
GR4	20.0	80	0

Table 3.2 Variation of Temperature for Identifying Grease Formation in Mixer

Mixer Temperature °C	**Grease Formation**
150	
170	
200	
230	

Table 3.2 shows the variation of mixer temperature to examine the grease formation. The temperatures were set 150, 170, 200, and 230 °C respectively. The formation of grease was measured by the physical appearance of the grease inside the mixer. For example, homogenous, no multiple layer produce, and viscous.

3.3 Grease Characteristic Test

There were four major characteristics being examined during the research which were the consistency, oxidation stability, flash point, and the penetration of grease. These characteristic will provide information on the quality of produced grease

3.3.1 Penetration Test (ASTM D217)

Micro processor-based digital penetrometer is a good measurement apparatus for the test. Stainless steel with tip brass cone (45 °cone and weight 102.5 g) was allowed to free drop and penetrates the grease surface for 5s.

3.3.2 Oxidation Stability Test (ASTMD4048)

Oxidation of grease was conducted by submerged a copper strip into 100 ml grease sample. The sample was heat at water bath with a constant temperature of 100◦C and duration of 24 hours. The colour of the copper was compared with copper strip corrosion colour standard.

3.3.3 Dynamic Viscosity Test (ASTMD1092.)

Apparent viscosity of grease was determined using Brookfield viscometer. The test was conduct by using LV-4 spindle with constant 20 RPM at 25°C. This test is ideal way to determining the flow characteristic of grease through lines, dispensing equipment as well as pumpability.

3.3.4 Dropping Point Test (ASTM D2265)

The test apparatus consists of a grease cup with a small hole in the bottom, test tube, two thermometers, a container, stirring device if required and an electric heater. The inside surfaces of the grease cup are coated with the grease to be tested. A thermometer is inserted into the cup and held in place so that the thermometer does not touch the grease. This assembly is placed inside a test tube. The test tube is lowered into the container which is filled with oil. Another thermometer is inserted into the oil/block

The test was conduct by heating up the oil while being stirred, at a rate of 8 to 12 RPM until the temperature is approximately 30°C below the expected dropping point. The heat is reduced until the test tube temperature is 4°C or less than the oil/block temperature. Once the temperature has stabilized the sample is inserted
The dropping point is the temperature recorded on the test tube thermometer, plus a correction factor for the oil/block temperature, when a drop of grease falls through the hole in the grease cup. If the drop trails a thread, the dropping temperature is the temperature at which the thread breaks

CHAPTER 4

RESULT & DISCUSSION

4.0 Introduction

From each test that had been conducted, the results give sufficient information towards certain quality of lubricant base oil grease. Two parameters that affect the quality of grease were the thickener concentration and the reaction temperature. Table 4.1 shows summary of the composition (wt %) of chemical used to prepare grease.

Table 4.1 Compositions of Thickener, Spent Lubricant And Additive.

Greases	**Thickener (Wt %)**	**Spent lubricant (Wt %)**	**Antioxidant (Wt %)**
GR1*	5	80	15
GR2*	10	80	10
GR3*	15	80	5
GR4*	20	80	0

* Reaction temperature of 150, 170, 200°C, and 230 °C for each grease

4.1 Effect of Thickener Composition

First parameter that had been examined in this research was the thickener composition. Lithium thickeners were varied into suitable amount, so that the grease can take effect. 5, 10, 15, and 20% of thickener were being used in this research.

4.1.1 Penetration Test ASTM D217

Grease's consistency is its resistance to deformation by an applied force. The development of lubricating grease with the right consistency requires optimization of components and preparation protocol. Important performance properties such as adhesion, rheology, and lubrication are largely dependent on the grease hardness and its ability to maintain a stable lubricating film at the metal contact zone. Manipulating the base oil quantity and fatty acid composition can help controlling the grease hardness. Figure 2.4 represented the result of increasing the amount of thickener towards penetration of cone. As resulted, the penetration of the cone decrease as the thickener amount is increase. This indicates that the grease become brittle and hard along with the increasing of thickener amount. More thickener will provide more complex microstructure of the grease itself. The space between structures become small and compact, thus reduce the force ability to penetrate the grease structure. By comparing the value of cone penetration with the NLGI standard, grease class type can be identified. The corresponding value of NLGI standard as show at Table 1.5

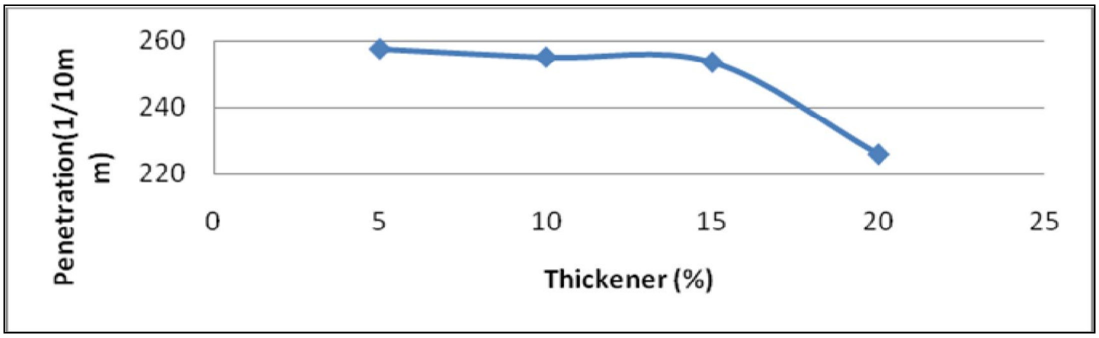


Figure 4.1 Graph of Cone Penetration versus Thickener Composition

Table 4.2 NLGI Grease Classification

NLGI Grease Classification		
NLGI Number	**ASTM Worked Penetration 0.1mm (3.28 x 10 ft) at 25°C**	**Consistency**
0000	445-475	Semifluid
00	400-430	Semifluid
0	355-385	Very Soft
1	310-340	Soft
2	265-295	Common Grease
3	220-250	Semihard
4	175-205	Hard
5	130-160	Very Hard
6	85-115	Solid

As taken average value of grease in this research and comparing with NLGI standard, most of the grease samples were type 2 and 3. Most commercial grease is within NLGI number 2 which is indicate that the consistency of grease was between soft and semi hard. Thus, soap with 5 to 10% thickener amount resulted in a soft grease (GR1 &GR2).That was capable of better wetting the metal surface if applied. Comparatively harder grease (GR3&GR4) under the geometry of wear test would be pushed to the side by rotating ball and were unable to return to relubricate the metals in contact. This condition is not suitable if the grease is applied onto high speed rotating gear or motor. Grease that is too stiff may not feed into areas requiring lubrication, while grease that is too fluid may leak out. Normally, grease consistency depends on the type and amount of thickener used and the viscosity of its base oil.

Based on previous studied, by M. A. Delgado et al.,(2005) as a metal soap is dispersed in a mineral oil, the crystallized soap particles arrange themselves to form a characteristic microstructure depending on soap concentration, base oil viscosity, and type of metal soap used, due to the balance of forces between the colloidal particles and the oil medium. These soap fibers are distributed in a random manner within a given volume, as can be seen in Figures 4.2, where selected micrographs of the systems studied, at magnifications of 5000× shown. As can be observed, a well-formed entanglement network among structural units generally appears, although this microstructure is significantly affected by both thickener concentration and oil base viscosity. Large particles in the form of platelets are found for the lowest soap concentration, while entangled fibers clearly develop at concentrations higher than 14% w/w, the density of physical entanglements being larger as soap concentration increases, i.e., 20% w/w, thus allow increasing the grease hardness.

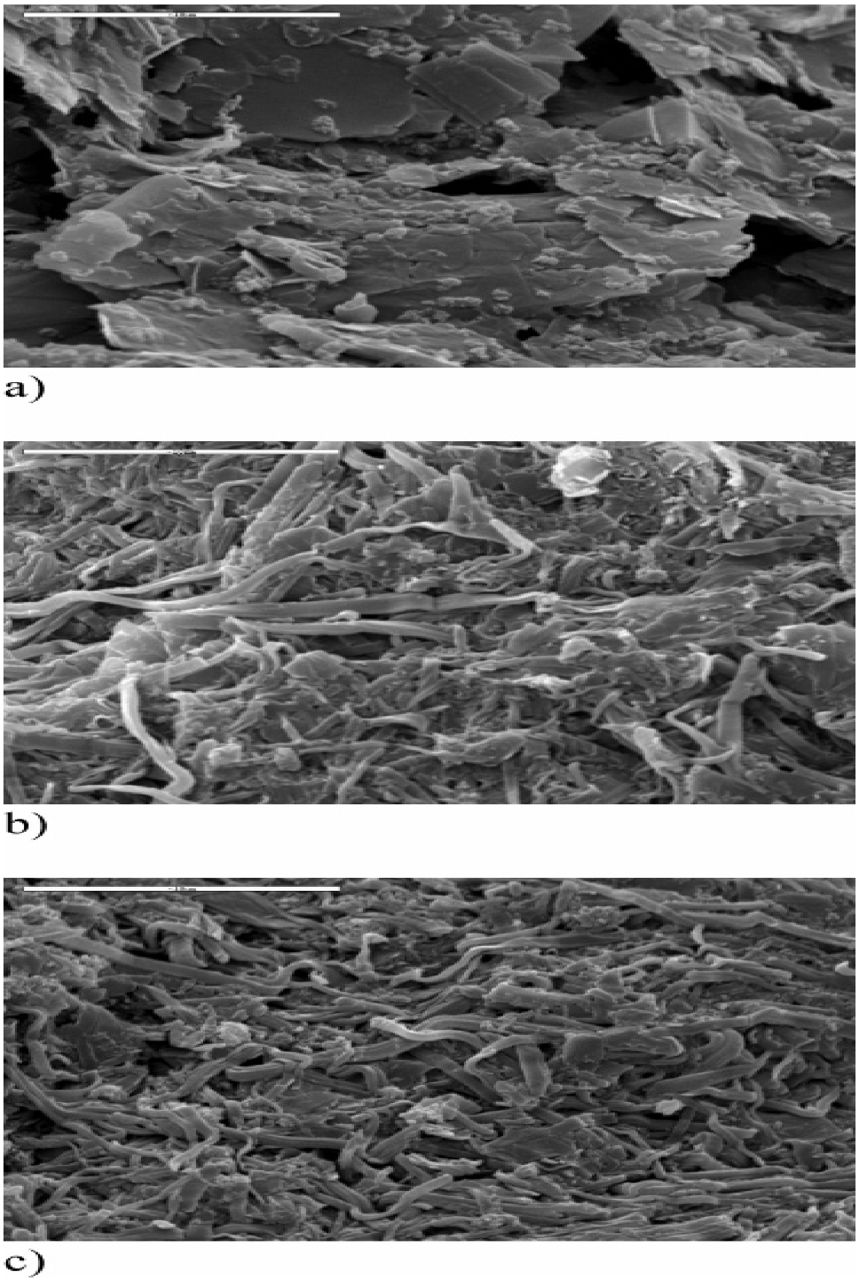


Figure 4.2 SEM micrographs (5000×) of lithium lubricating greases with different soap concentrations: (a) 8% (w/w), (b) 14% (w/w) and (c) 20% (w/w)

4.1.2 Dynamic Viscosity Test ASTM D1095

Figure 4.3 shows the viscous flow curves at 25°C for lubricating grease having different thickener amount. From the figure, it is clearly show that, increasing of the thickener amount will increase the dynamic viscosity of the grease. Referring to the graph, Cp is represented as dynamic viscosity unit in Centi Poise. In microstructure study, as a metal soap is dispersed in a mineral oil, the crystallized soap particles arrange themselves to form a characteristic microstructure depending on soap concentration, oil viscosity and type of metal soap used. Large amount of soap in the grease will compacting the fiber soap matrix within the grease. The forces that connect each fibber will resist the grease to move. Higher viscosity of grease will be form. Choosing a low viscosity and high viscosity of grease is very important in many applications, especially on high or low rotating tools such as bearing or gearbox. As speed increases, the required viscosity of the grease decreases. If the viscosity is too high, fluid friction generates excessive heat and reduces grease lifetime. For every 15° C increase, the expected grease life is cut in half. If viscosity is too low, boundary friction between the mating components generates heat — and excessive wear of the components.

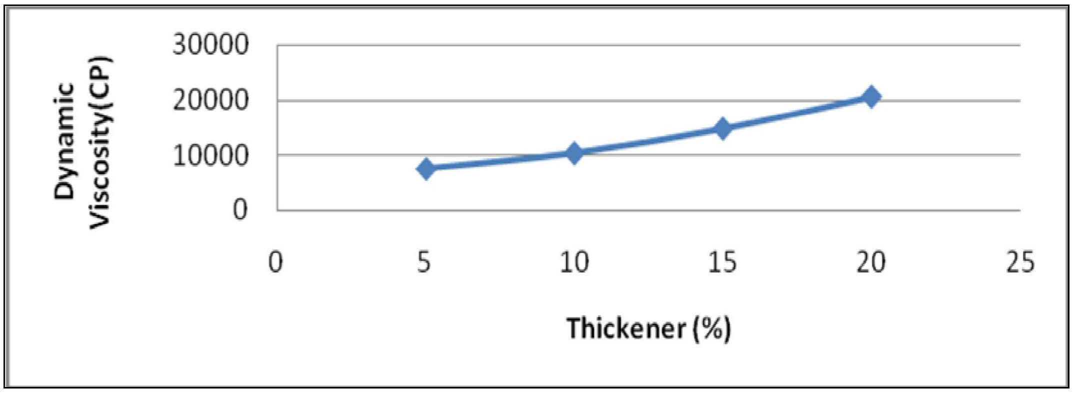


Figure 4.3 Graph of Dynamic Viscosity Having Different Thickener Amount

Based on previous studied, it is well-known that, when a shear stress or a shear rate is applied to a lubricating grease sample, a reorientation of fibers in the micro structural network takes place and, as a result, a shear-thinning flow behavior is observed. The research conducted by observing the value of *k* and *n* as the thickener concentration increase (C. Valencia *et al.*, 2005).

$$\acute{\eta} = \mathbf{k}\gamma^{\mathbf{n-1}}$$

where "*k*" and "*n*" are the consistency and flow indexes, respectively.

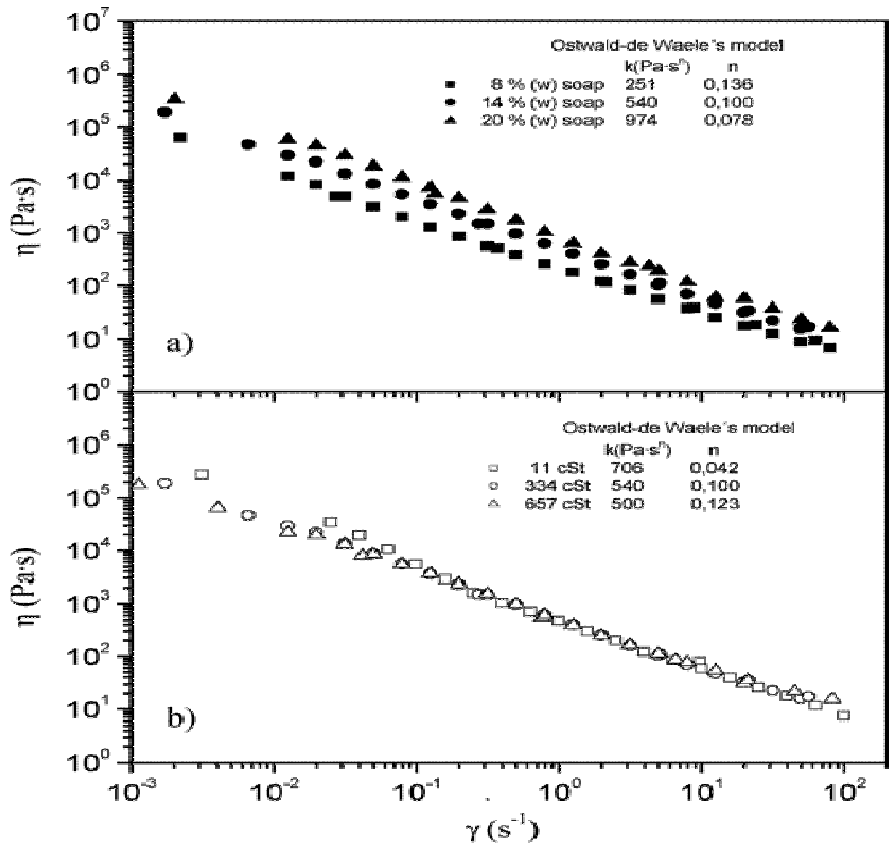


Figure 4.4 Viscous Flow Curves, At 25 °C, For Lubricating Greases Having Different Thickener Concentrations

As shows in Figure 4.4, viscosity clearly increases with soap concentration, yielding higher values of the consistency index, *k.* while, the flow index, *n*, decreases by increasing the thickener concentration, being quite close to zero for the highest concentration studied, which is representative of the typical yielding behavior shown by these materials

4.1.3 Dropping Point Test ASTM D2265

Referring to the graph in Figure 4.5, the increasing of thickener composition will lead to the increasing of grease dropping point. The thickener function is to tighten the grease by introducing crystalline structure that held the oil. This sponge structure is connecting by Van Der Walls forces. Increasing the soap concentration will make the grease become harder because the structural or network is increase. This will contribute to a longer time and higher temperature needed to break the chain or network structure, thus change the phase of the grease into liquid.

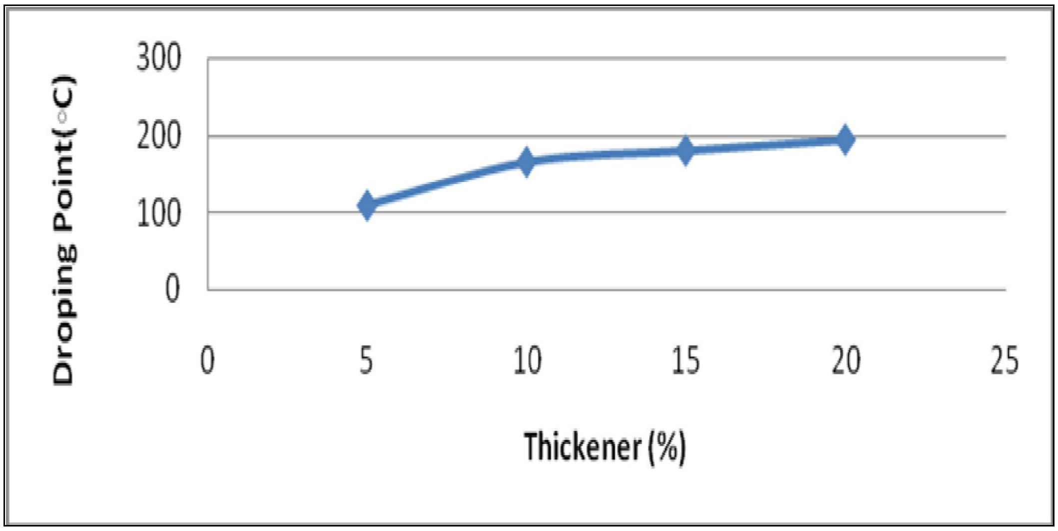


Figure 4.5 Graph of Grease Dropping Point versus Thickener Amount

Based on previous research done by Browning G. A, (1995) the improved volatility characteristics of grease with higher thickener amount are attributed to better interaction between soap fiber structure and the confined base oil. The fiber – base oil interaction profile of the grease matrix is not fully developed in grease with less thickener and therefore resulted in ‘bleeding’ of the base oil at test temperature.

4.1.4 Oxidation Stability Test ASTM D4048

Oxidation test was conducted by using copper strip corrosion test. Oxidation and corrosion is intimately related to each other. High corrosion towards copper indicates low oxidation stability and shorter grease lifetime.

Table 4.3 Result of Grease Corrosion towards Copper Strip Test

GREASE	*THICKENER (WT %)*	*REACTION TEMPERATURE (◦C)*	*CORROSION*
GR1	5	200	4b
GR2	10	200	4a
GR3	15	200	3b
GR4	20	200	3a

Shows at Table 4.3, four types of greases, GR1, GR2, GR3, and GR4, with different amount of thickener. GR1 contains less amount of thickener compare to GR4. The corrosion of copper strip represent by the color change of copper strip. The colour of copper strip change to almost dark will indicate the high corrosives of the grease and not stable especially in extreme condition. In table 4.3, the corrosive of grease decrease as the amount of thickener increase. GR1 which contain the less thickener (5%) indicate type 4b corrosion and change the copper into dark blue colour even though it contain the highest amount of additive. Compare to GR4 which contain 20% thickener, result in small reduction of corrosive toward the copper with dark tarnish in colour. To make a good stability of grease it is not only dependent on the additive it also considers the type of thickener and base oil in used. All sample indicates slight corrosive towards copper due to contamination inside the waste lubricant. This trap metal and sludge tend to react with copper and corrode the surface. Meanwhile, the amounts of thickener also influence the corrosivitiy and oxidation stability of grease in extreme condition. Thickener is act as a holding space of oil and additives inside the grease. This gel structure will hold the antioxidant additive to remain in place. Less thickener shows not enough space to hold these and additive not soluble in the grease give high tendency of corrosion.

The finding is supported by the previous research Brajendra K. et al., (2006) where the grease samples were synthesized with different thickener/oil ratios to study the effect of the soap on oxidation stability and wear behavior. The oil being used is soybean oil. The oxidation stability is determined by measuring the weight loss in greases during Thin Film Micro Oxidation (TFMO) process. TFMO is a test choice for quantitative evaluation of grease thermal and oxidative stabilities. Base on the study, the weight loss decreased with the increasing of soap content in grease. Greater weight loss in grease represents the low oxidation stability of the grease.

4.2 Effect of Reaction Temperature

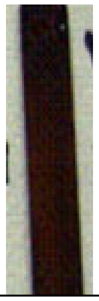
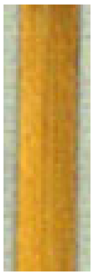
Very important parameter to ensure grease can be formed is the reaction temperature. The temperature was controlled inside the reaction process. The formation of grease was indicate by the physical appearance of the compound. Grease is form during the mixture of thickener with base oil is completely homogeneous. The color may change as well; brownish or lighter than initial mixture. In the formation of grease, particles can only react when they collide. If substance is heated, the particles move faster and collisions occur frequently.

4.2.1 Oxidation Stability Test ASTM D4048

Referring to the Table 4.4, the corrosivity of grease samples were slightly decrease as reaction temperature increase. At low temperature, lithium based grease was not completely formed. The soap to oil interaction was not stable at low temperature due to limited energy supplied by the heat, hence lead to incomplete reaction. Proper amount of thickener also contribute to stability of the grease. Higher temperature give much time and space for the grows of fiber microscopic structure inside the grease and this structure had been discussed before, will give greater influence towards the characteristic of the grease as well as oxidation stability. Heating the grease with such high temperature will help the grease reach its complete maturity in inside structure which then can hold the soap, oil and additive molecules in one place. Evaporation of this molecule due to extreme condition will result in a distraction and damage of the grease. This situation is supported by previous research which preferred high reaction temperature to produce lithium based grease. The studied by Sevim Z. Erhan et al (2005), preferred temperature at 190◦C for the preparation of grease. It is supported by Brajendra K. Sharma,(2005) also used the preferred reaction temperature at 200◦C.

As the improvement of microstructure technologies and good understanding of grease behavior, most commercial lithium based grease type use high temperature of mixing or reaction to produce a high quality of grease.

Table 4.4 Result of Grease Corrosion towards Copper Strip Test

GREASE	***THICKENER (Wt%)***	***REACTION TEMPERATURE (◦C)***	***CORROSION***	
				
GR1	5	150	4a	
		170	4b	
		200	4b	
		230	4b	
GR2	10	150	4b	
		170	4a	
		200	4a	
		230	4a	
GR3	15	150	3b	
		170	3b	
		200	3a	
		230	3a	
GR4	20	150	3a	
		170	2c	
		200	2c	
		230	2c	

4.2.2 Penetration Test ASTM D217

Reaction temperature gives significant effect towards grease characteristic. Heating process during preparation of grease is very useful medium to supply reaction energy or activation energy for the chemical reaction, thus produce grease. As indicated on the Figure 4.6, four reaction temperatures had been set up which were 150, 170, 200, and 230°C. Result shows that the grease hardness increase as the reaction temperature increased. The penetration of cone was much deeper for the grease at lower reaction temperature. This is due incomplete formation of thickener structure to produce grease. The fiber chain of thickener is not ready enough to hold the base oil molecule onto place hence, soft grease is produce. At suitable high temperature (230°C), the mixture gain enough energy to complete the process and the grows of sponge-like structures was fairly optimum thus create grease characteristic. Referring to the figure, each grease with increasing thickener amount, the grease become harder and after comparing the penetration value with NLGI standard, the grease is classified in group 2.

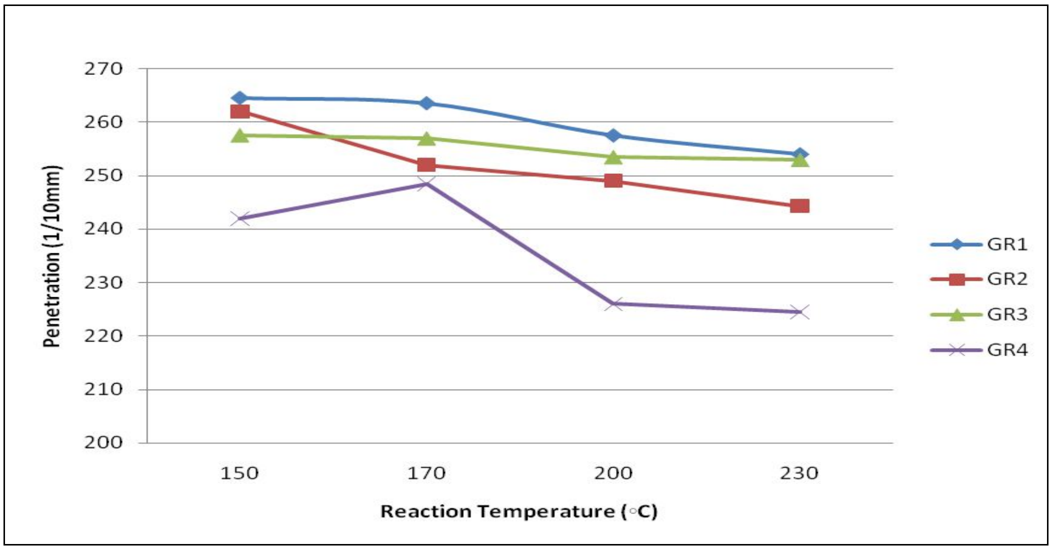


Figure 4.6 Graph of Grease Cone Penetration Having Different Reaction Temperature

4.2.3 Dynamic Viscosity Test ASTM D1095

Figure 4.7 shows the effect of reaction temperature on the dynamic viscosity of grease. The figure shows that the dynamic viscosity of grease is increase with increasing of reaction temperature. The soap fiber structure of based lithium soap grease influences its tribological performance such as friction and wear. Higher reaction temperature indicates unrestricted growth of soap fiber within grease. The lubricant oil molecule will spread through the soap structure and hold them onto place. The low viscosity behavior supply by the base oil and the elasticity behavior of thickener create a viscous behavior of the grease. Referring to the Figure 4.7, the viscosity of grease is increase gradiently for GR1 which contain less amount of thickener. The same condition occurs for other greases within temperature of 170◦C. however, beyond this temperature, ranging from 200 to 230◦C, the viscosity of greases start to constant. This indicates the best temperature for the reaction to occur is within this range. This is proved and supported by current situation which most of grease that produce in commercial market are using temperature within the range of 200 to 230◦C (Crone I. *et al.*, 2003)

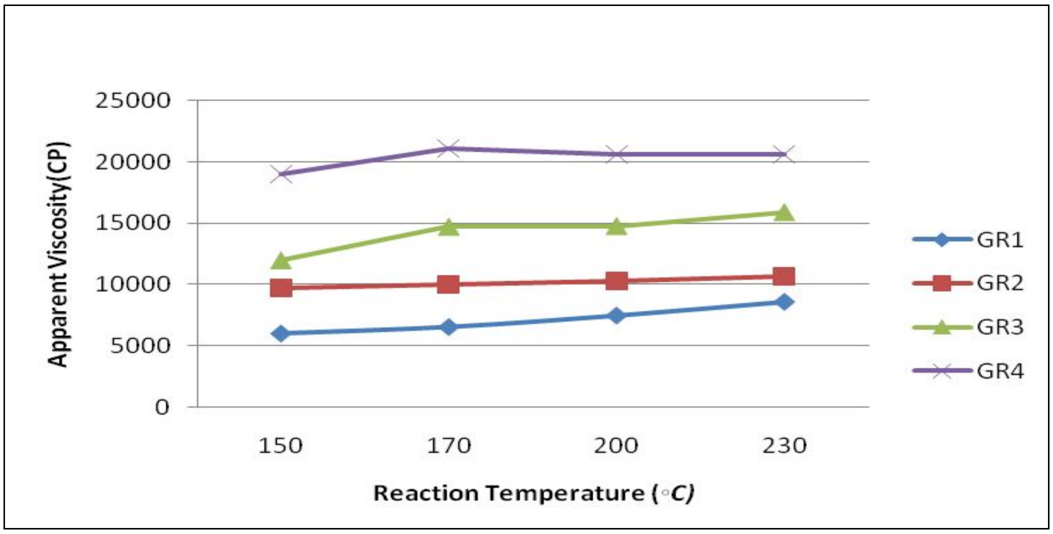


Figure 4.7 Graph of Grease Dynamic Viscosity Having Different Reaction Temperature

4.2.4 Dropping Point Test ASTM D2265

Dropping point is the optimum temperature at which grease can operate. Beyond this temperature, the grease will loss it firm and hardness and may leak into other side of machinery part. The trend of the graph indicates increasing of dropping point as reaction temperature is increase. Taken 230◦C as the normal reaction temperature, it shows that the operating temperature is maximum at GR4 with 195◦C. Comparing the result for each greases, the dropping point of grease is not much dependable towards reaction temperature of grease. This is because, the dropping point of greases are much depending on thickener composition along with the solubility of the oil component. Grease must not soften and flow during normal operating temperatures. Note, grease consistency and firmness will change when the machinery warms up from ambient temperature to operating temperature. The dropping point property is useful for checking the quality and uniformity of a specific manufacturer's product and for comparing various grease brands for specific application.

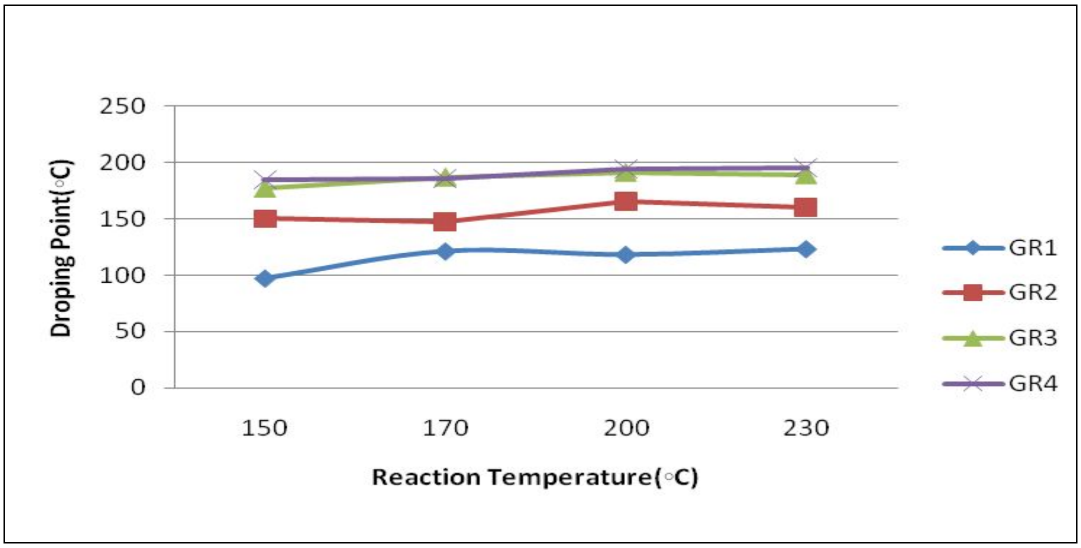


Figure 4.8 Graph of Grease Dropping Point Having Different Reaction Temperature

CHAPTER 5

CONCLUSION

5.0 Introduction

From this research, it is desire to prove that the spent lubricant oil can be used to prepare lithium based grease. The usage of waste lubricant oil in many application can be extended to maximum. The problem involving environment quality can now be reduce with the development of new technologies and good understanding of grease behavior.

5.1 Conclusion

From the result, it can be concluded that spent lubricant can be utilized as a main composition in the preparation of grease. The spent lubricant which is not been refined serve certain quality towards the characteristic of the grease although it slightly not comparable to common grease, however, this lubricating grease can be applied to other purposes. Good quality of grease is produced by the combination of good base oil, thickener and additive. However, several characteristics of lubricating grease are depending on the thickener composition within the grease. Thickener plays a major influence in determining grease property. The suitable amount of thickener mainly, will lead to the greater proportion of performance of the lubricating grease.

As the scope of this research is to investigate the effect of thickener composition towards characteristics of grease it can be concluded that:

i. Proper temperature of mixing provide sufficient energy for grease to formed

ii. By increasing amount of thickener, the corrosion decrease

iii. By increasing amount of thickener and reaction temperature will decrease the consistency of the grease.

iv. By increasing amount of thickener and reaction temperature will increase of apparent viscosity of the grease

v. By increasing amount of thickener and reaction temperature will increase the dropping point of the grease.

5.2 Recommendations for Future Study

i. Spent lubricant from light source such motorcycle and light machinery is prefer to be utilized as raw material as it less contaminate compare to heavy vehicles, which generate combustion and in contact with other harm component such as H_2S and SO_4.

ii. Use variety of thickener to compare the characteristic of grease. In this work, lithium base is choose as thickener, besides there are lot other thickener types can be use as example sodium base, aluminium base, and much more. Each thickener gives different characteristic and performance towards grease.

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APPENDIX A

MSDS: STEARIC ACID

Material Safety Data Sheet
Stearic acid MSDS

Section 1: Chemical Product and Company Identification

Product Name: Stearic acid

Catalog Codes: SLS2320, SLS3742

CAS#: 57-11-4

RTECS: WI2800000

TSCA: TSCA 8(b) inventory: Stearic acid

CI#: Not available.

Synonym: Octadecanoic acid; 1-Heptadecanecarboxylic acid; Stearophanic acid; n-Octadecanoic acid

Chemical Name: Stearic Acid

Chemical Formula: C18H36O2

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Stearic acid	57-11-4	100

Toxicological Data on Ingredients: Not applicable.

Section 3: Hazards Identification

Potential Acute Health Effects: Slightly hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Not available.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Not available.
Repeated or prolonged exposure is not known to aggravate medical condition.

Figure A.1

MSDS: LITHIUM HYDROXIDE:

LITHIUM HYDROXIDE			0913 March 1998
CAS No: 1310-65-2 RTECS No: OJ6307070 UN No: 2680	LiOH Molecular mass: 23.95		
TYPES OF HAZARD/ EXPOSURE	**ACUTE HAZARDS/SYMPTOMS**	**PREVENTION**	**FIRST AID/FIRE FIGHTING**
FIRE	Not combustible.		In case of fire in the surroundings: all extinguishing agents allowed.
EXPLOSION			
EXPOSURE		**PREVENT DISPERSION OF DUST! AVOID ALL CONTACT!**	**IN ALL CASES CONSULT A DOCTOR!**
Inhalation	Sore throat. Cough. Burning sensation. Shortness of breath. Laboured breathing. Symptoms may be delayed (see Notes).	Ventilation (not if powder), local exhaust, or breathing protection.	Fresh air, rest. Half-upright position. Refer for medical attention.
Skin	Redness. Pain. Skin burns. Blisters.	Protective gloves. Protective clothing.	Remove contaminated clothes. Rinse skin with plenty of water or shower. Refer for medical attention.
Eyes	Redness. Pain. Severe deep burns.	Face shield, or eye protection in combination with breathing protection.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then take to a doctor.
Ingestion	Abdominal pain. Burning sensation. Headache. Nausea. Shock or collapse. Vomiting. Weakness.	Do not eat, drink, or smoke during work.	Rinse mouth. Do NOT induce vomiting. Give nothing to drink. Refer for medical attention. See Notes.

SPILLAGE DISPOSAL	**PACKAGING & LABELLING**	
Sweep spilled substance into dry containers. Wash away remainder with plenty of water. Do NOT let this chemical enter the environment. (Extra personal protection: complete protective clothing including self-contained breathing apparatus).	UN Hazard Class: 8 UN Pack Group: II	Airtight. Do not transport with food and feedstuffs.

EMERGENCY RESPONSE	**STORAGE**
Transport Emergency Card: TEC (R)-751	Separated from food and feedstuffs, strong oxidants, strong acids. Dry. Well closed.

0913	LITHIUM HYDROXIDE
IMPORTANT DATA	
Physical State; Appearance COLOURLESS HYGROSCOPIC CRYSTALS. **Chemical dangers** The substance decomposes on heating (924°C), producing toxic fumes. The solution in water is a strong base, it reacts violently with acid and is corrosive to aluminium and zinc. Reacts with oxidants. **Occupational exposure limits** TLV not established.	**Routes of exposure** The substance can be absorbed into the body by inhalation of its aerosol and by ingestion. **Inhalation risk** Evaporation at 20°C is negligible; a harmful concentration of airborne particles can, however, be reached quickly when dispersed. **Effects of short-term exposure** The substance is corrosive to the eyes, the skin and the respiratory tract. Corrosive on ingestion. Inhalation of the substance may cause lung oedema (see Notes). Exposure may result in death. The effects may be delayed. Medical observation is indicated.
PHYSICAL PROPERTIES	
Boiling point (decomposes): 924°C Melting point: 450-471°C	Density: 1.46 g/cm^3 Solubility in water, g/100 ml at 20°C: 12.8
ENVIRONMENTAL DATA	
This substance may be hazardous to the environment; special attention should be given to water organisms.	
NOTES	
The symptoms of lung oedema often do not become manifest until a few hours have passed and they are aggravated by physical effort. Rest and medical observation are therefore essential. Immediate administration of an appropriate spray, by a doctor or a person authorized by him/her, should be considered.	

Figure A.2

MSDS: SODIUM DIETHYL DITHIOCARBAMATE

Material Safety Data Sheet
Sodium diethyldithiocarbamate trihydrate MSDS

Section 1: Chemical Product and Company Identification

Product Name: Sodium diethyldithiocarbamate trihydrate

Catalog Codes: SLS2596, SLS4018

CAS#: 20624-25-3

RTECS: EZ6550000

TSCA: TSCA 8(b) inventory: Sodium diethyldithiocarbamate trihydrate

CI#: Not available.

Synonym:

Chemical Name: Not available.

Chemical Formula: (C2H5)2NCS2Na.3H2O

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Sodium diethyldithiocarbamate trihydrate	20624-25-3	100

Toxicological Data on Ingredients: Sodium diethyldithiocarbamate trihydrate: ORAL (LD50): Acute: 1500 mg/kg [Rat]. 1500 mg/kg [Mouse].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of eye contact (irritant), of ingestion. Hazardous in case of skin contact (irritant, permeator), of inhalation. Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Not available.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Not available.
Repeated or prolonged exposure is not known to aggravate medical condition.

Figure A.3

APPENDIX B

ANALYSIS DATA

To investigate the effect of thickener percentage composition towards grease consistency

Equipment : Penetrometer

Table B.1 Determining Consistency Of Grease

Greases	**Heating temperature**	**Thickener (Wt %)**	**Penetration (1/10mm)**
GR1	150	5.0	264.49
	170	5.0	263.5
	200	5.0	257.5
	230	5.0	254
GR2	150	10.0	262
	170	10.0	252
	200	10.0	249
	230	10/0	244.3
GR3	150	15.0	257.6
	170	15.0	257
	200	15.0	253.5
	230	15.0	253
GR4	150	20.0	242
	170	20.0	248.5
	200	20.0	226
	230	20.0	224.5

Figure B.1: Apparatus for Determining Consistency Of Grease

To study the effect of thickener towards dynamic viscosity of grease

Equipment : Viscometer, LV- 4 Spindle RPM: 20

Temperature : 25°C

Table B.2 Determining Viscosity Of Grease

Greases	**Heating temperature**	**Thickener (Wt %)**	**Viscosity (CP)**
GR1	150	5.0	5979
	170	5.0	6509
	200	5.0	7429
	230	5.0	8546
GR2	150	10.0	9718
	170	10.0	9988
	200	10.0	10,298
	230	10.0	10,675
GR3	150	15.0	11,937
	170	15.0	14,697
	200	15.0	14,757
	230	15.0	15,894
GR4	150	20.0	19,022
	170	20.0	21,056
	200	20.0	20,606
	230	20.0	20,556

Figure B.2: Apparatus for Determining Dynamic Viscosity of Grease

To study the effect of thickener towards dropping point of grease.

Equipment : Droping Point Analyzer

Table B.3 Determining Droping Point Of Grease

Greases	**Heating temperature**	**Thickener (Wt %)**	**Drop point (◦C)**
GR1	150	5.0	97
	170	5.0	121
	200	5.0	118
	230		123
GR2	150	10.0	150
	170	10.0	147.3
	200	10.0	165.5
	230		160
GR3	150	15.0	177
	170	15.0	187
	200	15.0	191
	230		189
GR4	150	20.0	184.5
	170	20.0	185.5
	200	20.0	194
	230		195

Figure B.3: Apparatus for Determining Dropping Point of Grease

Figure B.4: Apparatus for Determining Oxidation Stability of Grease

ASTM COPPER STRIP CORROSION STANDARD

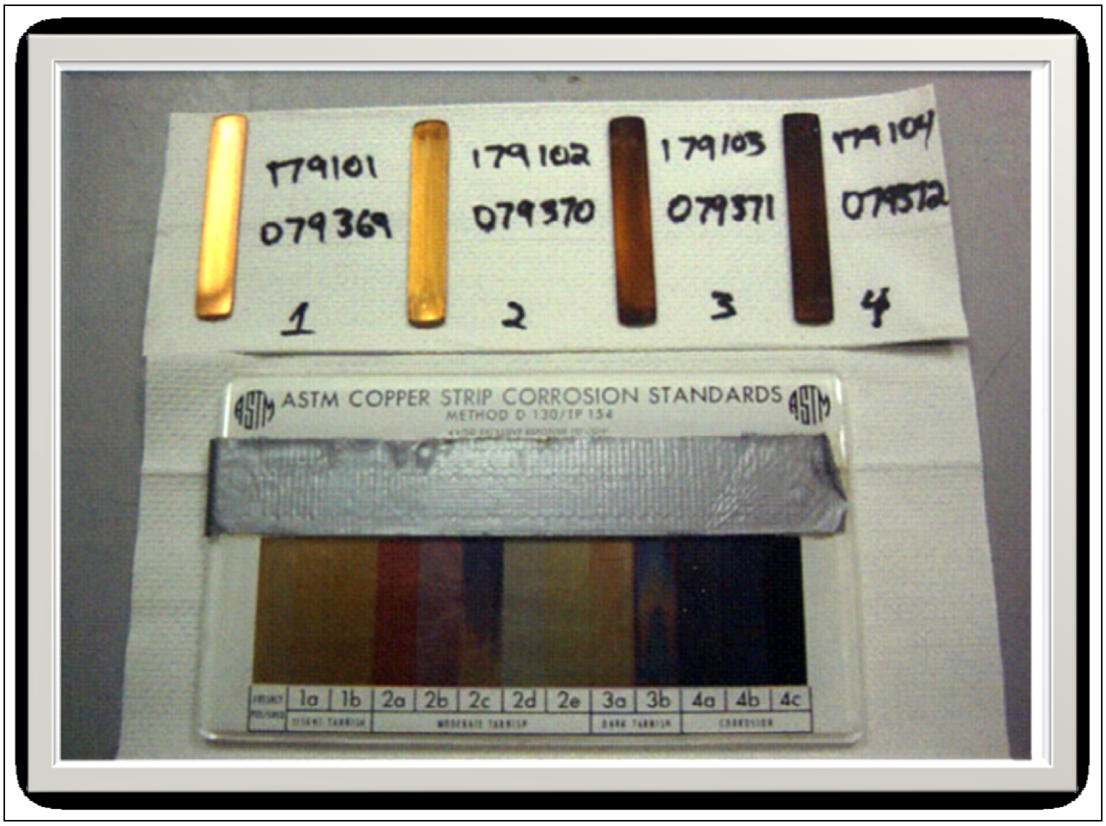

Figure B.4