PRODUCTION OF GREASE FROM USED LUBRICANT: A FEASIBILITY STUDY

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A report submitted in partial fulfillment of the Requirement for the reward of the degree of Bachelor of Chemical Engineering

Faculty of Chemical Engineering and Natural Resources
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I declare that this thesis entitle "*Production of Grease from Used Lubricant*"
Is the result of my own research except as cited in the references. The thesis
has not been accepted for any degree and is not concurrently submitted
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Special Dedication of This Grateful Feeling to My…

Beloved mother;

Pn. Koret Bt Md Lim

Supportive Lecturer

Prof. Dr Rosli Mohd. Yunus, Mr. Zulkifli Jemaat, Ms Noralisa,

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ABSTRACT

Grease is a mixture of a fluid lubricant usually petroleum oil and a thickener (soap) dispersed in the oil. The base oil (petroleum) can be changed by using the used lubricant. Other then base oil, thickener may play an important role in the mixture. Soap thickeners are formed by reacting metallic hydroxide, or alkali, with a fat, fatty acid, or ester. Since the petroleum prices were increase each year, using used lubricant as base oil is the best solution to produce grease in the low cost at the same time it will decrease the water pollution. Three type of soap are used in this production that is aluminum soap, calcium soap and sodium soap. Viscometer was use in this experiment to analyze the quality and performances of grease product to select the best type of soap to produce grease. The viscosities of grease are test by change the spindle speed for each type of soap. The different percent of soap are use to know the effect of percent of soap to the viscosity of grease. In this experiment, grease was successfully produced from used lubricant. The viscometer test and sample preparation on grease was visualized in this research, where the viscosity of grease was decrease with the increasing of spindle speed and the viscosity was increase with the increasing of percentage soap (thickener) added. The best way to produce grease is with sodium soap, where sodium soap is the strongest thickener compare to another thickener. Using this type of soap, only small amount of this thickener needed compare to another type of soap.

ABSTRAK

Gris ialah campuran cecair pelincir kebiasaannya minyak petroleum dan agen pengental (sabun) kemudian dilarutkan ke dalam minyak. Minyak petroleum boleh digantikan dengan minyak pelincir terpakai. Selain daripada minyak petroleum, agen pengental memainkan peranan yang penting di dalam campuran tersebut. Agen pengental dihasilkan melalui tidakbalas logam hidroksida, atau alkali dengan lemak, asid lemak atau ester. Memandangkan harge petroleum meningkat sejak kebelakangan ini, pilihan terbaik adalah dengan menggunakan minyak pelincir terpakai sebagai pengganti kepada minyak petroleum, ia bukan sahaja dapat mengurangkan kos pengeluaran gris malahan dapat mengurangkan pencemaran air yang berpunca daripada pembuangan minyak pelincir terpakai ke dalam sungai. Tiga jenis sabun pengental yang digunakan dalam penghasilan gris dalam kajian ini adalah sabun aluminium, sabun calsium dan sabun natrium. Viscometer digunakan didalam kajian ini utuk menganalisis kualiti dan prestasi gris untuk pemilihan sabun pengental terbaik dalam penghasilan gris. Kelikatan gris tersebut dikaji dengan mengubah kelajuan pemutar untuk setiap jenis sabun pengental. Perbezaan peratusan sabun pengetal juga disukat kelikatannya untuk mengkaji kesan kuantiti sabun pengental kepada kelikatan gris tersebut. Didalam kajian ini gris telah berjaya dihasilkan dengan menggunakan minyak pelincir terpakai manggantikan minyak petroleum. Penghasilan gris dan analisis kelikatan telah direalisasikan didalam kajian ini, yang mana kelikatan gris berkurang dengan pertambahan kelajuan pemutar dan bertambah dengan pertambahan kuantiti sabun pengental. Cara yang terbaik untuk penghasilan gris adalah dengan menggunakan sabun natrium, ia adalah kerana natrium merupakan sabun pengetal yang terbaik dikalangan tiga jenis sabun pengental tersebut.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Grease is a semi fluid to solid mixture of a fluid lubricant, a thickener, and additives. The fluid lubricant that performs the actual lubrication can be petroleum (mineral) oil, synthetic oil, or vegetable oil. The thickener gives grease its characteristic consistency and holds the oil in place. Common thickeners are soaps and organic or inorganic non soap thickeners.

Soaps are the most common emulsifying agent used, and the type of soap depends on the conditions in which the grease is to be used. Different soaps provide differing levels of temperature resistance (relating to both viscosity and volatility), water resistance, and chemical reactivity. Powdered solids may also be used, such as clay, which was used to emulsify early greases and is still used in some inexpensive, low performance greases.

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. Grease has been described as a temperature-regulated feeding device, when the lubricant film between wearing surfaces thins, the resulting heat softens the adjacent grease, which expands and releases oil to restore film thickness.

Greases are employed where heavy pressures exist, where oil drip from the bearings is undesirable, and/or where the motions of the contacting surfaces are discontinuous so that it is difficult to maintain a separating lubricant film in the bearing. Grease-lubricated bearings have greater frictional characteristics at the beginning of operation. Under shear, the viscosity drops to give the effect of an oil-lubricated bearing of approximately the same viscosity as the base oil used in the grease.

1.2 Problem Statement

Grease is a mixture from a fluid lubricant (usually petroleum oil) and thickener soap). Nowadays the petroleum price were increase each year, one of the factors because the decreasing of oil reserve and the increasing of demand. Because of the increasing of raw material price, we need to find the solution to produce grease in the low cost.

Nowadays majority of developing countries face water pollution. The major source of water pollution is oil (petroleum and waste lubricants). Most of factories discard their waste into the river and sea. To rectify this situation we propose to recycle the waste to produce grease.

The aim of this research is to find the solution that can reduce the water pollution and at the same time can use the waste to produce grease. This project will benefit not only to the environment, but also the entrepreneur and society.

In this research grease will be produce by using used lubricant oil based on suitable method. The product will be testing their viscosity by using viscometer, to know the quality and the best method to produce grease.

1.3 Objective

Produce grease from used lubricant using different type of soap. Test the performance of the grease by testing viscosity of the product, to define the best type of soap to produce grease.

1.4 Scope

I. To produce the grease from used lubricant using different type of soap
II. To define the effect of percent soap to the viscosity of grease
III. To define the viscosity of the grease using different spindle speed

CHAPTER 2

LITERATURE REVIEW

2.1 Grease Background

Grease is a semi fluid of a fluid lubricant, a thickener, and additives. The fluid lubricant that performs the actual lubrication can be petroleum (mineral) oil, synthetic oil, or vegetable oil. 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. (John A., 1981)

Common thickeners are soaps and organic or inorganic no soap 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.

Grease has been described as a temperature-regulated feeding device, when the lubricant film between wearing surfaces thins, the resulting heat softens the adjacent grease, which expands and releases oil to restore film thickness.

2.2 Properties

Soap thickeners are formed by reacting a metallic hydroxide, or alkali, with a fat, fatty acid, or ester. The type of soap used depends on the grease properties desired. Calcium (lime) soap greases are highly resistant to water, but unstable at high temperatures. Sodium soap greases are stable at high temperatures, but wash out in moist conditions. Lithium soap greases resist both heat and moisture.

A mixed base soap is a combination of soaps, offering some of the advantages of each type. A complex soap is formed by the reaction of an alkali with a high-molecular weight fat or fatty acid to form soap, and the simultaneous reaction of the alkali with a short-chain organic or inorganic acid to form a metallic salt (the complexing agent). Complexing agents usually increase the dropping of grease.

Lithium, calcium, and sodium greases are common alkalis in complex soap greases (John A, 1981). Non soap thickeners, such as clays, silica gels, carbon black, and various synthetic organic materials are also used in grease manufacture. A multi purpose is designed to provide resistance to heat, as well as water, and may contain additives to increase load-carrying ability and reduce rust.

2.3 Function

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 drastically to the power required to operate the machine, mostly at startup. (Boehringer, 1992)

2.3.1 Applications suitable for grease.

Grease and oil are not the same. 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 from time to time or is in storage for an extended period of time. Because grease remains in place, a lubricating film can instantly form.
II. Machinery that is not easily reached for frequent lubrication. High-quality greases can lubricate isolated or relatively inaccessible components for extended periods of time without frequent replenishing.
III. Machinery operating under extreme conditions such as high temperatures and pressures, shock loads, or slow speed under heavy load. Under this situation, grease provides thicker film that is required to protect and sufficiently lubricate, whereas oil films can be too thin and can break.

2.3.2 Functional properties of grease.

I. Functions to minimize leakage and to keep out contaminants. Because of its stability, grease acts to prevent lubricant leakage and also to prevent entrance of corrosive contaminants and strange materials. It also acts to keep seals effective (whereas oil would simply seep away).

II. Easier to contain than oil. Oil lubrication can require an expensive system of circulating equipment and complex maintenance devices. In comparison, grease is good quality of stiffness, is easily confined with simplified, and less costly maintenance devices.

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

IV. Fluid level does not have to be controlled and monitored.

2.3.3 Disadvantages of grease:

I. Poor cooling. Grease cannot disperse heat by convection like circulating oil.

II. Resistance to motion. Grease has more resistance to motion at start-up than oil, so it is not appropriate for low torque or high speed operation.

III. More difficult to handle than oil for dispensing, draining, and refilling. Also, exact amounts of lubricant cannot be as easily metered.

2.4 Grease Characteristics

2.4.1 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. (Boehringer 1992)

To differentiate 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.

2.4.1.1 Bleeding, migration

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 conditions. 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. From (Boehringer 1992)

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

The most important feature of grease is its stiffness 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 changed by handling or working. ASTM D 217 and D 1403 methods measure penetration of worked 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 ºC (77 ºF). 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 semi fluid. 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.(NLGI lubricant grease guide 4th ed. 28 Feb 99)

Table 2.1: NLGI Grease Classification

NLGI Grease Classification		
NLGI Number	ASTM Worked Penetration 0.1 mm (3.28 × 10 ft) at 25 °C (77° F)	Consistency
000	446-475	Semi fluid
00	400-430	Semi fluid
0	255-385	Very soft
1	310-340	Soft
2	265-285	Common grease
3	220-250	Semi hard
4	175-205	Hard
5	130-160	Very hard
6	85-115	solid

2.4.1.3 Corrosion and rust resistance.

This is 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.

2.4.1.4 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.

2.4.1.5 Evaporation.

The mineral oil in grease evaporates at temperatures above 177 ºC (350 ºF). Excessive oil evaporation causes grease to freeze due to increased thickener concentration. Therefore, higher evaporation rates require more frequent regarding lubrication.

2.4.1.6 Oxidation stability.

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

2.4.1.7 High-temperature effects.

High temperatures damage greases more than they damage oils. Grease, by its nature, cannot disperse heat by convection like circulating oil. Therefore, without the ability to transfer away heat, extreme temperatures result in accelerates oxidation or even carbonization where grease hardens or forms a shell. Effective grease lubrication depends on the grease's consistency.

High temperatures bring 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 ºC (350 ºF). High temperatures, above 73-79 ºC (165-175 ºF), can dehydrate certain greases such as calcium soap grease and cause structural breakdown. The higher evaporation and dehydration rates at high temperatures require more frequent grease replacement.

2.4.1.8 Low-temperature effects.

If the temperature of grease is lower enough, it will become so viscous that it can be classified as hard grease. Pump ability 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 flow point is considered the low temperature limit of grease.

2.4.1.9 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.

2.4.1.10. Water resistance.

This is the ability of grease to hold up the effects of water with no change in its ability to lubricate. Soap or water may suspend the oil in the grease, forming a mixture 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.

2.5 Fluid Lubricants

Fluid lubricants used to formulate grease are normally petroleum or synthetic oils. For petroleum oils in general, naphthenic oils tend to chemically mix better with soaps and additives and form stronger structures than paraffinic oils. Synthetic oils are higher in first cost but are effective in high-temperature and low temperature extremes. With growing environmental concerns, vegetable oils and certain synthetic oils are also being used in applications requiring nontoxic or biodegradable greases. (marcel dekker 1985)

The base oil selected in formulating grease should have the same characteristics as if the equipment is to be lubricated by oil. For instance, lower-viscosity base oils are used for grease applications at lower temperatures or high speeds and light loads, whereas higher-viscosity base oils are used for higher temperatures or low speed and heavy load applications.

2.6 Soap Thickeners

Dispersed in its base fluid, a soap thickener gives grease its physical character. Soap thickeners not only provide consistency to grease, they affect desired properties such as water and heat resistance and pump ability. They can affect the amount of an additive, such as a rust inhibitor, required to obtain a desired quality. The soap influences how a grease will flow, change shape, and age as it is mechanically worked and at temperature extremes. Each soap type brings its own characteristic properties to grease.

The principal ingredients in creating soap are a fatty acid and an alkali. Fatty acids can be derived from animal fat such as beef tallow, lard, butter, fish oil, or from vegetable fat such as olive, castor, soybean, or peanut oils. The most common alkalis used are the hydroxides from earth metals such as aluminum, calcium, lithium, and sodium. Soap is created when a long-carbon-chain fatty acid reacts with the metal hydroxide.

The metal is included into the carbon chain and the resultant compound develops a polarity. The polar molecules form a fibrous network that holds the oil. Thus, a somewhat rigid gel-like material "grease" is developed. Soap concentration can be varied to obtain different grease thicknesses. Furthermore, viscosity of the base oil affects thickness as well. Since soap qualities are also determined by the fatty acid from which the soap is prepared, not all greases made from soaps containing the same metals are identical. The name of the soap thickener refers to the metal (calcium, lithium, etc.) from which the soap is prepared. From (Weiheim Wikey VCH, 2001)

2.7 Additives

An additive that mix together to form grease is as surface protecting and effectively improves the overall performance of grease. Solid lubricants such as molybdenum disulfide and graphite are added to grease in certain applications for high temperatures (above 315 ºC or 599º F) and extreme high pressure applications. Incorporating solid additives requires frequent grease changes to prevent accumulation of solids in components.

2.8 Grease Type

2.8.1 Calcium grease.

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. 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 ºC (175º 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 ºC (200 ºF). 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 hydrous (waterless) grease. Since dehydration is not a concern, anhydrous calcium grease can be used continuously to a maximum temperature of around 110 ºC (230 ºF).

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 ºC (500 ºF) can be obtained and the maximum usable temperature increases to approximately 177º C (350 ºF). With the exception of poor pump ability 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.

2.8.2 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 ºC (250 ºF), 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 flexibility. It cannot compete with water resistant, more heat resistant multipurpose greases. However, it still recommended for certain heavy duty applications and well sealed electric motors.

2.8.3 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 ºF), 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.

Aluminum complex grease has a maximum usable temperature of almost 100 ºC (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.

2.9 Compatibility

Greases are considered incompatible when the physical or performance characteristics of the mixed grease fall below original specifications. In general, greases with different chemical compositions should not be mixed. Mixing greases of different thickeners can form a mix that is too firm to provide sufficient lubrication or more commonly, a mix that is too soft to stay in place.

Combining greases of different base oils can produce a fluid component that will not provide a continuous lubrication film. Additives can be diluted when greases with different additives are mixed. Mixed greases may become less resistant to heat or have lower shear stability. If a new brand of grease must be introduced, the component part should be disassembled and thoroughly cleaned to remove all of the old grease. If this is not practical, the new grease should be injected until all traces of the prior product are flushed out. Also, the first grease changes should be more frequent than normally scheduled.

2.10 Grease Application Guide

When selecting grease, it is important to determine the properties required for the particular application and match them to specific grease. A grease application guide is shown in Table 2.2. It shows the most common greases, their usual properties, and important uses. Some of the ratings given are subjective and can vary significantly from supplier to supplier. Common ASTM tests for the grease characteristics describe are shown in Table 2.3.

Table 2.2: Grease Application Guide

Grease Application Guide (Boehringer, 1992)								
Properties	Aluminum	Sodium	Calcium-Conventional	Calcium - Anhydrous	Lithium	Aluminum Complex	Calcium Complex	Lithium Complex
Dropping point (EC)	110	163-177	096-104	135-143	177-204	260+	260+	260+
Dropping point (EF)	230	325-350	205-220	275-290	350-400	500+	500+	500+
Maximum usable temperature (EC)	79	121	93	110	135	177	177	177
Maximum usable temperature (EF)	175	350	200	230	275	350	350	350
Water resistance	Good to excellent	Poor to fair	Good to excellent	Excellent	Good	Good to excellen	Fair to excellent	Good to excellent
Work stability	Poor	Fair	Fair to good	Good to excellent	Good to excellent	Good to excellent	Fair to good	Good to excellent
Oxidation stability	Excellent	Poor to good	Poor to excellent	Fair to excellent	Fair to excellent	Fair to excellent	Poor to good	Fair to excellent
Protection against rust	Good to excellent	Good to excellent	Poor to excellent	Poor to excellent	Poor to excellent	Good to excellent	Fair to excellent	Fair to excellent

Pumpability (in centralized system)	Poor	Poor to fair	Good to excellent	Fair to excellent	Fair to excellent	Fair to good	Poor to fair	Good to excellent
Oil separation	Good	Fair to good	Poor to good	Good	Good to excellent	Good to Excellent	Good to excellent	Good to excellent
Appearance	Smooth and clear	Smooth To fibrous	Smooth and buttery	Smooth and buttery	Smooth and buttery	Smooth and buttery	Smooth and buttery	Smooth and buttery

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter explains conceptual study, laboratory experimental work, analyzing and completion of the project. The detailed procedure of the experimental work is discussed throughout this chapter. In general, the experiment consists of two major section, laboratory work (production grease) and data analysis.

3.2 Overall Methodology

The experiments were carried out according to the standard procedure outline in section 3.3. The experiments were divided into two parts. The first part was to prepare the samples of grease by using used lubricant as base oil instead of petroleum oil. The second part of the experiment involved analyzing the product by determines the viscosity of the grease using viscometer. Figure 3.1 shows the outline of the overall methodology where Experiment Work 1 was producing grease from used lubricant, 3 different type of soap is used in this experiment. For the Experiment Work 2 where the product dropping point and viscosity was analyzed. Then the comprehensive discussion was carried out on the viscosity of the grease, to know and analyzed the quality of the grease product.

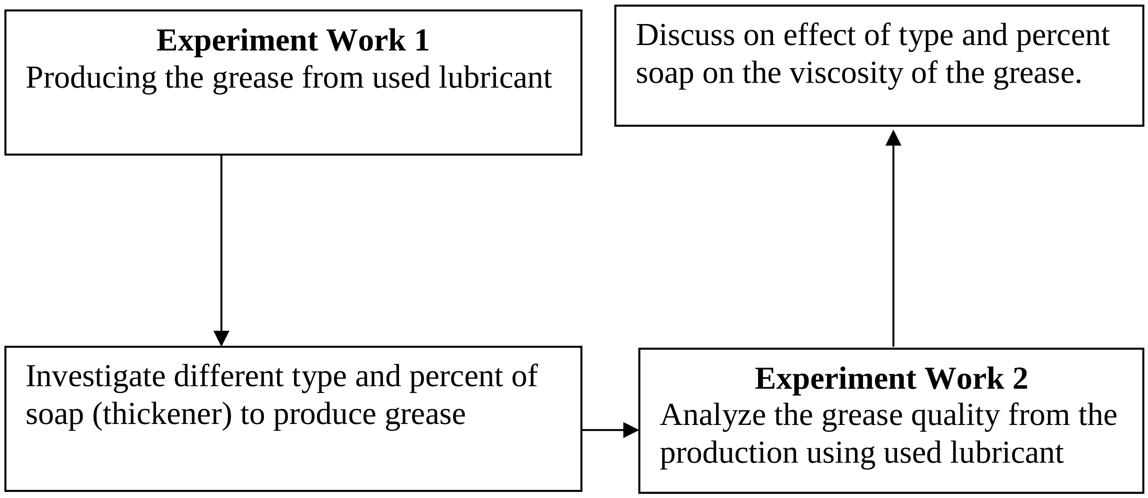

Figure 3.1: Outline of overall methodology

3.3 Experimental Methodology

3.3.1 Production of Grease

Used lubricant were collect from mechanical lab, used lubricant is a major component to produce grease. Used lubricant is base oil for production grease instead of petroleum oil. For production grease, soap is used as a thickener for the base oil. Three major type of thickener is used, calcium soap, sodium soap and aluminum soap. Metallic hydroxides were mixed with fatty acid (cooking oil) with ration 15% and 85% by mass at the room temperature. Four different soap ratios is used, which is 10%, 20%, 30% and 40% by volume mixed with base oil for each of soap. Base oil and soap (thickener) mixture were heated up to 200°C using the rotary evaporator bath. At the same time the mixture were stir to improve the mixing efficiency. The mixture was heated for 1½ hour then is cooling down to get the product.

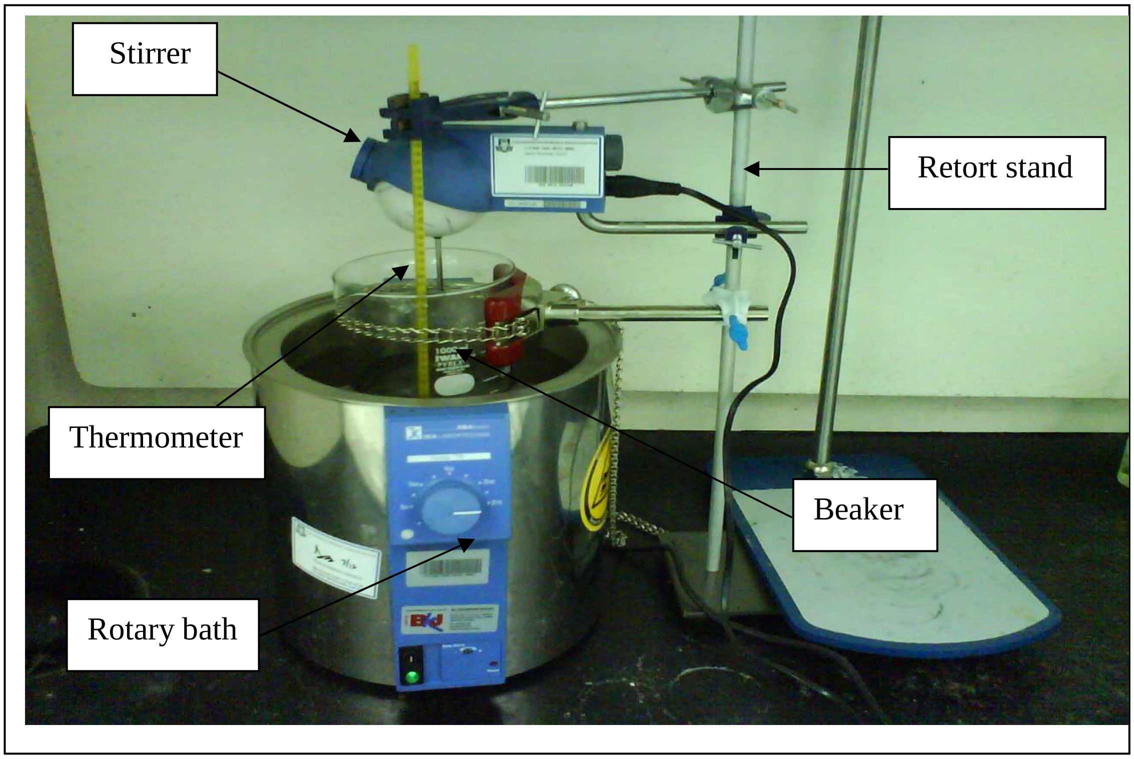

Figure 3.2: Laboratory Scale for Production Grease

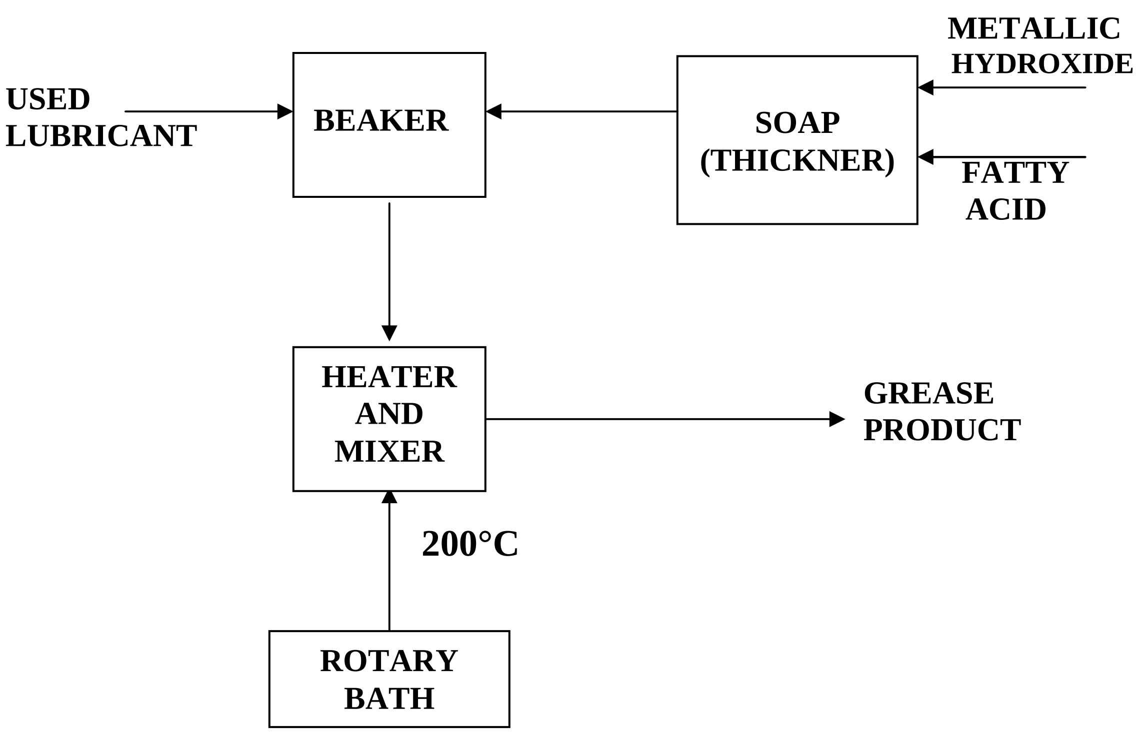

Figure 3.3: Block Diagram Production Grease Process Flow

3.3.2 Analysis Using Viscometer (Brookfield DV-III Ultra Programmable Rheometer)

The Brookfield DV-III ultra programmable rheometer measure fluids and semi fluids parameters of shear stress and viscosity at given shear rates. Viscosity is a measure of a fluids and semi fluid to flow. The principle of operating of the DV-III Ultra is to drive a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer. The viscosity measurement range of the DV-III Ultra (in centipoises or CP) is determined by the rotational speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and the full scale torque of the calibrated spring.

Viscosity is the measure of the internal friction of a fluid. This friction becomes apparent when a layer of fluids is made to move in relation to another layer. The greater the friction, the greater the amount of force required to cause this movement, which is called ‘shear’. Shearing occurs whenever the fluid is physically moved or distributed, as in pouring, spreading, spraying, mixing. Highly viscous fluids, therefore, required more force to move than less viscous material.

3.3.2.1 Rheometer

Rheometer is equipment use to measure viscosity. It is advancement in viscosity measurement which can be connected with PC and available with software. This instrument, with variable speed capability, allows easy handling and programming of complication measurement.

Figure 3.4: Brookfield DV-III Systems

3.3.2.2 Spindle

Rheometer works with spindle suitable foe most applications within the viscosity range of the instrument. Different types of spindle are required for different type of samples to obtain optimum result. Examples of spindles are:

- Disk spindle
- Cylindrical spindles
- Cone/plate spindles
- T-bar spindles

Figure 3.5: Cylindrical Spindle-LV 2

3.3.2.3 Analyze using LV spindle

For analyze using LV spindle, firstly the water jacked and straight bar are take off. Then 60ml sample are pour into the 100ml beaker (avoid the bubble). Then the powers of viscometer (at the back of black color base) are switch on. Button 2 is press to operate in standalone mode. After a short pause the display will prompt “REMOVE SPINDLE, LEVEL RHEOMETER AND PRESS THE MOTOR ON/OFF KEY TO AUTOZERO”. The autozero is started by pressing the MOTO ON/OFF key. The screen flashes approximately 15 second and display “AUTOZERO IS COMPLETE REPLACE SPINDLE AND PRESS ANY KEY”. The spindle LV2 is attach to the coupling and immerse the spindle into the sample by slowly bring down the rheometer to level of cylinder attach to spindle is just immerse to the sample.

The spindle must be center in the beaker. The spindle is change by pressing the SELECT SPDL. The data of instrument Torque (%), Viscosity (cP) is recorded. The parameter is toggled from one to another using the select display key. After finish the analysis, motor and power is switch off.

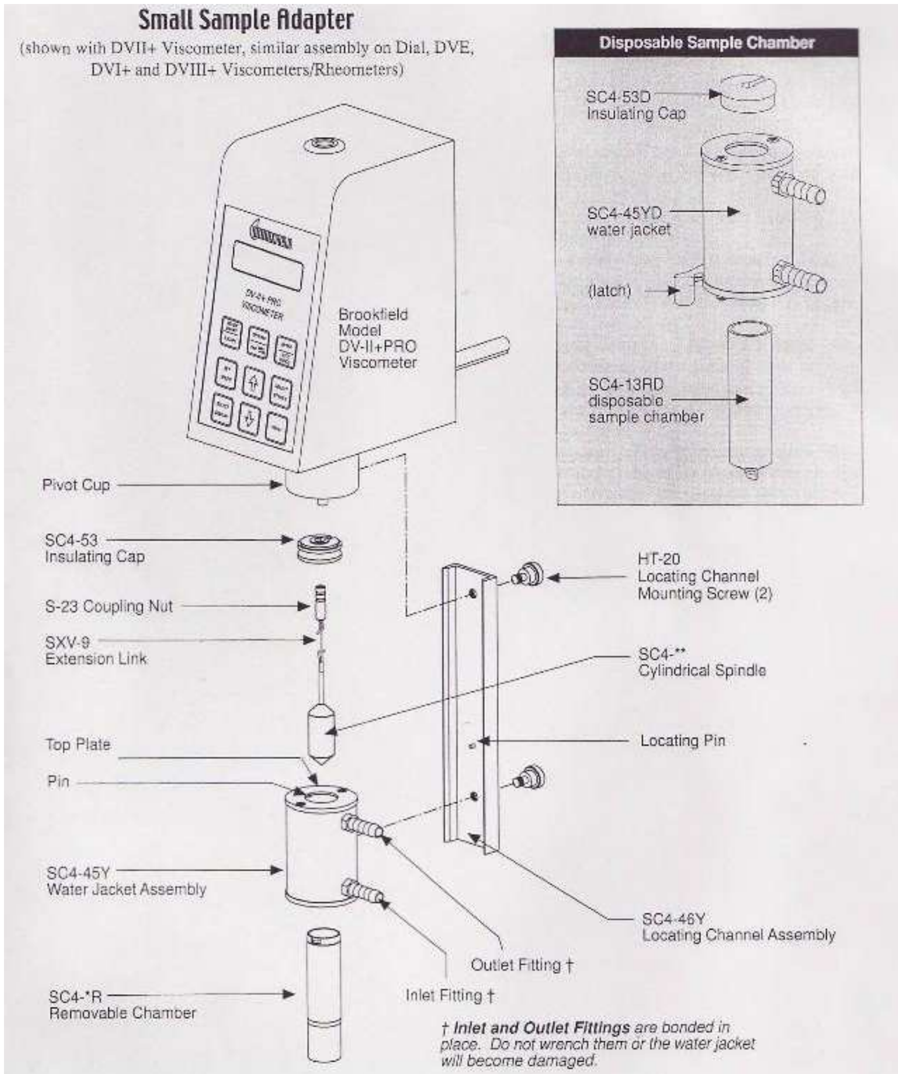

Figure 3.6: LV spindle

3.3.2.4 Analyze using Helipath spindle

For analyze using Helipath spindle, powers of viscometer (at the back of black color base) are switch on. Button 2 is press to operate in standalone mode. After a short pause the display will prompt “REMOVE SPINDLE, LEVEL RHEOMETER AND PRESS THE MOTOR ON/OFF KEY TO AUTOZERO”. Then the autozero is started by pressing the MOTOR ON/OFF key, the screen flashes approximately 15 second and display “AUTOZERO IS COMPLETE REPLACE SPINDLE AND PRESS ANY KEY. The weight is attach to the closer assembly and the T-bar (T-A) spindle is grip. High of rheometer is adjusted until the cross is completely immersed in the sample.

The sample is ensuring center in the beaker, and the spindle number is change by pressing the SELECT SPDL. Spindle T-A must be key in by using code number 91. The instrument data Torque (%), Viscosity (cP) is recorded and the parameters are toggled from one to another using the selected display key. After finishing the analysis, motor and power is switch off. The rheometer is push up and the closer assembly is takes off.

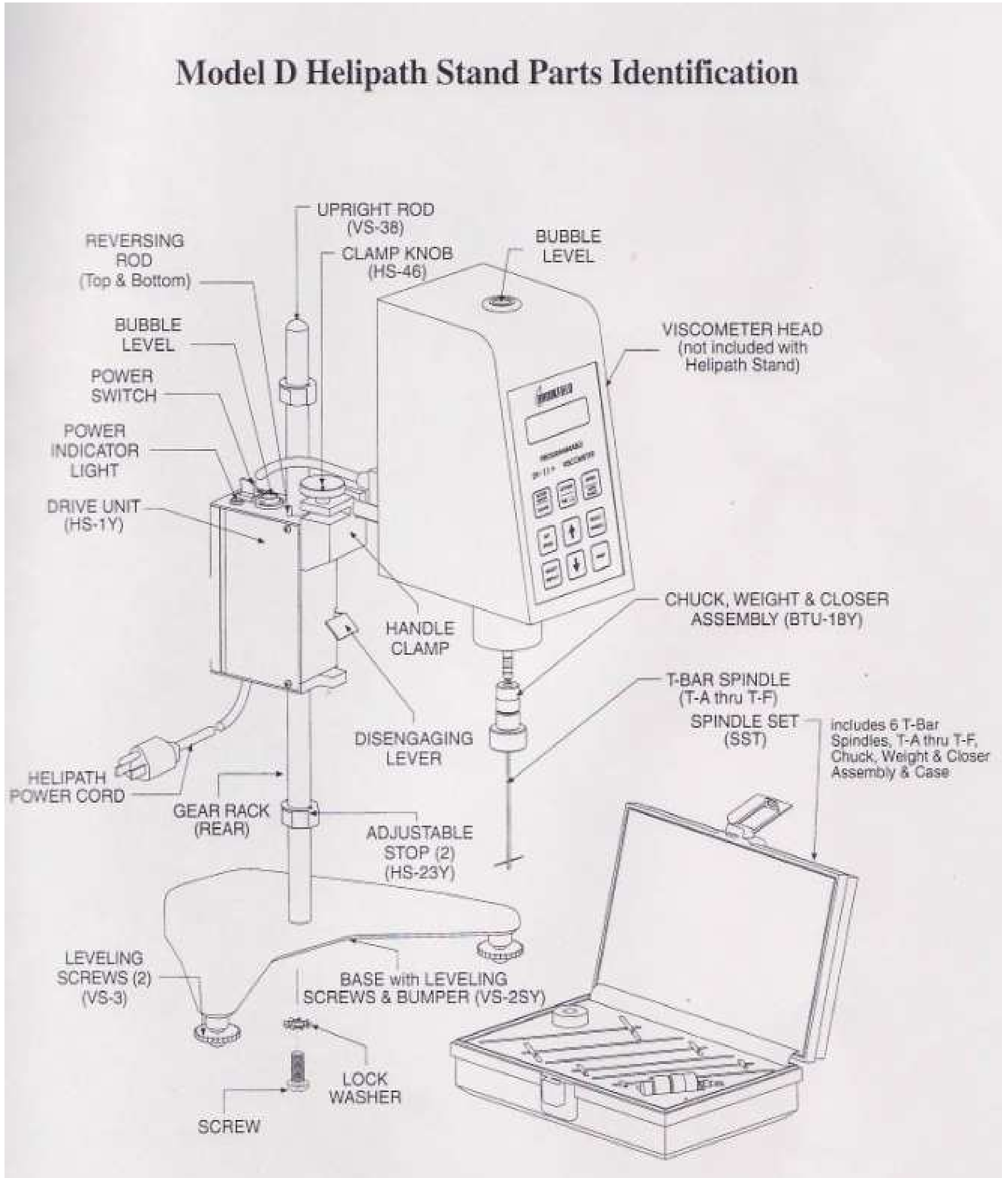

Figure 3.7: Helipath spindle

3.3.2.5 Calculation of Viscosity

Using the viscometer we can get the value of viscosity in digital reading. To calculate viscosity in manual, the dial reading or % torque are multiple with the factor corresponding to the viscometer spindle and speed used. Different types of spindle have different Spindle Factor value, for this experiment spindle SC4-34 has been used for Small Sample Adapter and T-A spindle for Helipath Stand spindle.

Table 3.1: Spindle Factor for Small Sample Adapter

LV Viscometer					
Speed RPM	Spindle Number				
	18	**31**	**34**	**16**	**25**
60	0.5	5	10	20	80
30	1	10	20	40	160
12	2.5	25	50	100	400
6	5	50	100	200	800
3	10	100	200	400	1.6K
1.5	20	200	400	800	3.2K
0.6	50	500	1K	2K	8K
0.3	100	1K	2K	4K	16K

Table 3.2: Spindle Factor for Helipath Stand spindle

Speed (RPM)	LVF & LVT Vascometers					
	Spindle Number					
	T-A	**T-B**	**T-C**	**T-D**	**T-E**	**T-F**
12	15.6	31.2	78	156	390	780
6	31.2	62.4	156	312	780	1.56K
3	62.4	124.8	312	624	1.56K	3.12K
1.5	124.8	249.6	624	1.248K	3.12K	6.24K
0.6	312	624	1.56K	3.12K	7.8K	15.6K
0.3	624	1.248K	3.12K	6.24K	15.6K	31.2K

Viscometer = Torque × Spindle Factor

(Unit in centipoises, cP)

CHAPTER 4

RESULT AND DISCUSSION

4.1 Sample Preparation And Analysis

In this chapter, analysis of the grease was performed. Experimental parameters were evaluated. The effect of spindle speed (RPM), percentage of soap (thickener) on the viscosity and sample preparation was studied. The value of viscosity and the sample product are show in this chapter.

4.1.1 Sample Preparation

The samples of grease are prepared by using method in chapter 3. The percentage of soap (thickener) was the major parameter in preparing the sample. Three type of soap are used in this experiment, aluminum soap, calcium soap, and sodium soap. Different percentages of soap (thickener) are prepared for each type of soap, 10%, 20%, 30% and 40%. Overall there is 12 samples are prepared for this experiment. The different type of soap is to determine the best type of soap to produce grease. Two method of analyzing are used in this experiment, which is to analyze the effect of spindle speed and effect of percentage soap.

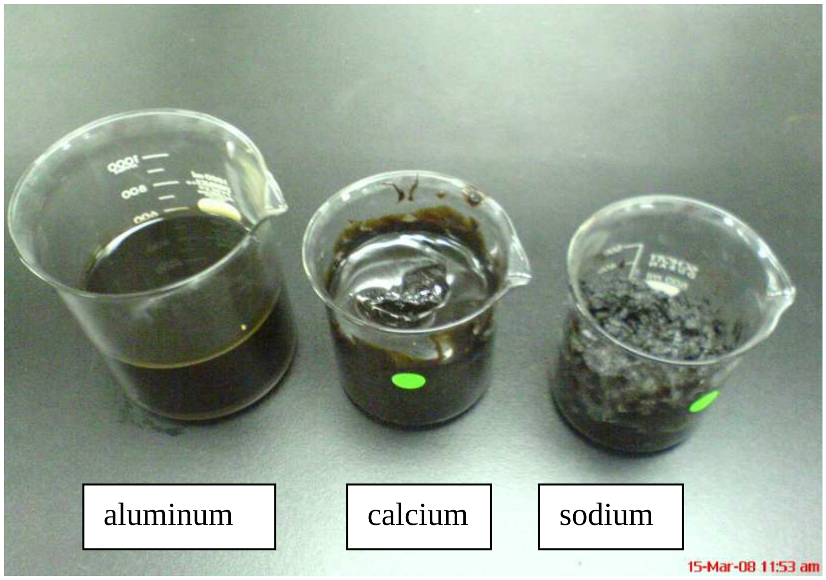

Figure 4.1: Product Sample for 20% Soap (thickener) and type of soap used

Table 4.1 show the overall physical characteristic of product sample according to the type of soap (thickener) and percent of soap (thickener) used. The phase of product are change depends on the percent soap (thickener) used. For aluminum soap the phase change from fluid to semi fluid, 10% until 30% soap the phase are in fluid phase. For 40% soap the phases are in semi fluid (soft). Calcium soap at 10% is fluid, 20% to 30% semi fluid (soft), and 40% semi fluid (hard). For sodium soap at 10% is semi fluid (soft), 20% to 30% is semi fluid (hard) and 40% semi fluid (very hard).

Table 4.1: Physical Characteristic of Product Grease

Physical characteristic product grease			
Type of soap / % of soap	aluminum	calcium	sodium
10%	fluid	fluid	semi fluid (soft)
20%	fluid	semi fluid (soft)	semi fluid (hard)
30%	fluid	semi fluid (soft)	semi fluid (hard)
40%	semi fluid (very soft)	semi fluid (hard)	semi fluid (very hard)

4.1.2 Sample Analysis

The sample analysis are divided into two parts, the first part is to analyze the effect of spindle speed in RPM (rate per minutes) to the viscosity of grease. The second part is to analyze the effect of percentage soap (thickener) to the viscosity of grease.

4.1.2.1 Effect of spindle speed RPM to viscosity

For analysis effect of spindle speed (RPM) to the viscosity, the percentage of soap is constant. The value of percent soap is 20% for each type of soap.

a) aluminum soap

For aluminum soap the spindle used is LV 2, the sample for 20% aluminum soap grease are in the liquid form. Table 4.2 shows the viscosity is decreasing with the increase of spindle speed. The data in table 4.2 was transform into graft in figure 4.2.
show clearly the decreasing of viscosity. All the result of viscosity from computer is show in appendix A.1 to A.5.

Table 4.2: Viscosity of Grease in Different RPM

Spindle Speed (RPM)	5	10	15	20	25
Viscosity (cP)	459.9	444.91	459.1	411.51	387.52

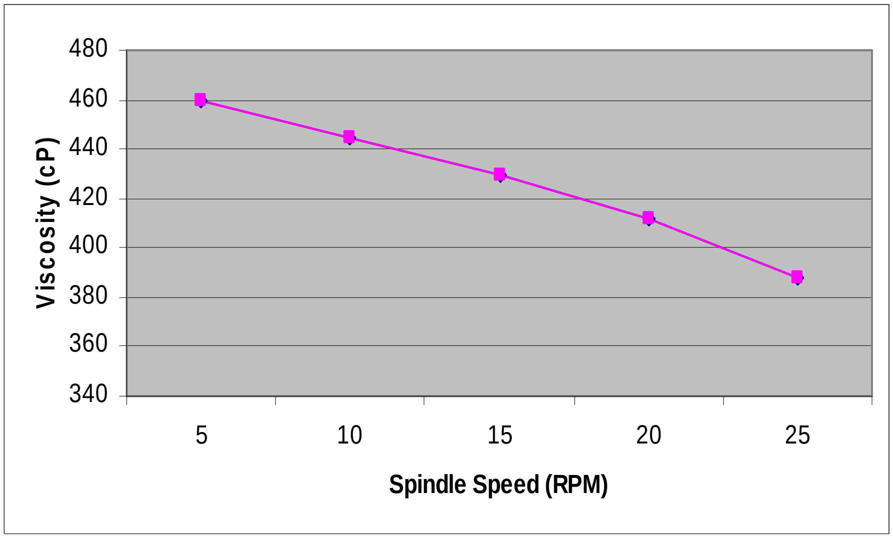

Figure 4.2: Spindle Speed (RPM) versus Viscosity (cP)

b) Calcium soap

For calcium soap spindle use is LV 2 spindle, the sample for 20% calcium soap grease are in semi liquid form. Table 4.3 shows the viscosity is decreasing with the increase of spindle speed. The data in table 4.3 was transform into graft in figure 4.3 show clearly the decreasing of viscosity. All the result of viscosity from computer is show in appendix A.6 to A.9.

Table 4.3: Viscosity of Grease in Different RPM

Spindle Speed (RPM)	5	10	15	20	25
Viscosity (cP)	26394.36	13197.18	8798.12	6598.59	5278.87

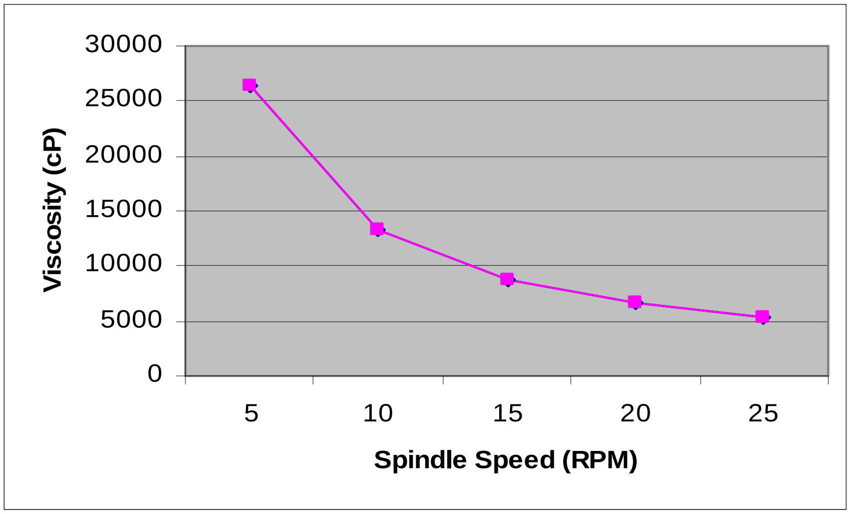

Figure 4.3: Spindle Speed (RPM) versus Viscosity (cP)

c) Sodium soap

For sodium soap spindle use is LV 2 spindle, the sample for 20% sodium soap grease are in semi liquid form. Table 4.4 shows the viscosity is decreasing with the increase of spindle speed. The data in table 4.4 was transform into graft in figure 4.4 show clearly the decreasing of viscosity. All the result of viscosity from computer is show in appendix A.10 to A.14.

Table 4.4: Viscosity of Grease in Different RPM

Spindle Speed (RPM)	2	4	6	8	10
Viscosity (cP)	65985.92	32992.96	21995.31	16496.48	13197.18

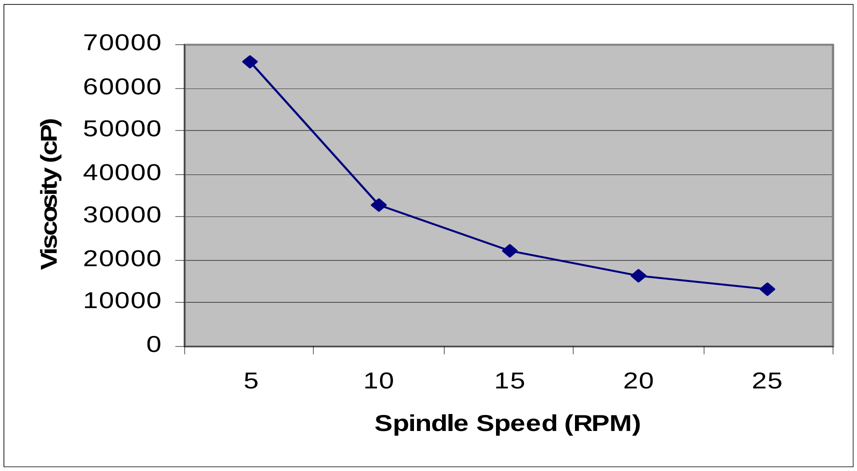

Figure 4.4: Spindle Speed (RPM) versus Viscosity (cP)

Viscosity of grease can be effect by certain parameters. Spindle speed (RPM) was found to be one of the important factors, where the higher spindle speed increased the lower viscosity of the grease reading. Figure 4.2, Figure 4.3 and Figure 4.4 show the viscosity pattern of spindle speed. The viscosity was decreased with the increase of spindle speed. The percentages of soap are found to be constant for all three sample analysis. Second parameters effect to the viscosity is type of soap, the lowers viscosity values are from aluminum soap, the range value is between 387.52 to 459.9cP. The second lowers are from calcium soap, between 5,278.87 to 26,394.36Cp. Viscosity of grease from sodium soap was found to be the highest value between three types of soap. The value is 13,197.18 to 65,985.92Cp.

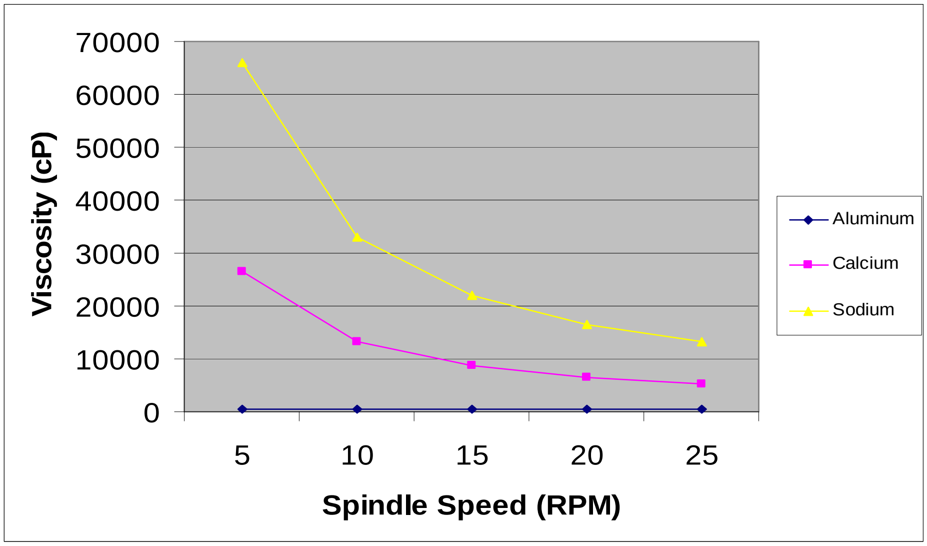

Figure 4.5: Comparisons on Viscosity of Grease in Different Type of Soap

Generally, high spindle speed gives impact to the viscosity of grease. Spindle speed represents the force give to the grease, and the greater amount of force gives the greater friction of the liquid. The greater friction of liquid gives the lowest viscosity of the grease. Different type of soap gives different value of viscosity. Sodium soap was found to be the strongest thickener. It shows at the value of viscosity with sodium soap. The strong thickener gives the high value of viscosity. The second and third strongest thickener is calcium and aluminum soap.

4.1.2.2 Effect of Percentage Soap to Viscosity

For analysis effect of percentage soap (thickener), three samples are tested for aluminum soap sample. The percent of soap test is 10%, 20% and 30%. Three samples are chosen because of the same phase, so that we can see effect of percent soap to the viscosity by constant the spindle speed (RPM). The sample is tested at the same phase because different phase give a big different of viscosity and the effect of percentage soap cannot be analysis clearly.

Second major parameters that affect the viscosity are the percentage of soap (thickener). High amount of soap (thickener) will give the high viscosity of the grease. Figure 4.6 shows the increasing of viscosity with the percent of soap (thickener). The highest viscosity is at 30% soap and the lowest viscosity at 10% soap. From the analysis we know that high amount of soap are needed for the high viscosity of grease. In industry and machinery equipment, they need high viscosity of grease to ensure the grease not squeeze out during the machinery work in high pressure and temperature.

Table 4.5: Viscosity of Grease in Percentage Aluminum Soap (Thickener)

Percentage of Soap (Thickener)	Viscosity (cP)
10	340.43
20	411.51
30	523.11

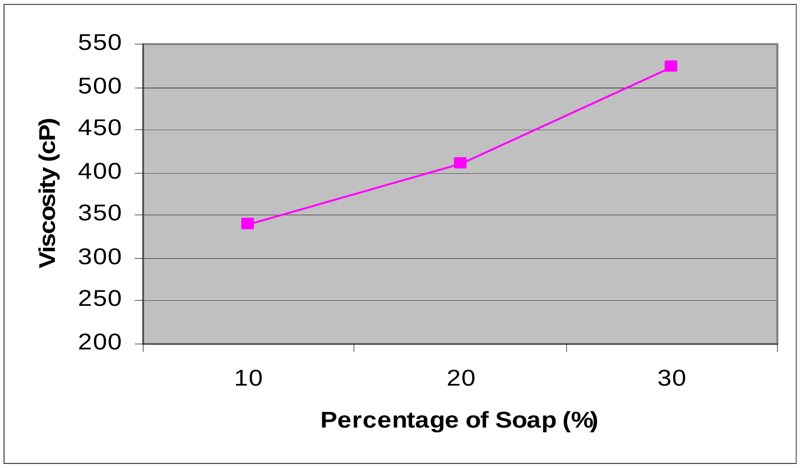

Figure 4.6: Percentage of Aluminum Soap versus Viscosity (cP)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the results that have been produced in this research, it was found that grease was successfully produced by mixing used lubricant, a thickener, and additives. Soap thickeners are formed by reacting a metallic hydroxide or alkali, with a fat, fatty acid, or ester. The type of soap used depends on the grease properties desired.

By referring Figure 3.1 we can conclude, grease can be produce from used lubricant using the method in flow chart 1. Different parameter had been used to produce grease, which is the type of soap (aluminum, calcium, sodium) and the percent of soap (thickener) had been used. The viscometer test and sample preparation on grease was visualized in this research, from the analysis result we found that the viscosity of grease was decrease with the increasing of spindle speed for all three type of soap used and the viscosity was increase with the increasing of percentage soap (thickener) added.

Analysis also showed that sodium soap was the strongest thickener for the grease production. The value of viscosity by using sodium soap is the highest compare to calcium and aluminum soap. Calcium soap is the second strongest and aluminum soap are the weakest thickener. The high percentage soap also needed for grease, so that it can hold in the high pressure and temperature. In this analysis the highest percentage of thickener have the highest value of viscosity.

5.2 Recommendation

In this research show that grease was successfully produced by mixing used lubricant, a thickener, and additives. Soap thickeners are formed by reacting a metallic hydroxide or alkali, with a fat, fatty acid, or ester. Since in industrial production, they usually use petroleum oil as base oil for production grease. This research proposes replace the petroleum oil with used lubricant, it will decrease the production cost for the grease and also give good effect to the environments.

The best way to produce grease is with sodium soap, where sodium soap is the strongest thickener compare to another thickener. Using this type of soap, only small amount of this thickener needed compare to another type of soap. This will decrease the production cost of the grease, grease with sodium soap also can hold in the high pressure and temperature condition for example in the factory and machinery work.

The quality of grease using used lubricant as base oil cannot same as quality using petroleum oil, because of contaminant and impurity of component in the used lubricant, filter the used lubricant before use it in production grease is one of the recommendation for this research, by eliminate the contaminant and impurity component. The second recommendation is by added the used lubricant with the petroleum oil to improve the quality of the production.

For the method of production grease, the time of heating and mixing can be increase more then 1 ½ hour to increase the mixing efficiency of the grease. By increase the mixing efficiency, the quality of grease can be improve by ensure the component of grease are mix well.

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APPENDIX A

RHEOMETER RESULT AND VESCOSITY OF THE GREASE

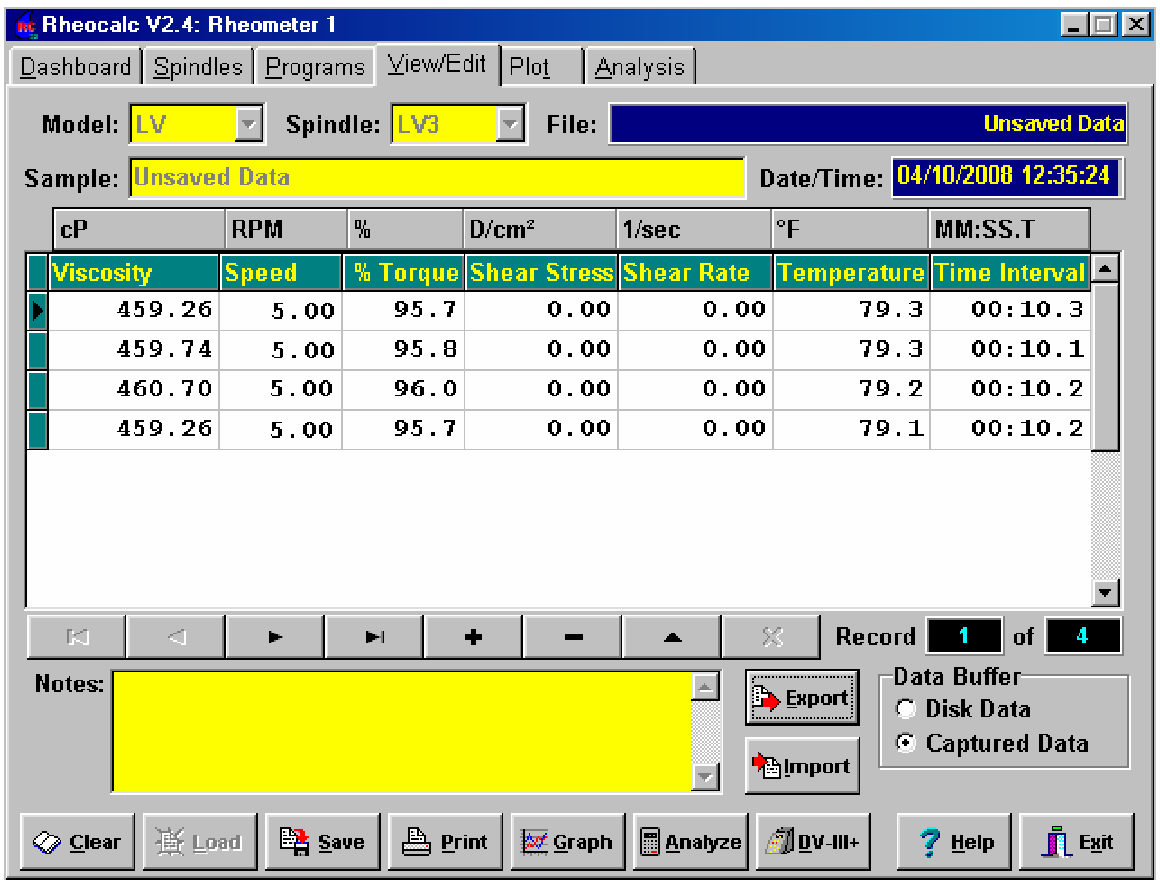

Figure A.1: Analysis of Grease Viscosity for Aluminum Grease at RPM 5

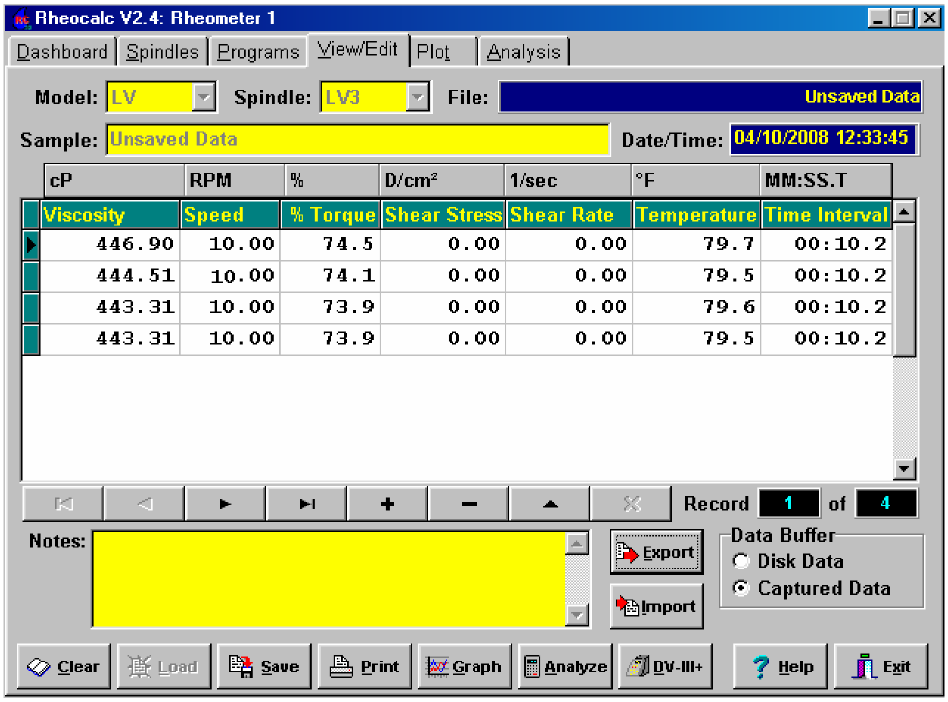

Figure A.2: Analysis of Grease Viscosity for Aluminum Grease at RPM 10

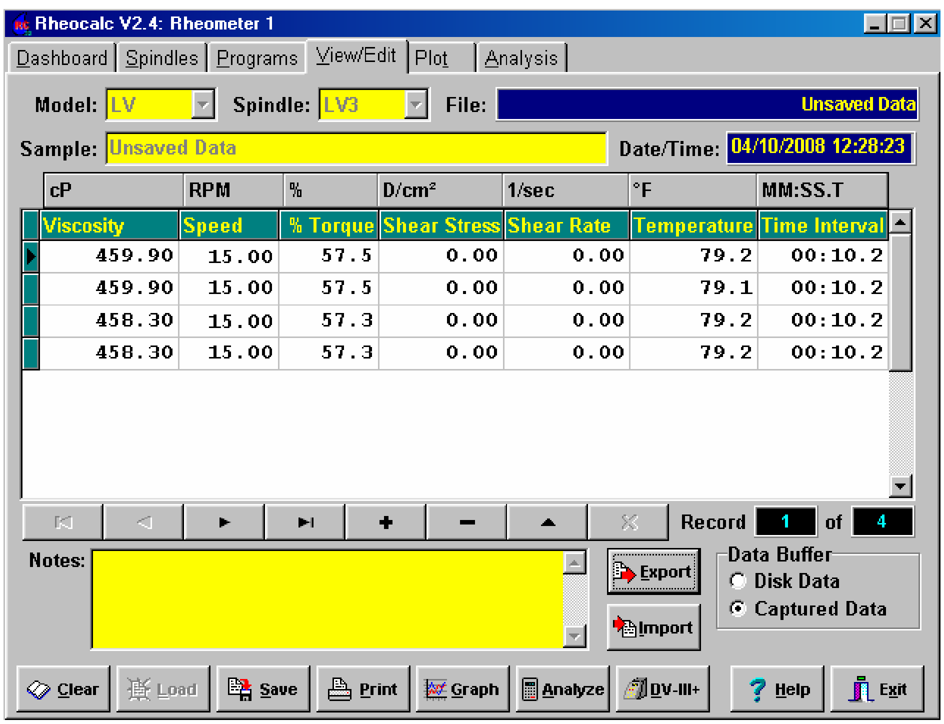

Figure A.3: Analysis of Grease Viscosity for Aluminum Grease at RPM 15

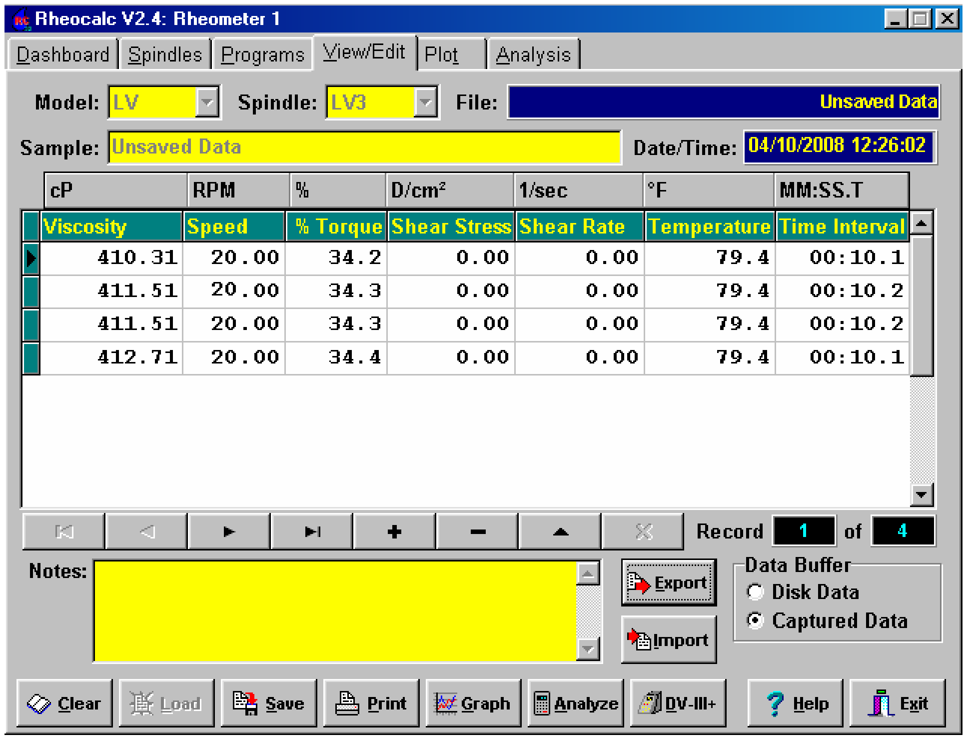

Figure A.4: Analysis of Grease Viscosity for Aluminum Grease at RPM 20

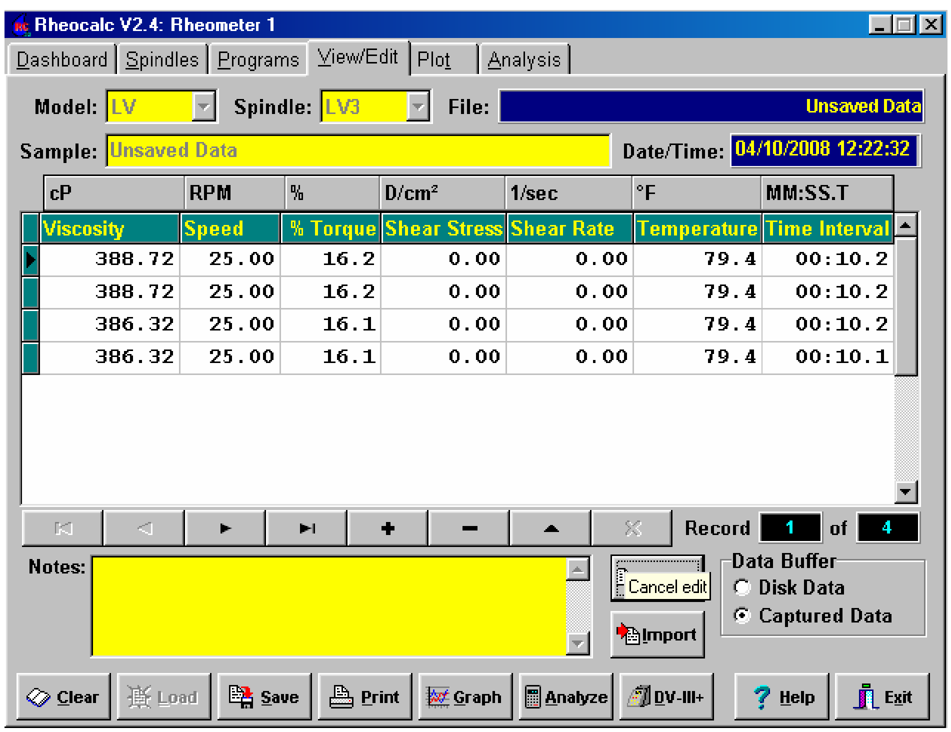

Figure A.5: Analysis of Grease Viscosity for Aluminum Grease at RPM 25

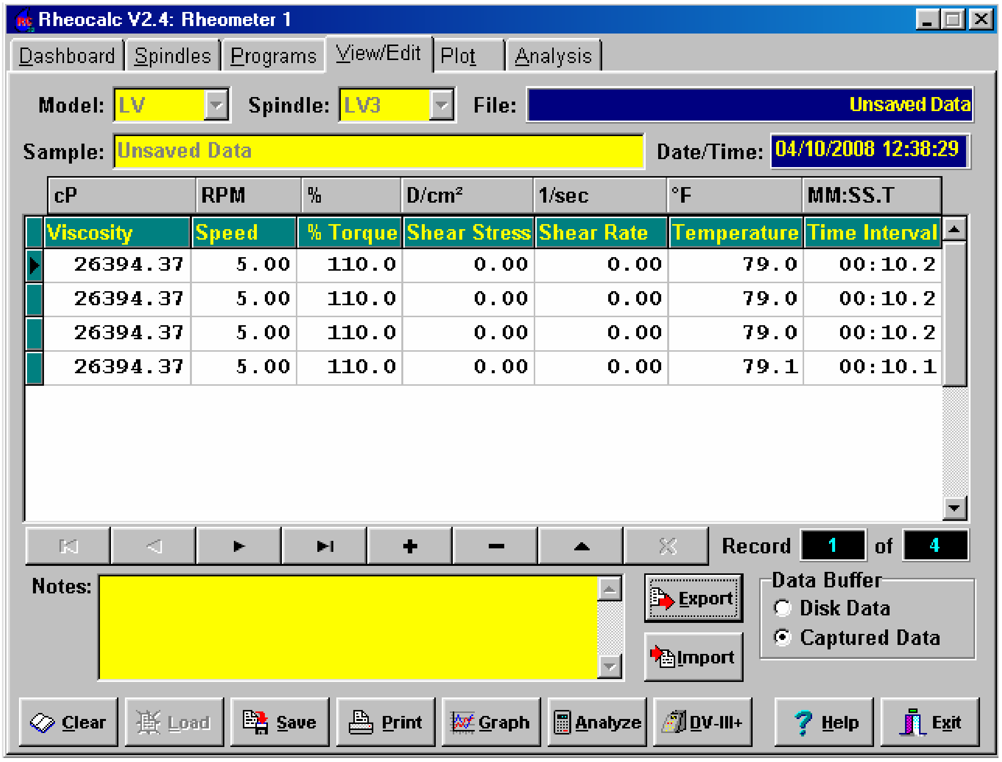

Figure A.6: Analysis of Grease Viscosity for Calcium Grease at RPM 5

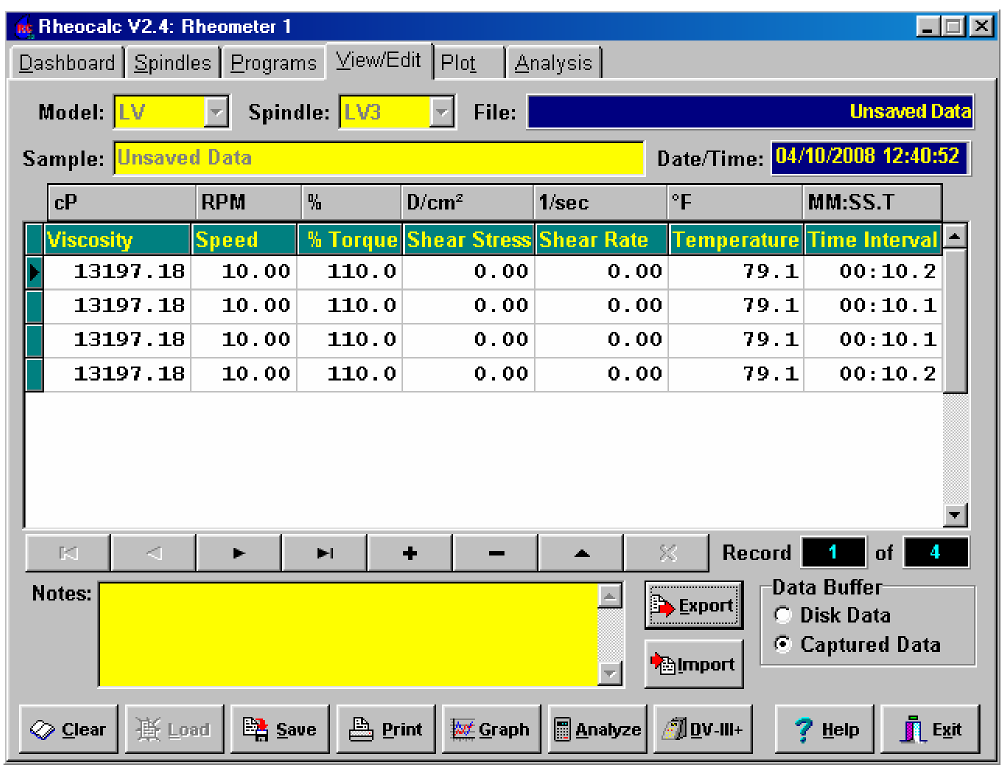

Figure A.7: Analysis of Grease Viscosity for Calcium Grease at RPM 10

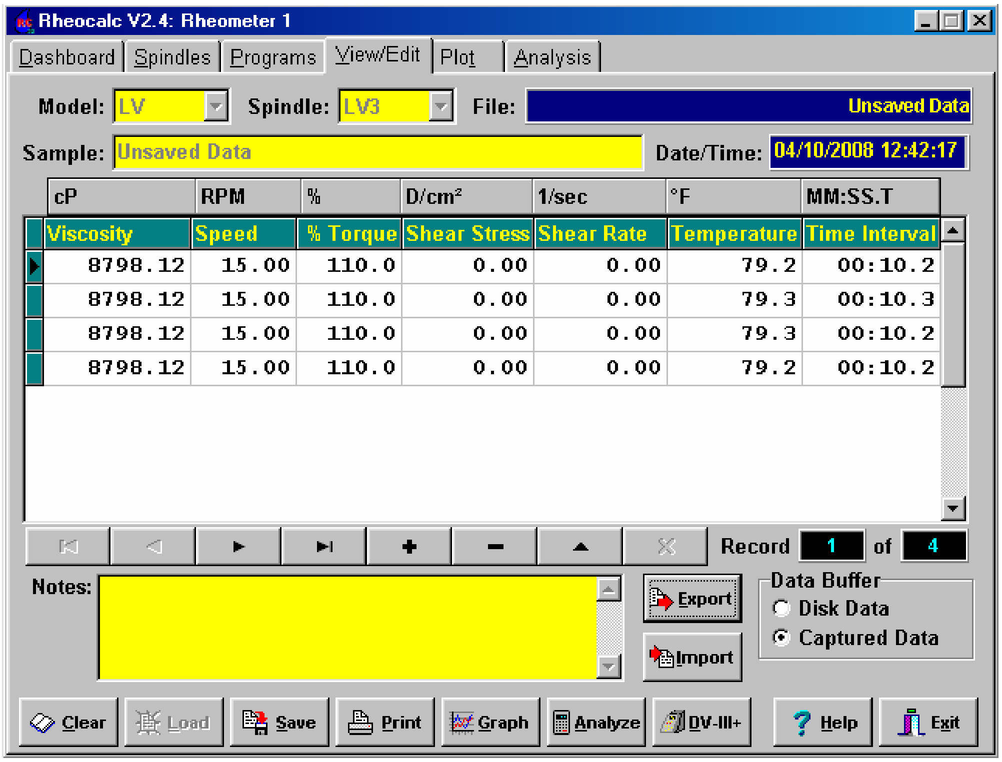

Figure A.8: Analysis of Grease Viscosity for Calcium Grease at RPM 15

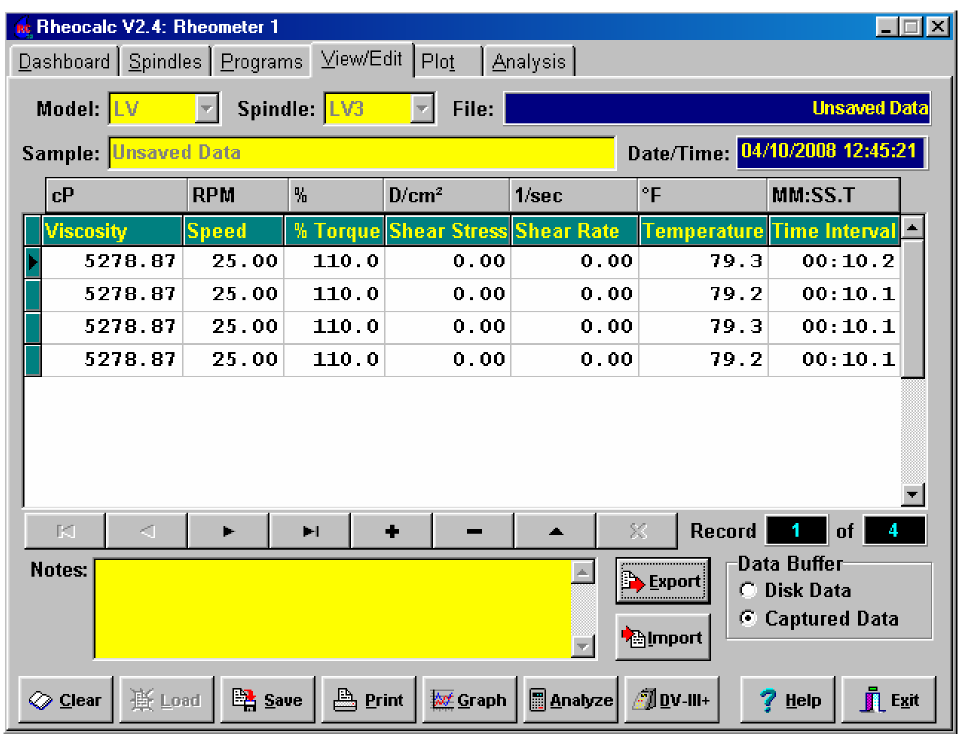

Figure A.9: Analysis of Grease Viscosity for Calcium Grease at RPM 25

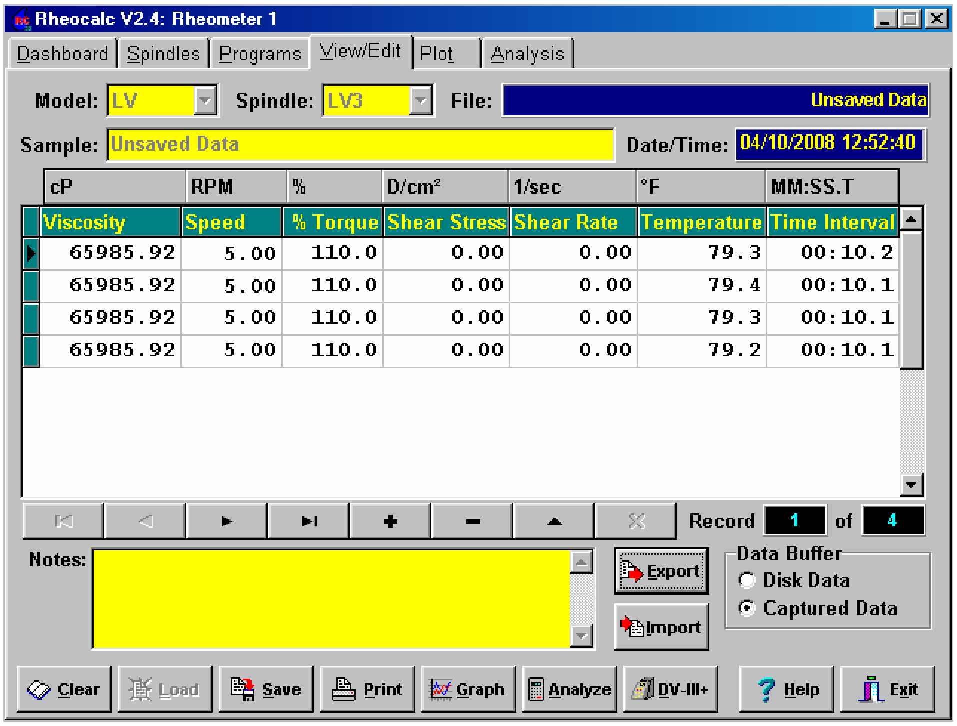

Figure A.10: Analysis of Grease Viscosity for Sodium Grease at RPM 5

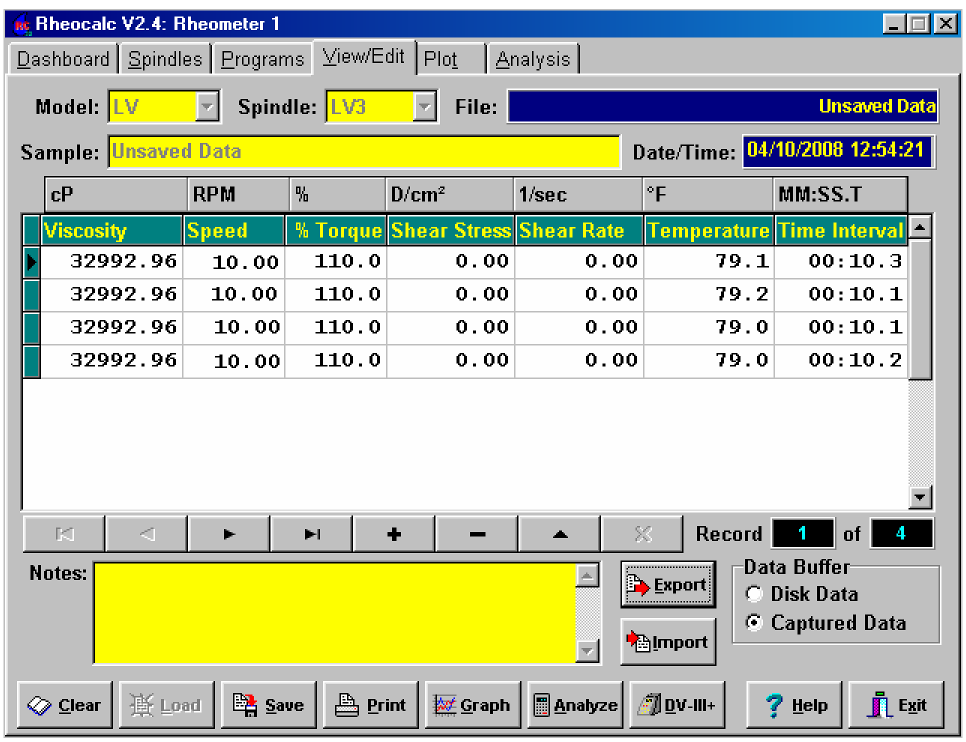

Figure A.11: Analysis of Grease Viscosity for Sodium Grease at RPM 10

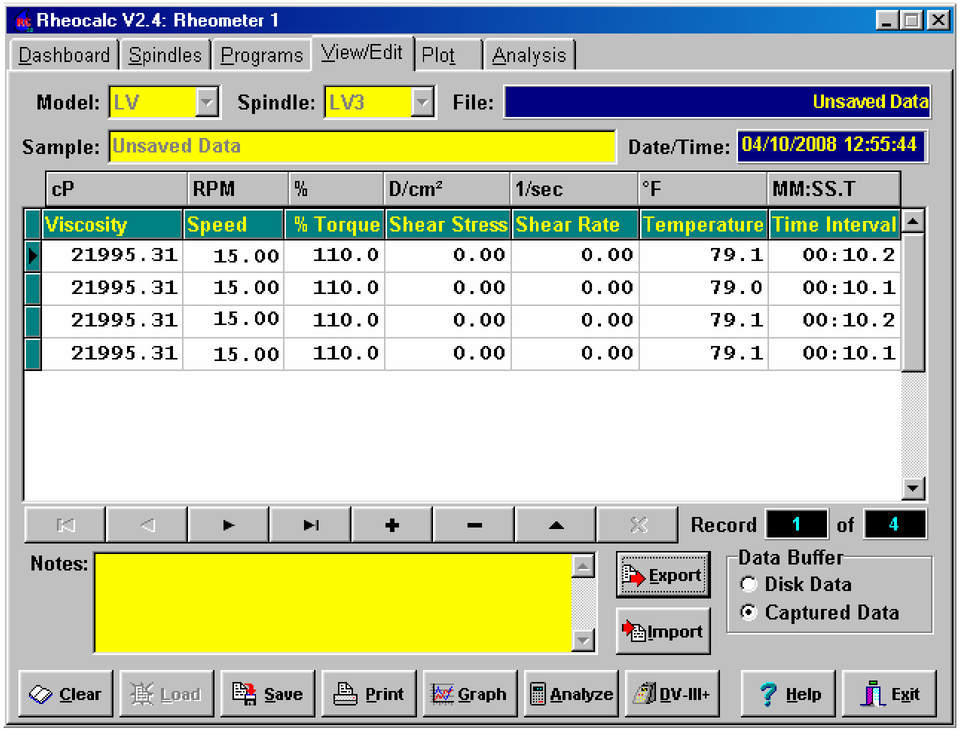

Figure A.12: Analysis of Grease Viscosity for Sodium Grease at RPM 15

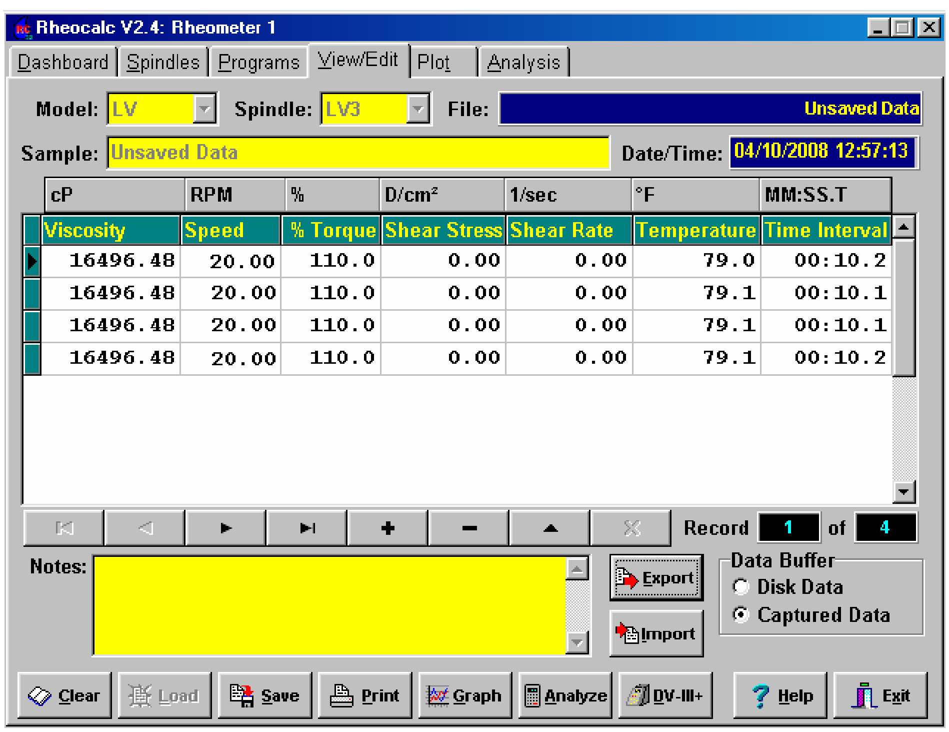

Figure A.13: Analysis of Grease Viscosity for Sodium Grease at RPM 20

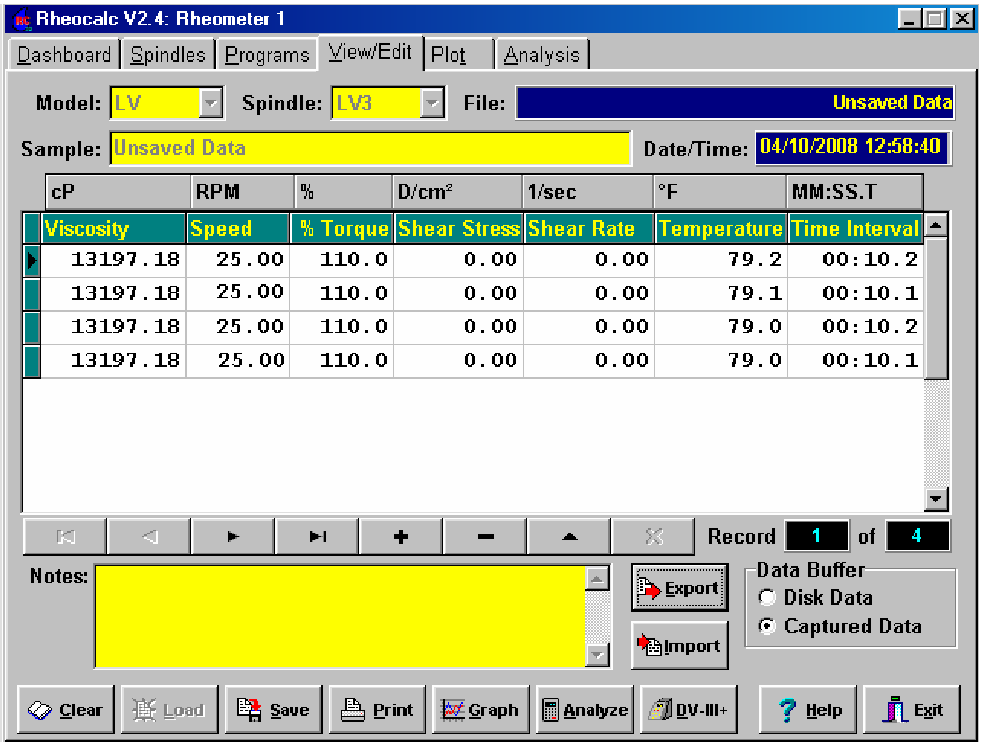

Figure A.13: Analysis of Grease Viscosity for Sodium Grease at RPM 25

APPENDIX B

Table B.1: ASTM Tests for Grease Characteristics

ASTM Tests for Grease Characteristics		
Grease Characteristic	**ASTM Test Method**	**Description**
Apparent viscosity / pumpability	D 1092 - Measuring Apparent Viscosity of Lubricating Greases	Apparent viscosities at 16 shear rates are determined by measuring the hydraulic pressure on a floating piston which forces grease through a capillary tube. Eight different capillary tubes and a 2-speed hydraulic gear pump are used.
Consistency and shear stability	D 217 - Cone Penetration of Lubricating Grease D 1403 - Cone Penetration of Lubricating Grease Using One-Quarter and One-Half Scale Cone	Depth, in tenths of a millimeter, a 150g (0.33-lb) cone penetrates the surface of worked and un worked grease at 25 EC (77 ºF) in 5 seconds. D 1403 is used when only a small amount of grease is

	Equipment D 1831- Roll Stability of Lubricating Grease	available. A 5- kg (11-lb) roller and 50 g (0.11 lb) of grease are put into a 165-rpm revolving chamber for 2 hours at room temperature. The difference in penetrations measured before and after rolling is an indicator of shear stability.
Corrosion and rust resistance	D 1743 - Determining Corrosion Preventive Properties of Lubricating Greases	A grease-packed bearing is spun for 1-minute at 1750 rpm. Excess grease is thrown off and a thin layer remains on bearing surfaces. The bearing is exposed to water and stored for 48 hours at 52 ºC (125 ºF) and 100% humidity. It is then cleaned and examined for corrosion
	D 4048 - Detection of Copper Corrosion from Lubricating Grease	A copper strip is immersed in grease inside a covered jar and heated in an oven or liquid bath for a specified time. The strip is removed, washed, and compared and

Heat resistance/Consistency	D 3232 - Measurement of Consistency of	classified using the ASTM Copper Strip Corrosion Standards.
Dropping point	D 566 - Dropping Point of Lubricating Grease D 2265 - Dropping Point of Lubricating Grease over Wide-Temperature Range	Grease and a thermometer are placed in a cup inside a test tube and heated until a drop falls through the cup. That temperature is the dropping point. The test tube assembly is heated in an oil bath for D 566 and inside an aluminum block oven for D 2265.
Evaporation	D 972 - Evaporation Loss of Lubricating Greases and Oils D 2595 - Evaporation Loss of Lubricating Greases over Wide-Temperature Range	Two liters per minute of heated air is passed over grease inside a chamber for 22 hours. Temperature range is 100 - 150 ºC (212 – 302) ºF) for D 972 and 93 - 315 ºC (200 - 599º F) for D 2595. Evaporation is calculated from grease weight loss, in percent. Can also indicate flow at

Oxidation Stability	Lubricating Greases at High Temperatures	high temperatures. Grease in a cylindrical opening in an aluminum block is heated at a rate of 5 ºC (10 ºF)/min while a trident probe turns at 20 rpm in the grease. A Brookfield viscometer attached to the probe measures torque at temperature increments. From this, apparent viscosities are determined at different temperatures.
Leakage	D 942 - Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method	A seal-less, grease-packed wheel bearing encircled by a collector ring is spun for 6 hours at 660 rpm at 105º C (221 ºF). Grease thrown off into the ring is weighed and leakage is determined.
	D 3336 - Performance Characteristics of Lubricating Greases in Ball-Bearings at Elevated Temperatures	Indicates oxidation from storage when grease charged with oxygen at 758 kPa (110 psi) is sealed in a “bomb” at 99 ºC (210º F). As grease oxidizes, it

		absorbs oxygen. Pressure is recorded at time intervals and degree of oxidation is determined by the corresponding drop in oxygen pressure. There are no ASTM tests for oxidation in service, but this test relates oxidation stability to failure rate of bearings at desired elevated temperatures.
Water Resistance	D 1264 - Determining the Water Washout Characteristics of Lubricating Greases D 4049 - Determining the Resistance of Lubricating Grease to Water Spray	Measures grease washout of a bearing turning at 600 rpm with water flowing at 5 mL/sec for 1 hour at 38 ºC (100 ºF) and 79 EC (175ºF). Measures removal of grease 0.8 mm (1/32 in) thick on a plate by water through a nozzle for hour at 38 ºC (100 ºF) and 79 ºC (175 ºF). 5 minutes at 38 ºC (100 ºF) and 275 kPa (40 psi)

APPENDIX C

PRODUCTION GREASE DEVICE

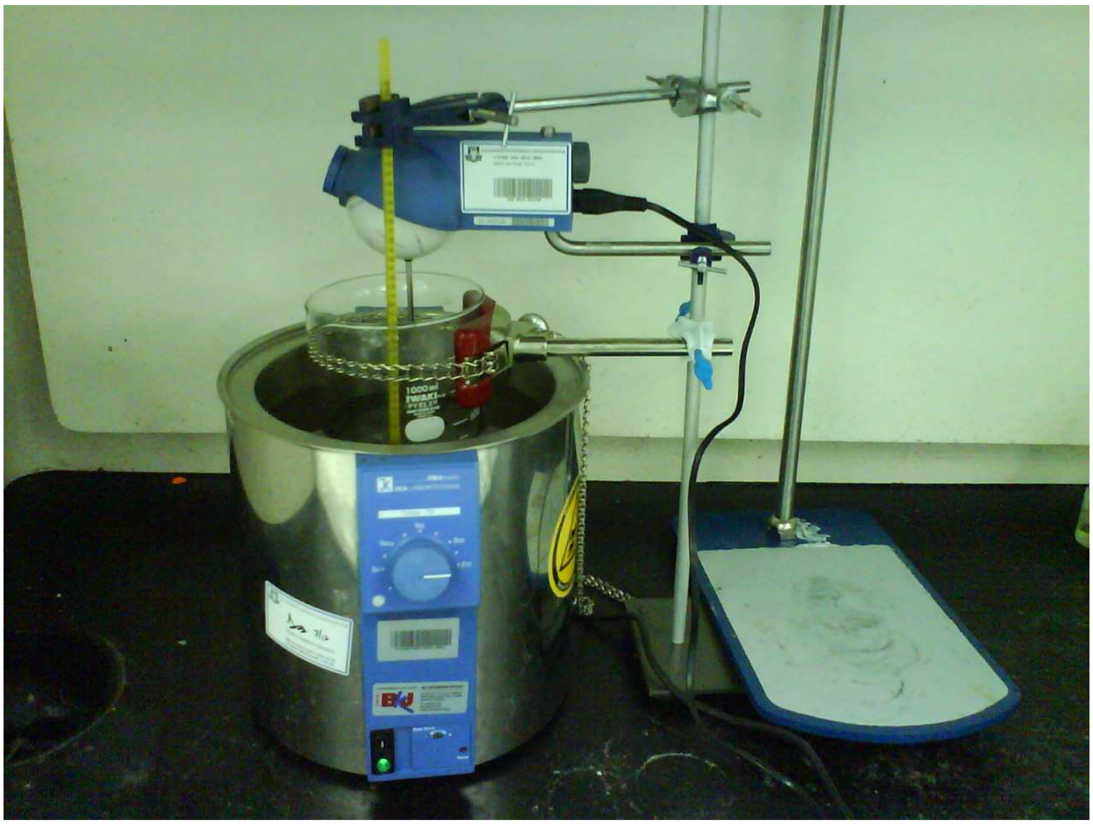

Figure C.1: Production Grease Equipment

VISCOMETER EXPERIMENT DEVICE

Figure C.2: Viscometer Unit

Figure C.3: Rheometer

Figure C.4: Cylindrical Spindle-LV 2