

**PRODUCTION OF GREASE FROM WASTE COOKING OIL**

**NORASMAH BINTI HAJI OTHMAN**

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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  
and support that made this work possible.

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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 aplikasi industri, alat-alat pertanian dan juga aplikasi 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 aplikasi 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