PRODUCTION OF GREASE FROM WASTE COOKING OIL

NORASMAH BINTI HAJI OTHMAN

UNIVERSITI MALAYSIA PAHANG

"I hereby declare that I have read this thesis and in my opinion this thesis has fulfilled the qualities and requirements for the award of Degree of Chemical Engineering"

Signature   :

Name of Supervisor   :

Date   : 2 May 2009

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NORASMAH BINTI HAJI OTHMAN

A thesis submitted in fulfillment
of the requirements for the award of the degree of
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Universiti Malaysia Pahang

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I declare that this thesis entitled "Production of grease from waste cooking oil" 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   :

Name   : Norasmah Binti Haji Othman

Date   : 2 May 2009

Dedicated especially to Father, Mother, Brothers and Sisters who give me inspiration
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ABSTRACT

Vegetable oils have significant potential as a base fluid and a substitute for mineral oil for grease formulation. The objective of this research is to produce grease from waste cooking oil via saponification of a stearic acid with a lithium hydroxide monohydrate, LiOH. In this research, preparation and formulation of bio-based grease and determination of optimum operating condition were studied. The produced bio-based grease is expected to have better performance, and apply in variety industry such as agriculture/farming, forestry applications and food processing. The stearic acid, lithium hydroxide monohydrate and waste cooking oil compositions were manipulated in this research. The operating temperature and mixing period were also studied. The characteristics of produced grease were analyzed in order to evaluate the performance of the grease. Among parameters analyzed were depth penetration test, dropping point and oxidation stability. While grease hardness was determined using depth penetration test, and their oxidative stabilities was determined using copper corrosion test. The dropping point temperature was tested using dropping point apparatus. Results indicate that the lithium hydroxide monohydrate, stearic acid and waste cooking oil content significantly affect grease hardness, oxidative stability and dropping point temperature. NLGI 2 (common grease) grease was formulated in this study with optimum condition.

ABSTRAK

Minyak masak mempunyai potensi yang baik untuk digunakan sebagai ganti kepada minyak mineral untuk formulasi gris. Objektif eksperimen ini adalah untuk menghasilkan gris daripada minyak masak terpakai dengan menggunakan kaedah saponifikasi asid lemak iaitu asid stearik dengan lithium hidroksida monohidrat, LiOH. Eksperimen ini meliputi pembuatan bio-gris dalam mencari keadaan optimum untuk memformulasi bio-gris supaya lebih baik dan dapat digunakan untuk applikasi industri, alat-alat pertanian dan juga applikasi perhutanan. Komposisi asid stearik, lithium hidroksida monohidrat dan juga minyak masak terpakai dimanipulasi di dalam eksperimen ini. Suhu operasi dalam pembuatan gris juga dimanipulasi. Ciri-ciri gris yang digunakan untuk industri dan applikasi automotif adalah sangat bergantung kepada kekerasan dan kestabilan oksidatif gris tersebut. Kekerasan gris adalah ditentukan dengan menggunakan ujian penetrasi dan ciri kestabilan oksidatif gris tersebut ditentukan dengan menggunakan ujian hakisan tembaga. Suhu dimana gris tersebut menjadi cecair diuji dengan menggunakan radas 'dropping point'. Komposisi asid stearik, lithium hidroksida monohidrat dan juga minyak masak terpakai memberikan kesan yang besar dalam menentukan kekerasan gris, kestabilan oksidatif dan juga suhu gris menjadi cecair. NLGI 2 gris diformulasi dalam eksperimen ini dengan keadaan yang optimum.

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CHAPTER 1

INTRODUCTION

1.1   Background of study

The search for environmental friendly materials that have the potential to substitute mineral oil in various industrial applications is currently being considered as top priority in fuel and energy management. This is largely due to the rapid depletion of world fossil fuel reserves and also increasing in awareness of environmental pollution from excessive mineral oil use and its disposal. A renewable resource such as vegetable oil is being considered as potential replacements for mineral oil base stocks in certain lubricant applications where immediate contact with the environment is expected. The nontoxic and biodegradable characteristic of vegetable oil-based lubricants will cause less danger to environment in case of accidental spillage or during disposal of the material. (A. Adhvaryu et al., 2005)

One of major product from the mineral oil is grease where the extensive usage increasing tremendously since nineteenth century. Grease is a preferred form of lubricant in certain applications because it gives low friction, easily confined, and has a long lubricating life at low cost. It is actually a lubricant of higher initial

viscosity than oil consisting originally of a calcium, sodium or lithium soap jelly emulsified with mineral oil. In general, grease consists of oil and/or other fluid lubricant that is mixed with another thickener substance, a soap, to form a solid. Specifically it contains 80% - 90% of oil. Soaps are the most common emulsifying agent used, and the type of soap depends on the conditions in which the grease is applied for. Various soaps provide differing levels of temperature resistance (relating to both viscosity and volatility), water resistance, and chemical reactivity.

Instead of using mineral oil as base oil to produce grease, vegetable oil is believed to be a potential source to meet the purpose. Development of vegetable oil based grease has been an area of active research for several decades (A. Adhvaryu et al., 2005). These products are very desirable in total loss lubricants such as railroads since their release will not cause any harm to the environment when it come in contact with soil or water.

Nowadays, instead of throwing or discarding it into the drain, the waste vegetable oil can be recycled into other precious material such as biodiesel and grease. While transforming from waste into wealth, it will cost minimal cost if compared to the mineral oil based grease. In addition, it is biodegradable and environmentally friendly.

Currently, waste vegetable oil in the United States is recycled for animal feed, pet food, and cosmetics. Since 2002, an increasing number of European Union countries have prohibited the inclusion of waste vegetable oil from catering in animal feed. Waste cooking oils from food manufacturing, however, as well as fresh or unused cooking oil, continues to be used in animal feed until today. (http://en.wikipedia.org/wiki/Lubricants)

Utilizing bio-based grease can reduce the environmental pollution in our country. It is estimated that 40% of all lubricants are released into the environment. (A. Adhvaryu et al., 2005)

Recycling, burning, landfill and discharge into water may achieve disposal of used lubricant. There are typically strict regulations in most countries regarding disposal in landfill and discharge into water as even small amount of lubricant can contaminate a large amount of water.

Most regulations permit a threshold level of lubricant that may be present in waste streams and companies spend hundreds of millions of dollars annually in treating their waste waters to comply with acceptable levels. Burning generates both airborne pollutants and ash rich in toxic materials, mainly heavy metal compounds. Thus lubricant burning takes place in specialized facilities that have incorporated special scrubbers to remove airborne pollutants and have access to landfill sites with permits to handle the toxic ash.

Unfortunately, most lubricant that ends up directly in the environment is due to general public discharging onto the ground, into drains and directly on landfills as trash. Other direct contamination sources include runoff from roadways, accidental spillages, natural or man-made disasters and pipeline leakages.

One of method for recycling waste lubricant is filtration process. Improvement in filtration technology has now made recycling a viable option (with rising price of base stock and crude oil). Typically various filtration systems remove particulates, additives and oxidation products and recover the base oil. The oil may get purified during the process. This base oil is then treated much the same as virgin base oil however there is considerable reluctance to use recycled oils as they are generally considered inferior. Base stock fractionally vacuum distilled from spent lubricants has superior properties to all natural oils, but cost effectiveness depends on many factors.

Spent lubricant may also be used as refinery feedstock to become part of crude oil. Again there is considerable reluctance to this as the additives, soot and wear metals will seriously poison/deactivate the critical catalysts in the process. Cost

prohibits carrying out both filtration (soot, additives removal) and re-refining (distilling, isomerisation, hydrocrack) however the primary hindrance to recycling still remains the collection of fluids as refineries need continuous supply in amounts measured in cisterns, rail tanks.

Occasionally, unused lubricant requires disposal. The best course of action in such situations is to return it to the manufacturer where it can be processed as a part of fresh batches. In environmental aspect, lubricants both fresh and used can cause considerable damage to the environment mainly due to their high potential of serious water pollution. Further the additives typically contained in lubricant can be toxic to flora and fauna. In used fluids the oxidation products can be toxic as well. Lubricant persistence in the environment largely depends upon the base fluid, however if very toxic additives are used they may negatively affect the persistence. (http://en.wikipedia.org/wiki/Lubricants)

Thus, to decrease the pollution, the utilization of alternative and environmental friendly grease is one of the solutions to overcome the problems. Bio-based greases are non-toxic making them the environmental alternative which is safe for both users and the environment.

1.2   Problem statement

Most current lubricants contain petroleum base stocks, which are toxic to environment and difficult to dispose of after use. Environmental concern continues to increase of pollution from excessive lubricant use and disposal, especially total loss lubricants. Over 60% of the lubricants used in the United States are lost to the environment (S.Z. Erhan et al., 2006). Vegetable oils have a capability to contribute

towards the goal of energy independence and security since they are a renewable resource. Vegetable oils with high oleic content are considered to be potential candidates as substitutes for conventional mineral oil-based lubricating oils and synthetic esters (Randles and Wright, 1992; Asadauskas et al., 1996).

Vegetable oils as lubricants are environmentally preferred to petroleum-based oil because they are biodegradable and non-toxic (Randles and Wright, 1992; Battersby et al., 1998). Other advantages include very low volatility due to the high molecular weight of the triglyceride molecule and excellent temperature– viscosity properties. Their polar ester groups are able to adhere to metal surfaces, and therefore, possess good lubricity. In addition, vegetable oils have high solubilizing power for polar contaminants and additive molecules. (S.Z. Erhan et al., 2006)

Development of vegetable oil-based greases has been an area of active research for several decades. As a result of active research, environmentally friendly lubricants and greases are already in the market. (Sharma et al., 2006)

However, all the researches that have been carried out are only considering vegetable oil as the raw material in producing grease. On the other hand, this research is concentrating waste cooking oil as the base oil stock for the production of grease. The comparison between proposed grease and available researches data is made in order to determine the quality of produced grease. This is to check whether waste vegetable oil will produce the same quality of grease compared to the available researches. It is known that, the advantages of using bio-based grease are biodegradable, environmental friendly and cheaper. Besides, by utilizing waste vegetable oil, the cost will be cheaper compared to virgin vegetable oil.

1.3   Objectives

The objective of this research is to produce grease from waste cooking oil via saponification of a fatty acid (stearic acid) with a metal hydroxide, which is lithium hydroxide monohydrate.

1.4   Scopes of study

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

i.   To study the effect of fatty acids composition, metal hydroxide and base oil quantity in controlling the grease hardness and formulation.

ii.   To study the formulation effect on the characteristics of produced bio-based grease in term of oxidative property, dropping point and penetration.

iii.   To study the effect of operating temperature in producing bio-based grease.

iv.   To characterize the produced bio-based grease and compare with commercial grease.

CHAPTER 2

LITERATURE REVIEW

2.1   Historical background on bio-based grease production

The first grease that been formulated were bio-based during 1400 B.C. which tallow was utilized to lubricate chariot wheels. From the year of 1859, there was petroleum grease were manufactured. An estimation of 98% of the petroleum grease dominated the marketplace in 2004. (Biresaw and Mittal, 2008)

Recently, due to increasing in petroleum prices, the use of bio-based oleochemicals as lubricant fluids, metalworking fluids and greases has increased dramatically. There were many researchers doing researches in formulating bio-based grease. The raw material that had been utilized was fresh vegetable oil such as soybean oil, cottonseed oil, canola oil, palm oil and epoxidized vegetable oil. (Biresaw and Mittal, 2008)

2.2   Raw materials

In formulation of bio-based grease, the raw materials that had been utilized in all of the researches done was fresh vegetable oil such as canola oil, palm oil, soybean oil and epoxidized vegetable oil.

Vegetable oils with high oleic content are potential substitutes for conventional petroleum-based lubricating oils and synthetic esters (Randles and Wright, 1992; Asadauskas et al., 1996). Vegetable oils are preferred as lubricants over synthetics because, unlike mineral-based oils, they are biodegradable, nontoxic, renewable, and relatively inexpensive (Battersby, N. et al. 1998; Asadauskas et al., 1996).

As lubricants, vegetable oils have very low volatility due to the high molecular weight of the triacylglycerol molecule, and they have a narrow range of viscosity changes with temperature. Polar ester groups enable the molecules to adhere to metal surfaces, and thereby impart good boundary lubrication to machined parts. In addition, vegetable oils have high solubilizing power for polar contaminants and additive molecules. (A. Adhvaryu et al., 2005)

In waste cooking oil, the free fatty acids content in it is much higher than in the fresh vegetable oil and it is more viscous than fresh vegetable oil. (Rafiqul Islam et al., 2008)

2.3   Grease

2.3.1   Grease component

Lubricating greases are semisolid colloidal dispersions of a thickening agent in a liquid lubricant matrix. They owe their consistency to a gel-forming network where the thickening agent is dispersed in the lubricating base fluid. Greases may include various chemical additives for specific property enhancement. As stated in Figure 2.1, a typical grease composition contains 60-95 wt. % base fluid (mineral, synthetic, or vegetable oil), 5-25 wt. % thickener (fatty acid soaps of alkali or alkaline metals), and 0-10 wt. % additives (antioxidants, corrosion inhibitors, anti-wear/extreme pressure, antifoam, tackiness agents).

Figure 2.1: Typical composition of grease

The base fluid imparts lubricating properties to the grease, whereas the thickener, essentially the gelling agent, holds the matrix together. This is a two-stage process. First, the absorption and adhesion of base oil in the soap structure results, and second, the soap structure swells when the remaining oil is added to the reaction mixture. Therefore, it is important to understand the structure and composition of the base fluid and thickener because in combination they can affect most of the physical and chemical properties of greases. (A. Adhvaryu et al., 2004)

Lubricating greases are generally highly structured suspensions, consisting of a thickener dispersed in mineral or synthetic oil. Fatty acid soaps of lithium, calcium, sodium, aluminum, and barium are most commonly used as thickeners. This component is added to increase the consistency of greases, preventing loss of lubricant under operating conditions and avoiding the penetration of contaminants, such as solid particles and water, without a significant reduction of the lubricating properties, which are mainly supplied by the oil. The thickener forms an entanglement network, which traps the oil and confers the appropriate rheological and tribological behavior to the grease. The performance of lubricating grease depends on the nature of its components and the microstructure achieved during its processing. Consequently, suitable structural and physical properties may be reached from a proper selection of the ingredients but, also, from a process optimization, as was previously reported. (J.M. Franco et al., 2006)

2.3.2   Grease characteristics

The semisolid nature of lubricating grease has several advantages over lubricating oils. Oxidative stability and consistency of the grease matrix control a wide variety of performance properties in grease lubrication: the ability to flow under force and subsequently lubricate hard-to-reach points; lowered friction coefficients through adhesion on the surface; effectiveness over a wide temperature range; water stability; acting as a physical barrier to seal out contaminants; decrease in dripping and spattering; decrease in frequency of relubrication (acts as sink for lubricating oils). It is important to note that grease structure and composition undergo significant modification while working by shearing and oxidation. The usefulness of grease in a particular application is controlled to a large extent by the

ability of the grease to sustain changes in temperature, pressure, operating environment, and shearing force. (A. Adhvaryu et al., 2004)

2.3.2.1 Apparent viscosity

At start-up, grease has a resistance to motion, implying a 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 "apparent viscosity". Apparent viscosity is the viscosity of a grease that holds only for the shear rate and temperature at which the viscosity is determined.

2.3.2.2 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. Syneresis is a special form of bleeding

caused by shrinking or rearrangement of the structure due to physical or chemical changes in the thickener.

2.3.2.3 Consistency, penetration, and National Lubricating Grease Institute (NLGI) numbers

The 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. 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 25^0C (77^0F). The depth, in tenths of a millimeter, to which the cone sinks into the grease, is the penetration. A penetration of 100 would represent solid grease while one 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 each classification.

Table 2.1: NLGI grease classifications

NLGI number	ASTM Worked Penetration 0.1 mm (3.28×10^{-4} ft) at 25^0C (77^0F)	Consistency
000	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

2.3.2.4 Contaminants

Greases tend to hold solid contaminants on their outer surfaces and protect lubricated surfaces from wear. If the contamination becomes excessive or eventually works its way down to the lubricated surfaces the reverse occurs, the grease retains abrasive materials at the lubricated surface and wear occurs.

2.3.2.5 Corrosion- and rust-resistance

This denotes the ability of grease to protect metal parts from chemical attack. The natural resistance of grease depends upon the thickener type. Corrosion-resistance can be enhanced by corrosion and rust inhibitors.

2.3.2.6 Dropping point

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. A few greases have the ability to regain their original structure after cooling down from the dropping point.

2.3.2.7 Evaporation

The mineral oil in grease evaporates at temperatures above 177^0C (350^0F). Excessive oil evaporation causes grease to harden due to increased thickener concentration. Therefore, higher evaporation rates require more frequent relubrication.

2.3.2.8 Fretting wear and false brinelling

Fretting is friction wear of components at contact points caused by minute oscillation. The oscillation is so minute that grease is displaced from between parts but is not allowed to flow back in. Localized oxidation of wear particles results and wear accelerates. In bearings, this localized wear appears as a depression in the race caused by oscillation of the ball or roller. The depression resembles that which occurs during Brinell hardness determination, hence the term "false brinelling". An example would be fretting wear of automotive wheel bearings when a car is transported by train. The car is secured, but the vibration of the train over the tracks causes minute oscillation resulting in false brinelling of the bearing race.

2.3.2.9 Oxidation stability

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

2.3.2.10 Pumpability and slumpability

Pumpability is the ability of a grease to be pumped or pushed through a system. More practically, pumpability is the ease with which pressurized grease can flow through lines, nozzles, and fittings of grease-dispensing systems. Slumpability, or feedability, is its ability to be drawn into (sucked into) a pump. Fibrous greases

tend to have good feedability but poor pumpability. Buttery-textured greases tend to have good pumpability but poor feedability.

2.3.2.11 Shear stability

Grease consistency may change as it is mechanically worked or sheared between wearing surfaces. Grease's ability to maintain its consistency when worked is its shear stability or mechanical stability. Grease that softens as it is worked is called thixotropic. Greases that harden when worked are called rheopectic.

2.3.2.12 High-temperature effects

High temperatures harm greases more than they harm oils. Grease, by its nature, cannot dissipate heat by convection like circulating oil. Consequently, without the ability to transfer away heat, excessive temperatures result in accelerated oxidation or even carbonization where grease hardens or forms a crust. Effective grease lubrication depends on the grease's consistency. High temperatures induce softening and bleeding, causing grease to flow away from needed areas. The mineral oil in grease can flash, burn, or evaporate at temperatures above $177^{0}C$ ($350^{0}F$). High temperatures, above $73\text{-}79^{0}C$ ($165\text{-}175^{0}F$), can dehydrate certain greases such as calcium soap grease and cause structural breakdown. The higher evaporation and dehydration rates at elevated temperatures require more frequent grease replacement.

2.3.2.13 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. As a guideline, the base oil's pour point is considered the low-temperature limit of grease.

2.3.2.14 Texture

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:

   i.   Brittle: the grease ruptures or crumbles when compressed.
   ii. Buttery: the grease separates in short peaks with no visible fibers.
   iii. Long fiber: the grease stretches or strings out into a single bundle of fibers.
   iv. Resilient: the grease can withstand moderate compression without permanent deformation or rupture.
   v.   Short fiber: the grease shows short break-off with evidence of fibers.
   vi. Stringy: the grease stretches or strings out into long, fine threads, but with no visible evidence of fiber structure.

2.3.2.15 Water resistance

This is the ability of grease to withstand the effects of water with no change in its ability to lubricate. Soap/water lather may suspend the oil in the grease,

forming an emulsion that can wash away or, to a lesser extent, reduce lubricity by diluting and changing grease consistency and texture. Rusting becomes a concern if water is allowed to contact iron or steel components.

2.3.3 Type of grease

The most common type of greases are described below.

2.3.3.1 Calcium grease

2.3.3.1.1 Calcium or lime grease

The first of the modern production grease 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 79^0C (175^0F) 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 93^0C (200^0F). 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. If

calcium grease is prepared from 12-hydroxystearic acid, the result is anhydrous (waterless) grease. Since dehydration is not a concern, anhydrous calcium grease can be used continuously to a maximum temperature of around 110^0C (230^0F).

2.3.3.1.2 Calcium complex grease

It 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 260^0C (500^0F) can be obtained and the maximum usable temperature increases to approximately 177^0C (350^0F). 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.

2.3.3.2 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 121^0C (250^0F), 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.

2.3.3.3 Aluminum grease

2.3.3.3.1 Aluminum soap grease

It is normally clear and has a somewhat stringy texture, more so when produced from high-viscosity oils. When heated above 79^0C (175^0F), this stringiness increases and produces a rubberlike 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.

2.3.3.3.2 Aluminum complex grease

It has a maximum usable temperature of almost 100^0C (212^0F) higher than aluminum-soap greases. It has good water-and-chemical resistance but tends to have shorter life in high-temperature, high-speed applications.

2.3.3.4 Lithium grease

2.3.3.4.1 Lithium soap 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 204^0C (400^0F) and can be used at temperatures up to about 135^0C (275^0F). It can also be used at temperatures as low as -35^0C (-31^0F). 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 good 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.

2.3.3.4.2.Lithium complex grease

Lithium complex grease and lithium soap grease have similar properties except the complex grease has superior thermal stability as indicated by a dropping point of 260^0C (500^0F). It is generally considered to be the nearest thing to a true multipurpose grease.

2.3.3.5 Other grease

Thickeners other than soaps are available to make greases. Although most of these are restricted to very special applications, two nonsoap greases are worthy of mention. One is organic, the other inorganic.

2.3.3.5.1 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 oilsoluble 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 177^0C (-4 to 350^0F) 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.

2.3.3.5.2 Polyurea complex grease

It 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.

2.3.3.5.3 Organo-clay

It 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 177^0C (350^0F). However, with frequent grease changes, this multipurpose grease can operate for short periods at temperatures up to its dropping point, which is about 260^0C (500^0F). A disadvantage is that greases made with higher-viscosity oils for high thermal stability will have poor low temperature performance. Organo-clay grease has excellent water-resistance but requires additives for oxidation and rust resistance. Work stability is fair to good. Pumpability and resistance to oil separation are good for this buttery textured grease.

2.3.4   Additives types

An additive may function in any of the following three ways:

i.   Protecting lubricated surfaces. Extreme pressure (EP) additives and rust inhibitors are included in this category. These additives coat the lubricated surfaces and prevent wear or rust.

ii.   Improving performance. Viscosity index improvers and antifoaming agents are examples. They make the oil perform in a desired manner for specific applications.

iii.   Protecting the lubricant itself. Antioxidants reduce the tendency of oil to oxidize and form sludge and acids.

The most common additives are listed in Table 2.2, and they are discussed individually in the following paragraphs.

2.3.4.1 Surface Additives

The primary purpose of surface additives is to protect lubricated surfaces. Extreme pressure additives, rust and corrosion inhibitors, tackiness agents, antiwear additives, and oiliness additives are included in this category. These additives coat the lubricated surfaces to prevent wear or rust.

2.3.4.1.1 Rust inhibitors.

Rust inhibitors are added to most industrial lubricants to minimize rusting of metal parts, especially during shipment, storage, and equipment shutdown. Although oil and water do not mix very well, water will emulsify, especially if the oil contains polar compounds that may develop as the oil ages. In some instances the water will remain either suspended by agitation or will rest beneath the oil on machine surfaces when agitation is absent. Rust inhibitors form a surface film that prevents water from making contact with metal parts. This is accomplished by making the oil adhere better or by emulsifying the water if it is in a low concentration.

2.3.4.1.2 Corrosion inhibitors

Corrosion inhibitors suppress oxidation and prevent formation of acids. These inhibitors form a protective film on metal surfaces and are used primarily in internal combustion engines to protect alloy bearings and other metals from corrosion.

2.3.4.1.3 Extreme pressure (EP) agents

Extreme pressure agents react with the metal surfaces to form compounds that have a lower shear strength than the metal. The reaction is initiated by increased temperature caused by pressure between asperities on wearing surfaces. The reaction creates a protective coating at the specific points where protection is required. This coating reduces friction, wear, scoring, seizure, and galling of wear surfaces. Extreme pressure additives are used in heavy loading or shock loading applications such as turbines, gears, and ball and roller bearings.

2.3.4.1.4 Tackiness agents

In some cases, oils must adhere to surfaces extremely well. Adding polymers composed of long-chain molecules or aluminum soaps of long-chain fatty acids increases the tackiness or adhesiveness of oils.

2.3.4.1.5 Antiwear (AW) agents

Additives that cause an oil to resist wear by coating the metal surfaces are called antiwear agents. Molecules of the antiwear compound are polar and attach (adsorb) themselves to metal surfaces or react mildly with the metal. When boundary lubrication conditions (direct contact between metal asperities) occur, such as in starting and stopping of machinery, these molecules resist removal more than ordinary oil molecules. This reduces friction and wear. However, they are effective only up to about 250^0C (480^0F).

2.3.4.1.6 Detergents and dispersant

Detergents and dispersant are used primarily in internal combustion engines to keep metal surfaces clean by preventing deposition of oxidation products.

2.3.4.1.7 Compounded oil

A small amount of animal fat or vegetable oil added to a mineral oil will reduce the coefficient of friction without affecting the viscosity. The ability of oil to provide a lower coefficient of friction at a given viscosity is often called oiliness or lubricity. When fatty oil is added to obtain this quality of oiliness, the lubricant is called compounded oil. Fatty oil adheres to metal more strongly than mineral oil and provides a protective film. Compounded oils are generally used in worm gears.

2.3.4.2 Performance-Enhancing Additives

These additives improve the performance of lubricants. Viscosity index improvers, antifoaming agents, emulsifiers, demulsifiers, and pour-point depressants are examples.

2.3.4.2.1 Pour-point depressants.

Oil's pour point is the temperature at which the oil ceases to flow under the influence of gravity. In cold weather, oil with a high pour point makes machinery

startup difficult or impossible. The stiffness of cold oil is due to paraffin waxes that tend to form crystal structures. Pour-point depressants reduce the size and cohesiveness of the crystal structures, resulting in reduced pour point and increased flow at reduced temperatures.

2.3.4.2.2 Viscosity index (VI) improvers

The viscosity index is an indicator of the change in viscosity as the temperature is changed. The higher the VI, the less the viscosity of an oil changes for a given temperature change. Viscosity index improvers are used to limit the rate of change of viscosity with temperature. These improvers have little effect on oil viscosity at low temperatures. However, when heated, the improvers enable the oil viscosity to increase within the limited range permitted by the type and concentration of the additive. This quality is most apparent in the application of multigrade motor oils.

2.3.4.2.3 Emulsifiers

In most industrial applications it is undesirable to have emulsified water in the oil. However, soluble oils require emulsifiers to promote rapid mixing of oil and water and to form stable emulsions. Soluble oils are used as lubricants and coolants for cutting, grinding, and drilling applications in machine shops.

2.3.4.2.4 Demulsifiers

Demulsifiers promote separation of oil and water in lubricants exposed to water.

2.3.4.3 Lubricant Protective Additives

Lubricant protective additives are employed to protect the lubricant instead of the equipment. Oxidation inhibitors and foam inhibitors are examples.

2.3.4.3.1 Oxidation inhibitors

Over time, hydrocarbon molecules will react to incorporate oxygen atoms into their structure. This reaction produces acids, sludge, and varnish that foul or damage metal parts. At low temperatures and under minimal exposure to oxygen, this process is very slow. At temperatures above 82^0C (180^0F) the oxidation rate is doubled for every -7.78 to -6.67^0C (18 to 20^0F) rise in temperature. Oxidation of hydrocarbons is a very complex chemical process and depends on the nature of the oil. Oxidation inhibitors reduce the quantity of oxygen reacting with oil by forming inactive soluble compounds and by passivating metal-bearing surfaces to retard the oxidation rate. As previously noted, oxidation inhibitors are consumed as the oil ages. Oil condition should be monitored periodically to ensure that essential additives are maintained at safe levels. Oxidation inhibitors are used in most industrial lubricant applications where oil is continuously circulated or contained in housing.

2.3.4.3.2 Foam inhibitors

In many applications, air or other gases may become entrained in oil. Unless these gases are released, foam is produced. Foaming can result in insufficient oil delivery to bearings, causing premature failure. Foam may also interfere with proper operation of equipment such as lubricating pumps and may result in false oil level readings. Under some circumstances foam may overflow from oil reservoirs. Foam inhibitors such as silicone polymers or polyacrylates are added to reduce foaming.

Table 2.2: Most common additives

Main Type	Function and Subtypes
Acid neutralizers	Neutralize contaminating strong acids formed, e.g., by combustion of high sulfur fuels or by decomposition of active EP additives.
Antifoam	Reduce surface foam.
Antioxidants	Reduce oxidation. Various types are: oxidation inhibitors, retarders; anticatalyst metal deactivators, metal passivators.
Antirust	Reduce rusting of ferrous surfaces swept by oil.
Antiwear agents	Reduce wear and prevent scuffing of rubbing surfaces under steady load operating conditions.
Corrosion inhibitors	Type (a) reduces corrosion of lead; type (b) reduces corrosion of cuprous metals.
Detergents	Reduce or prevent deposits formed at high temperatures, e.g., in internal combustion engines.
Dispersant	Prevent deposition of sludge by dispersing a finely divided suspension of the insoluble material formed at low temperature.
Emulsifiers	Form emulsions; either water-in-oil or oil-in-water, according to type.
Extreme pressure	Prevent scuffing of rubbing surfaces under severe operating conditions, e.g., heavy shock load, by formation of a mainly inorganic surface film.
Oiliness enhancers	Reduce friction under boundary lubrication conditions; increase load-carrying capacity where limited by temperature rise by formation of mainly organic surface films.
Pour-point depressants	Reduce pour point of paraffinic oils.
Tackiness agents	Reduce loss of oil by gravity, e.g., from vertical sliding surfaces, or by centrifugal force.
Viscosity index improvers	Reduce the decrease in viscosity due to increase of temperature.

(http://www.usace.army.mil/publications/eng-manuals/em1110-2-1424/toc.htm)

2.3.5 Thickeners

Lubricating greases are generally highly structured suspensions, consisting of a thickener dispersed in mineral or synthetic oil. This component is added to increase the consistency of greases, preventing loss of lubricant under operating conditions and avoiding the penetration of contaminants, such as solid particles and water, without a significant reduction of the lubricating properties, which are mainly supplied by the oil. The thickener forms an entanglement network, which traps the oil and confers the appropriate rheological and tribological behavior to the grease. (J.M. Franco et al., 2006)

Typical thickening agents used in lubricating greases include the alkali metal soaps, clays, polymers, asbestos, carbon black, silica gels and aluminum complexes. Soap thickened greases constitute the largest segment by far of the commercially available greases. (http://www.freepatentsonline.com/4392967.html)

CHAPTER 3

METHODOLOGY

3.1   Introduction

In this chapter, it covers the materials that will be used to conduct the research, how to synthesis the grease and the grease analysis method.

3.2   Material

Waste vegetable oil used as a base fluid, stearic acid, lithium hydroxide monohydrate and sodium diethyl dithiocarbamate as antioxidant additive.

i.   Waste vegetable oil was taken from Universiti Malaysia Pahang's cafeteria. This waste was filtered prior to synthesis process.

ii.   Stearic acid from Merck.

iii.   Lithium hydroxide monohydrate from Merck.

iv.   Sodium diethyl dithiocarbamate from Merck.

3.3   Synthesis of grease

200 mL waste cooking oil, 0.75 g of stearic acid and 3 g of additives were put in a 1000 mL beaker. Then, it was heated on a hot plate and mechanically stirred using a mixer blade until the temperature of 100^0C is reached. After that, 1 g of lithium hydroxide monohydrate was added to the mixture. It was then heated until the temperature of 180^0C is reached. After the temperature was reached, the mixture's temperature was maintained for about one hour. Then, it was gradually cooled down to room temperature. A mixture of lithium hydroxide monohydrate and stearic acid was taken in 1:0.75, 1:0.85, 1:0.95, 1:1 with equivalent weight ratio of the lithium hydroxide, and the amount of waste cooking oil and additive were constant. The heating temperature of the mixture was varied from 180^0C, 200^0C, 220^0C and 240^0C. This synthesis of grease process was taken place in a fume hood. Figure 3.1 shows the flow process of the synthesis of grease.

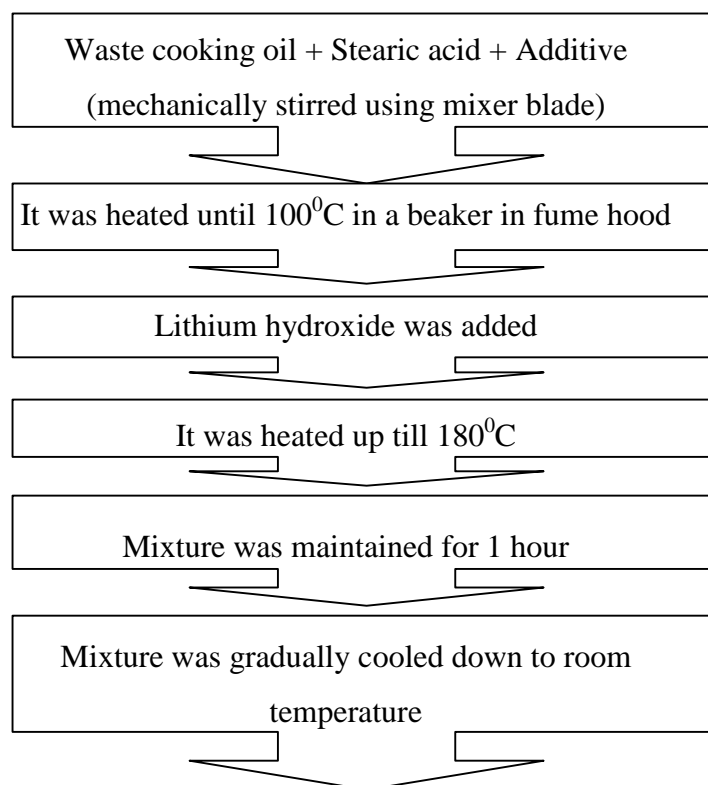| Waste cooking oil + Stearic acid + Additive (mechanically stirred using mixer blade) |
| It was heated until 100^0C in a beaker in fume hood |
| Lithium hydroxide was added |
| It was heated up till 180^0C |
| Mixture was maintained for 1 hour |
| Mixture was gradually cooled down to room temperature |

Figure 3.1: Synthesis of grease flow process

3.4   Analysis method

The analysis method consisted of three methods which were depth penetration test, dropping point apparatus and also copper corrosion test.

3.4.1   Depth penetration test

Depth penetration test is performed on petroleum products to determine consistency and shear stability (lubricating greases) for design, quality control and identification purposes.

The grease was placed in a penetration test apparatus. This apparatus consists of a container, a specially-configured cone and a dial indicator. The container was filled with the grease and the top surface of the grease was smoothed over. The cone was placed so that its tip just touches the grease surface and a dial indicator was set to zero at this position. A standard cone or needle was released from a penetrometer and allowed to drop freely into the sample for 5 seconds. The depth of penetration of the cone or needle into the sample was measured in tenths of a millimeter by the penetrometer. The analysis procedure followed ASTM standard D-217.

3.4.2   Dropping point

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. A few greases have the ability to regain their original structure after cooling down from the dropping point.

The test apparatus consists of a grease cup chromium plated brass with a small hole in the bottom, test tube with indentations, cork ring guide, two thermometer corks, thermometer depth gauge, two thermometers, a 400 mL Pyrex beaker, polished metal rod, stirring device with an electric heater. The inside surface of the grease cup was coated with the grease to be tested. A thermometer was inserted into the cup and held in place so that the thermometer did not touch the grease. This assembly was placed inside a test tube. The test tube was lowered into the beaker which was filled with thermal oil. Another thermometer was inserted into the thermal oil. To execute a test, the beaker containing thermal oil and test cell were heated, while being stirred. The dropping point was the average temperature that was recorded on the test tube thermometer and the temperature of the thermal oil when a drop of grease fell through the hole in the grease cup. If the drop trails a thread, the dropping temperature was the temperature at which the thread breaks. The analysis procedure followed ASTM standard D-566.

3.4.3 Copper corrosion test

This copper corrosion test is use to indicate the corrosion resistance and also the oxidative stability of the grease.

The copper rod was put in a bottle containing the samples. After that it was immersed in a water bath at a temperature of 100^0C for about 24 hours. Then the colour changed of the copper rod was recorded and graded according to ASTM Copper Strip Corrosion Standard. The analysis procedure followed ASTM standard D-4048.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

In this chapter, results and findings from the experiment is discussed in detail. The discussion is divided into effect of lithium hydroxide monohydrate, stearic acid and waste cooking oil composition on the grease hardness, effect of operating temperature in producing grease, effect of lithium hydroxide monohydrate quantity to the dropping point temperature and effect of additives composition.

4.2 Effect of lithium hydroxide monohydrate, stearic acid and waste cooking oil composition in determining the grease hardness

The hardness of the grease is determined by regulating the quantity of the lithium hydroxide monohydrate, stearic acid and also the waste cooking oil composition in the grease mixture. When the quantity of the lithium hydroxide

composition is increased, this will also lead to decrease of the waste cooking oil percentage in the grease composition. The percentage of this formulation in getting the NLGI 2 grease is less than stated in previous study (S.Z. Erhan et al., 2006) which is 95% of the base oil. In this experiment, the waste cooking oil percentage in getting the NLGI 2 grease was 95.35% which is higher than stated in the previous study. This is due to utilizing waste cooking oil as base oil instead of using fresh cooking oil. The waste cooking oil had been utilized before and this contributes to the grease hardness. It is from the metal such as calcium and sodium as a contaminant in the waste cooking oil.

As the quantity of the lithium hydroxide monohydrate increases from 1g to 5g, the NLGI grease hardness increases from 0 to 6. Figure 4.1 shows the effect of lithium hydroxide monohydrate on the grease hardness. As mentioned earlier the purpose of the metal soap is acts as thickener which is the gelling agent of the grease mixture and also increases the consistency of the grease composition. It is reported before by S.Z. Erhan et al., 2006.
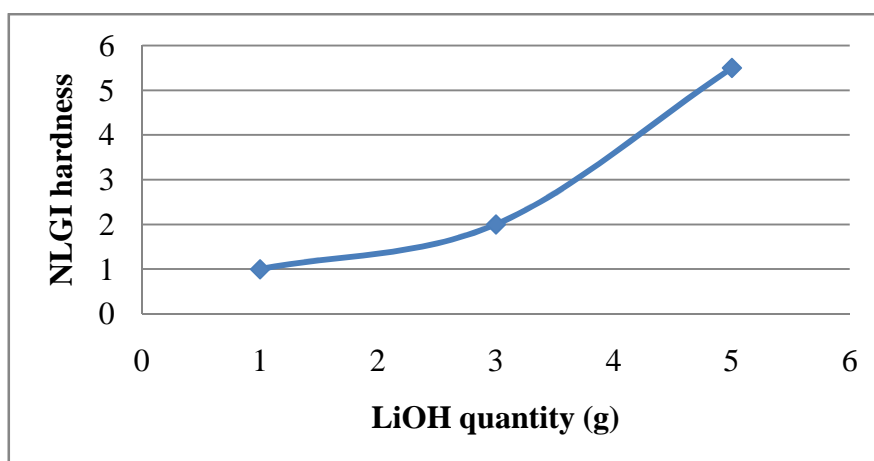

Figure 4.1: Effect of lithium hydroxide monohydrate quantity to the grease hardness

By increasing the ratio of the stearic acid in formulating the grease mixture, the NLGI grease hardness decreases from 2 to 1. Figure 4.2 shows the effect of stearic acid composition to the grease hardness. Results from previous study (A.

Adhvaryu et al., 2004) indicated that the optimum lithium hydroxide monohydrate to stearic acid equivalent ratio of 1:0.75 in the metal soap composition with soybean oil in the final formulation resulted in stable NLGI 2 grease as compared to one with 1:1 equivalent ratio. On the other hand, in this study, the 1:1 equivalent ratio is resulted in the NLGI 2 grease as compared to 1:0.75. This difference may be due to contaminant in the waste cooking oil such as calcium and sodium that already absorb in the waste cooking oil that also act as a thickener in the grease mixture. As in previous study, only fresh cooking oil is used and there is no contaminant in it.
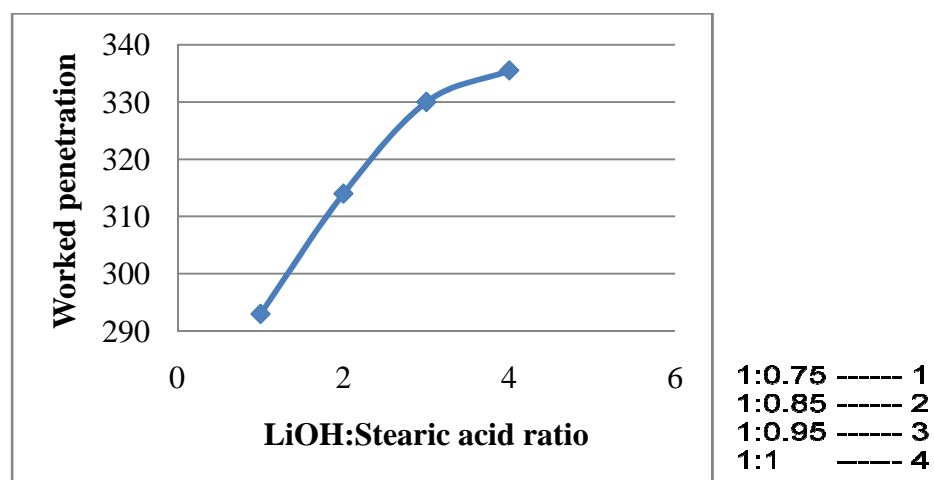

Figure 4.2: Effect of stearic acid composition to the grease hardness

4.3   Effect of operating temperature in producing grease

It is expected that the higher the operating temperature, the viscosity of the grease increased. (S.Z. Erhan et al., 2006) This can be seen in Figure 4.3 below. Figure 4.3 clearly illustrated the effect of operating temperature on grease hardness. The increased in operating temperature would result to the increase of NLGI hardness. This phenomenon is due to the molecule structure arrangement. When at

high temperature, the viscosity increase and decrease the mobility of the molecule and vice versa.
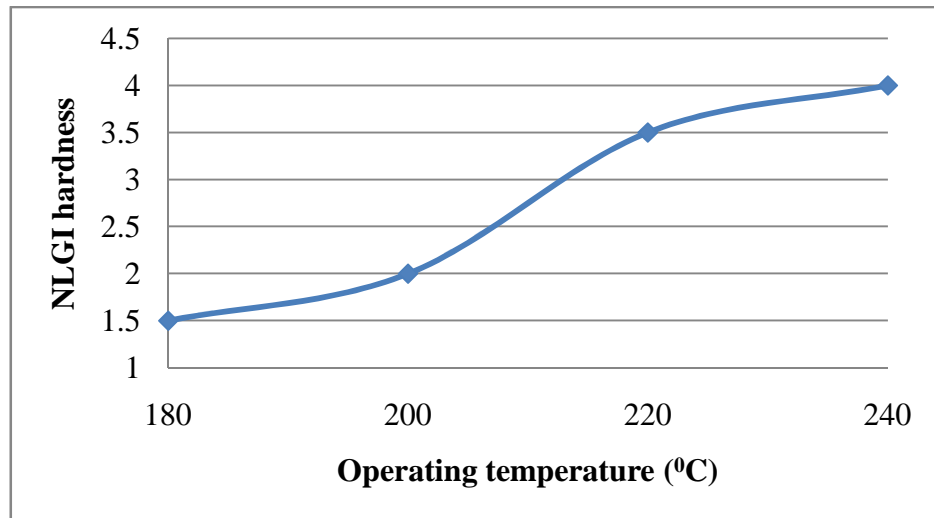
Figure 4.3: Effect of operating temperature to the grease hardness

4.4 Effect of lithium hydroxide monohydrate quantity to the dropping point temperature

As the lithium hydroxide monohydrate quantity increases from 1g to 5g, the dropping point temperature of the grease increases from 80^0C to 165^0C. As the percentage of the thickener increases, the grease hardness also increases (S.Z. Erhan et al., 2006) and this contribute to the higher dropping point temperature. As mentioned earlier, dropping point temperature is the temperature at which the grease becomes fluid enough to drip. If the percentage of the thickener is high, it will hold the base oil better and thus need higher temperature to change to fluid. Figure 4.4 shows the effect of lithium hydroxide monohydrate quantity to the dropping point temperature.
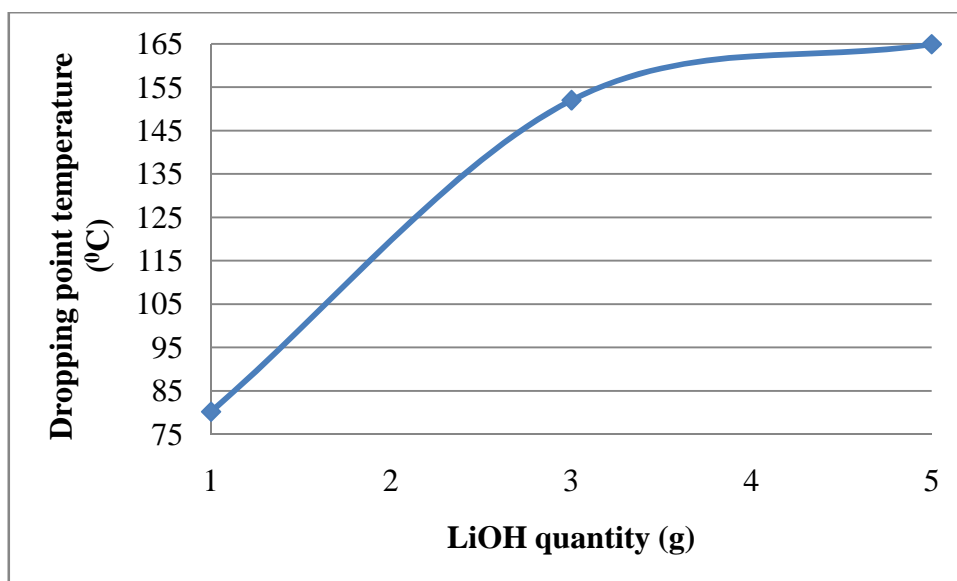
Figure 4.4: Effect of lithium hydroxide monohydrate quantity to the
dropping point temperature

4.5   Effect of additives composition

In this experiment, the additives composition that used was 1.59 wt. % and
3.13 wt. %. In the sample that used 1.59 wt. % of additives, the copper corrosion test
indicates grade slightly tarnished (1b) of the copper rod. On the other hand, the
sample that used 3.13 wt. % of additives, it indicates grade 4c which is stand for
corrosion. So, the optimum additives composition in this grease formulation is 1.59
wt. % which is still in the range of the typical additives composition which is 0-10
wt. % (S.Z. Erhan et al., 2006). This denotes the ability of this grease to protect
metal parts from chemical attack. Oxidation leads to carbonization and crust
formation on the metal surface (Biresaw and Mittal, 2008). Thus, this grease have
good oxidative stability. Table 4.1 indicates the grade of the grease corrosion effect.

Table 4.1: Effect of additives composition

Copper corrosion	Additives quantity (g)	Additives percentage (wt. %)
1b	3	1.59
4c	6	3.13

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1   Conclusion

As a conclusion, the lithium to fatty acid ratio and the nature of the fatty acid and base oil content can be carefully regulated and optimized to formulate grease with the desired hardness and stability. As long as the base oil is confined within the soap fiber network, NLGI no. 2 grease with good oxidative stability can be produced. Oxidative stability and other performance properties would fail if the base oil is released from the grease matrix due to overloading of soap with base oil. A higher metal to fatty acid ratio can result in greases with better oxidative stability. (S.Z. Erhan et al., 2006)

Table 5.1 indicates the comparison between the commercial grease and the grease that had been formulated in this study. The best operating temperature in producing this grease is 180^{0}C. The dropping point of the commercial grease is higher if compared to the grease sample. This may be due to percentage of the thickener is less if compared to the commercial grease. From this study, the percentage of the typical composition in the grease formulation using waste cooking oil   has   been   summarized   in   Table   5.2.

Table 5.1: Comparison between sample and commercial grease

- Sample –	- Commercial grease –
• NLGI type 2	• NLGI type 2
• Operating temperature – 180^{0}C	• Dropping point - 190^{0}C
• Dropping point - 160^{0}C	• Copper corrosion – slight tarnish
• % thickener – 3.11%	• Colour – yellowish
• % base oil – 95.34%	
• Copper corrosion – slight tarnish	
• Colour – blackish	

Table 5.2: Typical composition for grease

NLGI grade	Wt.% base oil	Wt.% thickener	Wt.% additive
0,1	97.35 – 97.48	0.93 – 1.058	1.59
2,3,4	95.34 – 95.71	2.73 – 3.11	1.55 – 1.56
5,6	93.4 – 94.97	3.48 – 5.08	1.52 – 1.55

5.2   Recommendation

In order to get better grease, the operating time of maintaining the operating temperature must be increased. This will lead to more time for the thickener to swell and absorb the base oil. Thus, stable grease matrix will be form.

Formulation of grease is a trial and error process. In this formulation using waste cooking oil as base oil, the lithium hydroxide monohydrate quantity should be increase to get the higher dropping point. Besides increasing the lithium hydroxide monohydrate quantity, the stearic acid ratio should also be increase to get the desired grease hardness.

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APPENDIX A

A)   Calculation for percentage of base oil thickener and additives

Sample 1

200 mL waste cooking oil + 1g LiOH + 0.75g stearic acid + 6g additives

Density of waste cooking oil = 920kg/m^3

So, mass of waste cooking oil = 184g

Mass of sample = 188.75g

Weight % of waste cooking oil = $\frac{184g}{1\ 175g}$ 100%   95.96%

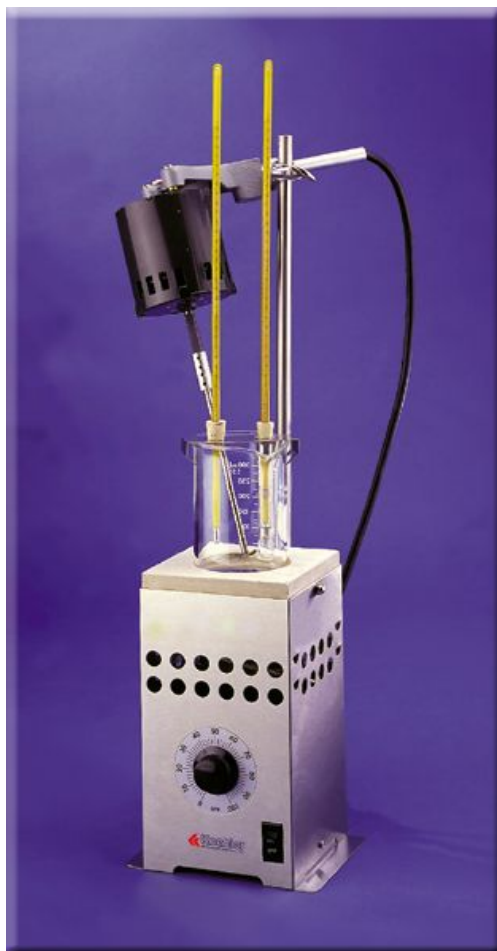The rest of the calculation is in the data table.

B)   Data

Sample	Wt.% thickener	Wt.% base oil	Wt.% additives
1	0.91	95.96	3.13
2,3,4,5	0.93	97.48	1.59
6,7,8,9	0.98	97.43	1.59
10,11,12,13	1.03	97.38	1.59
14,15,16,17	1.058	97.35	1.59
18,19,20,21	2.73	95.71	1.56
22,23,24,25	2.88	95.56	1.56
26,27,28,29	3.03	95.41	1.56
30,31,32,33	3.11	95.34	1.55
34,35,36,37	3.48	94.97	1.55
38,39,40,41	4.71	93.76	1.53
42,43,44,45	4.96	93.52	1.52
46,47,48,49	5.08	93.4	1.52

APPENDIX B

A)   Penetrometer

B)   Dropping point apparatus

C)   Copper corrosion standard