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PRODUCTION AND PERFORMANCE EVALUATION OF BIODEGRADABLE GREASE (BIOGREASE) FROM B*alanites aegyptiaca* (DESERT DATE) SEED OIL

BY

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JULY, 2016

TITLE PAGE

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By

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MSc/ENG/2896/2011-2012

A DISSERTATION SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES, AHMADU BELLO UNIVERSITY, ZARIA IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF A MASTER OF SCIENCE DEGREE IN MECHANICAL ENGINEERING

DEPARTMENT OF MECHANICAL ENGINEERING,
AHMADU BELLO UNIVERSITY,
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JULY, 2016

DECLARATION

I, **Abubakar Namadi Mohammed** declare that this dissertation titled **"Production and Performance Evaluation of Biodegradable Grease (Biogrease) From B*alanites aegyptiaca* (Desert Date) Seed Oil"** has been carried out by me in the Department of Mechanical Engineering. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this dissertation was previously presented for another degree or diploma at this or any other institution.

Abubakar Namadi MOHAMMED _____ _____

Name of Student Signature Date

CERTIFICATION

This Dissertation titled **"Production and Performance Evaluation of Biodegradable Grease (Biogrease) From B*alanites aegyptiaca* (Desert Date) Seed Oil"** By **Abubakar Namadi Mohammed** meets the requirements governing the award of the degree of Master of Science in Mechanical Engineering of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation..

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(Member Supervisory Committee) Signature Date

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(Head of Department) Signature Date

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(Dean School of Postgraduate Studies) Signature Date

DEDICATION

This work is dedicated to my entire family.

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All Thanks and Praise be to the Almighty Allah for seeing me through this phase of my life successfully. My sincere gratitude goes to my supervisors Professor C. O. Folayan and Dr F.O. Anafi whose tireless efforts and relentless prodding saw me through times of increased inertia.

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ABSTRACT

This work focused on the production and testing of biodegradable grease from *Balanites aegyptiaca* seed kernel oil. This was carried out by extracting the oil from the seeds using solvent n-Hexane extraction and reacting a given quantity of the extracted oil with a lithium soap to produce a lithium based biogrease. Consistency tests, drop point as well as gear wear tests were conducted on the developed biogrease samples and the results compared against that of a commercially available petroleum-based lithium grease - OANDO Crown grease EP2. The results showed that the oil combines with lithium stearate on a percentage weight basis of between 70% to 80%. Worked penetration values of the grease samples indicated a grease of consistency classification NLGI '0'. Dropping point values for the biogrease ranged from 65°C to 161°C with increasing dropping point value as percentage concentration of base oil increases, peaking at 161°C for 80% base oil. The cost of productionfor the bio grease was also calculated. The developed biogrease also showed good antiwear characteristics comparable to commercially available mineral grease.

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NOMENCLATURE

AF- Association Française does Normalisation

ASTM- American Society for Testing and Materials

DIN- Deutsches Institut fur Normung

DPA- Diphenylamine

EP- Extreme Pressure

ISO- International Standards Organisation

NLGI- National Lubricating Grease Institute

Wgi-Initial Weight of Gear(g)

Wgf- Final Weight of Gear (g)

γ- Kinematic Viscosity ($m^2/_s$)

μ- Dynamic Viscosity (Ns/m^2)

ρ- Density (kg/m^3)

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Olive oil was used as a lubricant as far back as 1650 BC. Various oils obtained from olive, rapeseed, castor beans, palm oil, and the fats from sperm whale, animal lard, and wool grease were used from around AD 50 until the early 19th century Sharma *et al*, (2006). The proliferation of machinery and machine components was unprecedented during the industrial revolution of the late 18th century – 19th century. This gave rise to a need to prolong the life of machine parts by reducing metal to metal contact, hence increasing useful working life of machine components. Lubrication was therefore not unknown, but its necessity was simply amplified during this period. A lot of substances and materials were therefore used as lubricants during this period with oils and fats from natural sources topping the list Haycock et al, (2004). These natural oils had limited stability. Serious efforts were initiated in the 1930s to develop synthetic lubricants for operation over wide temperature ranges (Sharma *et al*, 2006). However, the discovery of Crude oil changed that, soon after, almost all lubricants were made from petroleum products.

A lubricant is a substance designed to separate moving parts thereby minimizing friction and reducing wear. Greases are lubricating fluids that have been thickened with a gelling agent. A functioning grease should remain in contact with, and lubricate, the moving surfaces. It should not leak out under gravity or centrifugal action, or be squeezed out under applied pressure. Greases are the most versatile lubricant and are usually the first choice in lubricant selection due to their ease of application, lower maintenance requirements, fewer sealing requirements, greater effectiveness in stop and start conditions, and overall lower cost (Sharma *et al*, 2006).

The use of petroleum products as lubricants presents its own problems. First is the issue of exhaustibility. It is estimated that world oil reserves will dwindle and by the turn of the next century, there would not be enough reserves for the world demand at that time. Also since the 1970's Oil prices have steadily increased and coupled with dwindling reserves and rising demand, crude oil prices would reach uneconomical levels for common use. Secondly, there is the problem of environmental pollution. Mineral Oils (from petroleum) used as base oil for lubricants are not easily assimilated by the environment (Biodegradability) Aluyor *et al* ,2009).. They present serious pollution problems when dumped into the ecosystem. There is therefore, a growing need to produce lubricants that are renewable, environmentally friendly and cheap. This is an integral part of the emerging "green" culture which stipulates the use of resources in a manner that is efficient, sustainable, renewable with less negative impact to the environment (Nosonovski and Bhanat, 2010).

Biogrease is part of the lubricant market and will likely grow substantially due to economic, environmental and legislative factors (Sharma *et al*, 2006). Biodegradable grease or biogrease can be produced either from natural oils or from chemical synthesis. These synthetic chemicals, even though they have superior performance characteristics to vegetable oils when used as base oils, they are often very expensive to produce. This need for cheap, environmentally friendly and renewable lubricants has given rise to a renewed research interest and today, vegetable oils are drawing attention as biodegradable alternatives for synthetic esters as they are less expensive and are available from renewable sources (Stefanescu *et al* 2011).

Machinery use in applications that require intimate contact with the natural environment, such as agriculture, forestry and some earth moving equipment or the food processing industry all require that a safe (non toxic) substance be used as lubricant Barriga *et a*l, (2006). Also a

significant percentage of all used lubricants find their way into the environment. The use of biogrease will neutralize the effect of improper disposal or discharge of this into the environment.

The use of vegetable oils as energy sources or as feedstock for biodegradable lubricants presents its own dangers. Fear is being expressed by experts over the impact of cultivation of energy crops and its competition with food cultivation. These fears, have necessitated a shift towards natural but inedible or non food oils. There is abundance of some of these natural oils derived mostly from wild sources or from tree borne oil seeds (TBO). Countries like India have already commenced the identification and determination of TBO and their potential as feedstock for energy and other uses (Nosonovsky and Bhanat, 2010).

It is thus clear, that the use of vegetable oils as base stocks for the production of biodegradable lubricants and greases would witness a rise in importance especially with the recent call for efficient and sustainable use of bioresources. In laying the foundations for green tribology, Nosonovsky and Bhanat (2010), listed the use of biodegradable lubricants as one of the pillars of the discipline.

1.2 Statement of Research Problem

Nigeria is the most populous country on the African continent and coupled with its abundant oil reserves, it has become a home to millions of vehicles WHO, (2013). This is apart from, motorcycles, industrial, agricultural and general purpose machinery of all sorts. These machinery require lubrication and presently, the bulk of lubricants are manufactured from mineral (petroleum) based oils. It is estimated that petroleum greases accounted for 98% of all greases

sold in the United States in 2004 (Landsdown, 2004 as in Sharma, 2006). It is also known that a significant portion of lubricants used are released into the environment.

There is no survey on the estimated quantity of grease consumed locally in Nigeria, but the number of vehicles would give a good idea on the quantity of multipurpose grease estimated to be consumed locally in Nigeria. This quantity certainly presents a genuine concern about its disposal because a very significant percentage of that would go to total loss, dry lubrication systems such as those found in earth moving equipment.

Environmental concern continues to increase due to 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 (Erhan *et al*., 2006 as in Othman, 2009). A study in India, also estimated that 40% of all lubricants are released into the environment (Adhvaryu *et al*. 2005 as in Sharma (2006). It can thus, be safely assumed that a reasonable portion of lubricants consumed in this country is released into the ecosystem one way or another, since there is no organized disposal system for such.

The renewed interest in developing cheap, environmentally friendly and renewable lubricants world over has caused many countries to explore resources commonly available to them with a view to reap the benefits. Hence, lots of research has been undertaken in the US for instance on the suitability of using Rapeseed oil, canola oil, sunflower oil and castor oil while in India and some countries in SE Asia, Coconut oil, Rice bran oil, soybean oil, Palm Oil, Groundnut/Peanut oils have all been studied as possible alternative base oils for the production of bio lubricants (Nagendramma and Kaul, 2012). Vegetable oils even though a promising alternative present a veritable challenge since they are a source of food. The danger of using edible oils as base oils is an area of concern since it would mean instead of growing these oilseeds for their food value,

they would be grown solely for the exploitation of their oil for energy and other uses. This obvious conflict has necessitated a shift in strategy towards the exploitation of non-edible oils and tree borne oilseeds (TBO's) which are not used as food especially those that occur in the wild. A study in India, identified over a 100 species of tree borne non-edible oilseeds and their source potential as feedstock for energy and other uses has been reported by Subbarao, (2006) as in Chauhan and Chhiber (2013).

1.3 The Present Research

This research focused on the extraction and use of desert date seed oil as a feedstock for Lithium biogrease. The desert date seed oil was extracted and the oil used as a base oil to produce a simple lithium grease. The Consistency, Drop point and wear characteristics values of this grease was evaluated and compared with a simple, mineral oil based grease. This served to show the viability of *B.aegyptiaca* seed oil as base oil for the production of Lithium grease which can be used safely in agricultural machinery, forestry, mining, food processing and other areas in which the application of environmentally friendly grease is desirable.

1.4 Aim and Objectives

The aim of this research is to investigate the suitability of Desert Date seed oil as base oil with metal (Lithium) soap as thickener for production of biogrease.

1.4.1 The specific objectives

The specific objectives of this work are:

i. To extract the crude oil from the Desert Date seed
ii. To formulate and develop biodegradable Lithium grease from the extracted Desert Date seed oil.

iii. To conduct cone penetration, dropping point and wear characteristics tests on the developed grease.

iv. To carry out comparative analysis of the developed grease with petroleum based Lithium grease in order to determine its suitability as a complement for petroleum based grease.

1.5 Scope of The Study

This research work looked at the suitability or otherwise of using *Balanites aegyptiaca* (Desert date seed oil) to produce biodegradable Lithium grease, by varying the amount of base oil from 70% through 80% by weight to produce a set of biogrease samples and also the addition of the antioxidant, Diphenylamine (DPA) to produce four samples of the bio grease. The resulting biogrease samples were then subjected to consistency tests for worked and unworked penetration (ASTM D217), drop point tests (ASTM D566) and gear wear test (ASTM D4048). A sample of commercially available Multipurpose (Petroleum) Lithium grease (OANDO Crown EP2) (control) was then subjected to the same tests as the biogrease and the results were compared.

1.6 Significance of The Research

The current trend all around the world is towards environmentally friendly lubricants from renewable sources that are envisaged to eventually replace toxic, non renewable mineral oils. Vegetable oils offer a promising alternative but they present a veritable challenge since they are a source of food. This obvious conflict has necessitated a shift in strategy towards the exploitation of non-edible oils and tree borne oilseeds (TBO's) which are not used as food especially those that occur in the wild. *Balanites Aegyptiaca* or desert date seed is a wild growing TBO. It is quite cheap and is easily locally sourced especially in the Sahel belt of the

country where it is in plentiful supply. Its oil has been characterized as a viable feedstock for biodiesel production. There is therefore a need to exploit this resource in the quest for a cheap, renewable and environmentally friendly feed stock for biogrease as an alternative grease to petroleum based grease in case of high cost and scarcity of petroleum. Finally, to obtain a biodegradable grease.

1.7 Limitations

In the course of this research, only three tests were considered namely penetration values, dropping point and wear characteristics, initially copper corrosion test was proposed but due to the high cost it was abandoned. Presently, only Petroleum Training Institute, Effurun, Delta State carries out copper corrosion test.

Even the wear test was improvised as there is no facility in the country to conduct the standard wear tests. Other tests also, need to be carried out to provide a better picture of the characteristics and performance of the developed biogrease from Balanites aegyptiaca with Lithium stearate as thickener.

Other thickeners like Sodium, Calcium, Magnesium and Aluminium soaps have not been utilized.

Also most of the chemical additives proposed were unobtainable in the country. Proposal for imports required huge quantities not suitable for laboratory scale work.

CHAPTER TWO
2.0 LITERATURE REVIEW

2.1 Types of Grease and Their Chemical Composition

Grease according to the American Society for Testing and Materials (ASTM), is a solid to semifluid product of dispersion of a thickening agent in liquid lubricant. Other ingredients imparting special properties may be included. (ASTM D288). By this definition, grease therefore contains three components, the base oil, thickener and additive (see Figure 2.1). The base oil, which is 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, (1981) as in Yaakub, (2008). Common thickeners are soaps and organic or inorganic non-soap thickeners. The majority of greases on the market are composed of mineral oil blended with a soap thickener. Additives serve to enhance performance and protect the grease and lubricated surfaces. Typical grease contains 75%–95% base oil, 5%–20% thickener, and 0–20% additives (Yaakub, 2008).

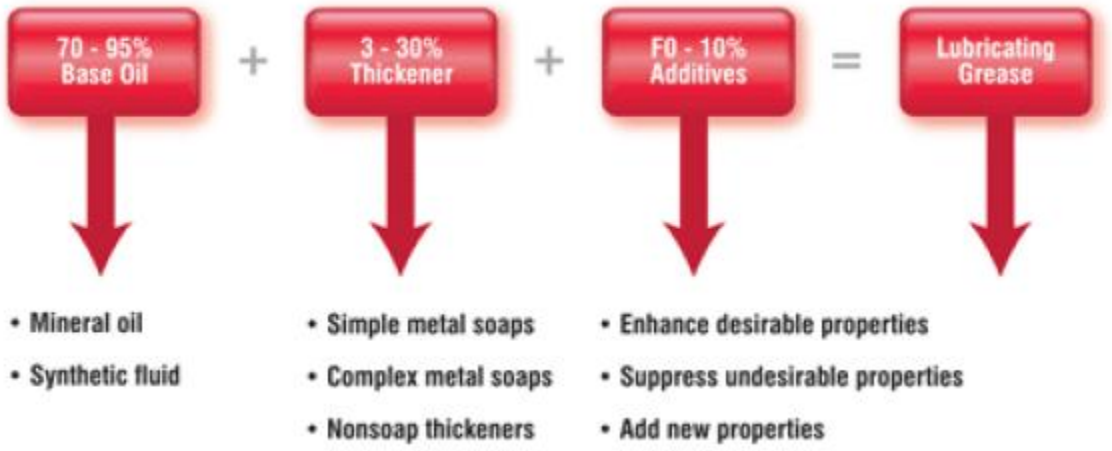


Figure 2.1 Composition of Grease (Yaakub, 2008)

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 (Yaakub, 2008)

2.1.1 Base Oil

Most greases produced today use mineral oil as their fluid component. These mineral oil-based greases typically provide satisfactory performance in most industrial applications. In temperature extremes (low or high), a grease that utilizes a synthetic base oil will provide better stability (Wright, 2008).

2.1.2 Thickener

The thickener is a material that, in combination with the selected lubricant, will produce the solid to semifluid structure. The primary type of thickener used in current grease is metallic soap.

These soaps include lithium, aluminum, sodium and calcium. Lately, complex thickener-type greases are gaining popularity. They are being selected because of their high dropping points and excellent load-carrying abilities. These complex greases are made by combining the conventional metallic soap with a complexing agent. The most widely used complex grease is lithium based. These are made with a combination of conventional lithium soap and a low- molecular-weight organic acid as the complexing agent (Wright, 2008).

Non-soap thickeners are also gaining popularity in special applications such as high-temperature environments. Important non-soap thickeners include clay and polyurea. Bentonite and silica aerogel are two examples of thickeners that do not melt at high temperatures (Wright, 2008).

2.1.3 Additives

Additives can play several roles in lubricating grease. These primarily include enhancing the existing desirable properties, suppressing the existing undesirable properties, and imparting new properties. The most common additives are oxidation and rust inhibitors, extreme pressure, antiwear, and friction-reducing agents (Wright, 2008).

In addition to these additives, boundary lubricants such as molybdenum disulfide (moly) or graphite may be suspended in the grease to reduce friction and wear without adverse chemical reactions to the metal surfaces during heavy loading and slow speeds (Wright, 2008).

2.2 Classification of Greases

Greases are classified in many ways but the commonest way of classification is by the type of thickening agent it contains. Thus if it contains a metallic soap thickener, it would be referred to as soap based grease and could be any of the underlisted greases.

2.2.1 Sodium soap grease

Sodium grease was developed for use at higher operating temperatures than the early hydrated calcium greases. Sodium grease can be used at temperatures up to 121°C 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 (Hughes, 2007).

2.2.2 Calcium soap grease

Calcium or lime grease, the first of the modern production greases, is prepared by reacting mineral oil with fats, fatty acids, small amount of water, and calcium hydroxide (also known as hydrated lime). The water modifies the soap structure to absorb mineral oil. Because of water evaporation, calcium grease is sensitive to elevated temperatures. It dehydrates at temperatures around 79°C at which its structure collapses, resulting in softening and, eventually, phase separation. Greases with soft consistencies can dehydrate at lower temperatures while greases with firm consistencies can lubricate satisfactorily to temperatures around 93°C. 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 an anhydrous (waterless) grease. Since dehydration is not a

concern, anhydrous calcium grease can be used continuously to a maximum temperature of around 110°C.

Calcium complex grease is prepared by adding the salt calcium acetate. The salt provides the grease with extreme pressure characteristics without using an additive. Dropping points greater than 260°C can be obtained and the maximum usable temperature increases to approximately 177 °C. 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 (Hughes, 2007).

2.2.3 Lithium soap grease

Lithium grease is smooth, buttery-textured and by far the most popular when compared to all others. The normal grease contains lithium 12-hydroxystearate soap. It has a dropping point around 205°C and can be used at temperatures up to about 150°C. It can also be used at temperatures as low as (-)35°C. 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 base. 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 Hughes, (2007).

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°C. It is generally considered to be the nearest thing to true multipurpose grease (Hughes, 2007).

2.2.4 Barium soap grease

Barium-soap greases are general-purpose types, valued for their ability to work over a wide temperature range. Their dropping point is approximately 194^{o}C or higher, although they are not intended to be used in continuous operation temperatures above 153^{o}C. Barium-soap greases are chosen for a variety of jobs, especially for nearly all types of bearing lubrications. They have high-soap content. However, this makes this type of lubricant less suitable for use at low temperatures and in very high-speed applications. They have a buttery of fibrous texture and are reddish-yellow or green in color (Hughes, 2007)

2.2.5 Aluminium soap grease

(1) Aluminium grease is normally clear and has a somewhat stringy texture, more so when produced from high-viscosity oils. When heated above 79°C this stringiness increases and produces a rubber like substance that pulls away from metal surfaces, reducing lubrication and increasing power consumption. Aluminum grease has good water resistance, good adhesive properties, and inhibits rust without additives, but it tends to be short-lived. It has excellent inherent oxidation stability but relatively poor shear stability and pumpability (Hughes, 2007).

Aluminium complex grease has a maximum usable temperature of almost 100 °C higher than aluminum-soap greases. It has good water-and-chemical resistance but tends to have shorter life in high-temperature, high-speed applications (Hughes, 2007).

2.2.6 Other greases

A non-soap based grease consists of inorganic substances like molybdenum disulphide and graphite, as well as organic thickeners. The organic thickeners are considered as non-abrasives, which have high capacity to absorb and "hold" the base oil.

2.2.7 Clay based grease

Organo-clay is the most commonly used inorganic thickener. Its thickener is modified clay, insoluble in oil in its normal form, but through complex chemical processes, converts to platelets that attract and hold oil. Organo-clay thickener structures are amorphous and gel-like rather than the fibrous, crystalline structures of soap thickeners. This grease has excellent heat-resistance since clay does not melt. Maximum operating temperature is limited by the evaporation temperature of its mineral oil, which is around 177 °C. However, with frequent grease changes, this multipurpose grease can operate for short periods at temperatures up to its dropping point, which is about 260 °C. 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 (Hughes, 2007).

2.2.8 Polyurea Greases

Polyurea is the most important organic non soap thickener. It is a low-molecular-weight organic polymer produced by reacting amines (an ammonia derivative) with iso cyanates, which results in an oil soluble chemical thickener. Polyurea grease has outstanding resistance to oxidation because it contains no metal soaps (which tend to invite oxidation). It effectively lubricates over a wide temperature range of -20 to 177 °C 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 (Hughes, 2007).

2.3 Uses of Grease

2.3.1 Applications Suitable for Grease

Grease and oil are not interchangeable. Grease is used when it is not practical or convenient to use oil. The lubricant choice for a specific application is determined by matching the machinery design and operating conditions with desired lubricant characteristics. Grease is generally used for:

i. Machinery that runs intermittently or 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 accessible for frequent lubrication. High-quality greases can lubricate isolated or relatively inaccessible components for extended periods of time without frequent replenishing. These greases are also used in sealed-for-life applications such as some electrical motors and gearboxes.
iii. Machinery operating under extreme conditions such as high temperatures and pressures, shock loads or slow speed under heavy load.
iv. Worn components. Grease maintains thicker films in clearances enlarged by wear and can extend the life of worn parts that were previously lubricated by oil Hughes, (2007).

2.3.2 Functional Properties of Grease

i. Grease functions as a sealant to minimize leakage and to keep out contaminants. Because of its consistency, grease acts as a sealant to prevent lubricant leakage and also to prevent entrance of corrosive contaminants and foreign materials. It also acts to keep deteriorated seals effective.

ii. Grease is easier to contain than oil. Oil lubrication can require an expensive system of circulating equipment and complex retention devices. In comparison, grease, by virtue of its rigidity, is easily confined with simplified, less costly retention devices.

iii. Grease holds solid lubricants in suspension. Finely ground solid lubricants, such as molybdenum disulfide (moly) and graphite, are mixed with grease in high-temperature service 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 Hughes, (2007).

2.3.3 Characteristics of grease

As with oil, grease displays its own set of characteristics that must be considered when being chosen for an application. The characteristics commonly found on product data sheets include the following:

2.3.3.1 **Pumpability**. Pumpability is the ability of a grease to be pumped or pushed through a system. More practically, pumpability is the ease with which a pressurized grease can flow through lines, nozzles and fittings of grease-dispensing systems.

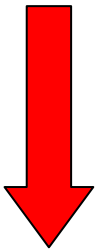
2.3.3.2 **Water resistance**. This is the ability of a grease to withstand the effects of water with no change in its ability to lubricate. A soap/water lather may suspend the oil in the grease, forming an emulsion that can wash away or, to a lesser extent, reduce lubricity by diluting and changing grease consistency and texture Hughes. (2007).

2.3.3.3 **Consistency**. Grease consistency depends on the type and amount of thickener used and the viscosity of its base oil. A grease's consistency is its resistance to deformation by an applied

force. The measure of consistency is called penetration. Penetration depends on whether the consistency has been altered by handling or working. ASTM D 217 and D 1403 methods measure penetration of unworked and worked greases. To measure penetration, a cone of given weight is allowed to sink into a grease for five seconds at a standard temperature of 25°C (77°F) Hughes. (2007).

The depth, in tenths of a millimeter, to which the cone sinks into the grease is the penetration. A penetration of 100 would represent a solid grease while a penetration of 450 would be semifluid. The NLGI has established consistency numbers or grade numbers, ranging from 000 to 6, corresponding to specified ranges of penetration numbers. Table 2.1 lists the NLGI grease classifications along with a description of the consistency Wright, (2008).

Table 2. 1 Grease Classification based on Consistency (Wright, 2008)

NLGI No	ASTM WORKED PENETRATION	CONSISTENCY
000	**445 - 475**	**Semi Fluid**
00	**400 -430**	**Increasing Consistency** 
0	**355 – 385**	
1	**310 – 340**	
2	**265 – 295**	
3	**220 – 250**	
4	**175 – 208**	
5	**130 – 160**	
6	**85 - 115**	**Firm Block**

2.3.3.4 **Dropping point.** Dropping point is an indicator of the heat resistance of grease. As grease temperature increases, penetration increases until the grease liquefies and the desired

consistency is lost. The dropping point is the temperature at which a grease becomes fluid enough to drip. The dropping point indicates the upper temperature limit at which a grease retains its structure, not the maximum temperature at which a grease may be used (Hughes, 2007).

2.3.3.5 **Oxidation stability.** This is the ability of a grease to resist a chemical union with oxygen. The reaction of grease with oxygen produces insoluble gum, sludges and lacquer-like deposits that cause sluggish operation, increased wear and reduction of clearances. Prolonged exposure to high temperatures accelerates oxidation in greases (Hughes, 2007).

2.3.3.6 **High-temperature effects**. High temperatures harm greases more than they harm oils. Grease, by its nature, cannot dissipate heat by convection like a circulating oil. Consequently, without the ability to transfer away heat, excessive temperatures result in accelerated oxidation or even carbonization where grease hardens or forms a crust. Effective grease lubrication depends on the grease's consistency. High temperatures induce softening and bleeding, causing grease to flow away from needed areas. The mineral oil in grease can flash, burn or evaporate at temperatures greater than 177°C (350°F) (Hughes, 2007).

2.3.3.7 **Low-temperature effects.** If the temperature of a grease is lowered enough, it will become so viscous that it can be classified as a hard grease. Pumpability suffers and machinery operation may become impossible due to torque limitations and power requirements. As a guideline, the base oil's pour point is considered the low-temperature limit of a grease (Hughes, 2007).

2.4 Manufacture of Lubricating Greases

The basic process for manufacturing of soap based greases is similar. This requires the reaction of the base oil with a thickener at high temperatures, typically exceeding 138 – 149°C. For non soap greases, like clay thickened grease, room temperature reactions and high mechanical shearing can be used to manufacture the grease (Honary and Richter, 2011).

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 (Yaakub, 2008)

The most common additives found in grease are anti-oxidants to prolong the life of grease, anti-corrosion agents to protect metal against attack from water or corrosive elements, anti-wear agent and extreme pressure additives to guard against excessive wear due to metal to metal contact.

Environmentally friendly lubricating greases may be produced by solely replacing the mineral base oil with vegetable oil.

2.4.1 Basic Process for Manufacturing Lithium Grease

When Lithium hydroxide is reacted with the base oil, a considerable amount of foam is generated along with water. This water will need to be boiled off at high temperatures, often exceeding 200C. To remedy the foaming problem, high pressure sealed vessels may be used to contain the foam until the reaction is completed. The water is eventually removed by venting the pressure vessel. Feeding lithium into the oil at a slower rate can help reduce foaming but it increases the

process time.Proper processing will ensure that the highest amount of grease is produced consistently from the same amount of thickener and the same quantity of oil (Honary and Richter, 2011).

2.5 Standard Tests for Grease Performance

Over the years, several institutes like: International Standards Organisation (ISO) – American Society for Testing and Materials (ASTM), - IP - *Deutsches Institut für Normung* (*DIN*; in English, the German Institute for Standardization) DIN, *Association Francaise des Normalisation* (AFOR), etc. have standardised tests that describe properties or performance attributes of lubricating greases.

Greases can thus be tested by a variety of methods, with each test providing useful information about a grease. The following properties of grease should however, be evaluated; oxidative stability, rheology, low temperature properties, friction and wear properties, and hardness (Sharma *et al*, 2011).

These tests do not only serve as screening tests which give directional indications of what can be expected when a grease is placed in service in a specific application, but also as physical standards for manufacturing control. Most of the grease tests that have been standardised, define or describe properties that are related to the performance tests in actual or simulated operating mechanisms. However, it must be recognised that they are laboratory tests and direct correlation between laboratory tests and field performance is rarely possible since the tests never exactly duplicate service conditions, and service conditions are never identical even in two outwardly similar applications. For these reasons, an understanding of the intent and significance of the tests is essential for those involved with the use of lubricating grease (Total, 2003).

2.5.1 Hardness/Consistency tests

2.5.1.1 Cone Penetration

The hardness of grease is its resistance to deformation by an applied force and is generally measured by the ASTM D 217 and D 1403 methods (Total, 2003).

Method/Standard: ASTM D 217; DIN 51804-T1 / ISO 2137; NF T 60-132 / IP50

Consistency is commonly measured by the Cone Penetration. In this test a normalised cone is allowed to sink for 5 sec., under its own weight into a sample of grease held at 25°C. The depth that the cone penetrates into the grease is expressed in tenths of a millimeter and reported as the penetration of the grease. If the cone sinks in 47.5 mm or more, the very soft grease is classified as NLGT 000, the softest classification. If it only sinks 8.5 mm or less, the hard grease is classified as NLGI 6, the hardest classification. Since the cone will sink further into soft greases, higher penetrations indicate softer greases. Current classification of greases by the National Lubricating Grease Institute (NLGI) places the softest grease a 000 grade (all "0" grades are normally referred to as central lubrication greases) and as the stiffest a grade 6. Note that grade 6 grease has the same consistency as a bar of soap. In fact grades 5 and 6 are no longer widely used. The stiffest grades available on the market presently are grade 3 or 4 (Sharma et al, 2008) and special procedures using one-quarter and one-half scale cones, method ASTM D 1403, are used for determining the penetration where samples are limited in quantity (Total, 2003).

2.5.1.2 Mechanical Stability

Method/Standard: ASTM D 217 / DIN 51804-T1 / ISO 2137; NF T 60-132 / IP50

Sample is worked for 60 strokes or more in a standard grease worker.

Often the grease is worked 100,000 strokes, but also 5,000 and 10,000 strokes are used. It is accepted that these **W .....** results give a basic impression on the **grease stability**.

In some cases some water is added to the grease to measure the stability when the grease contains water. This is normally done for grease used in wet environments.

2.5.1.3 Drop Point Procedure

Method/Standard: ASTM D566; IP 396 /NF T 60102C

The dropping point of a grease is the temperature at which a drop of oil released by the grease falls from the orifice of a test cup under prescribed test conditions. Materials such as conventional soap thickened greases do not have a true melting point but have a melting range during which the material becomes progressively softer.

In a standard cup containing grease, the grease is heated in a special computer controlled heating furnace (usually Mettler), within which the temperature at which a drop of material falls from the cup is detected and reported by electronic means. The dropping point of a grease is commonly, but mistakenly, taken as measure of the service capabilities of a grease at elevated temperatures.

In general, the dropping point is the temperature at which the grease passes from a semisolid to a liquid state under the conditions of test. This change in state is typical of greases containing as thickeners soaps of conventional types. Greases containing as thickeners materials other than conventional soaps can, without change in state, separate oil. This test method is useful to assist in identifying the grease as to type and for establishing and maintaining bench marks for quality control. The dropping point of a grease is not considered to have any bearing on service performance other than that normally a grease cannot be expected to resist leakage at temperatures above its dropping point, it does not establish the maximum usable temperature for the grease but a rule of thumb is however that dropping point minus 30% is operating

temperature. The results are considered to have only limited significance with respect to service performance as dropping point is a static test (ASTM D566, 2002)

2.5.1.4 Oxidation Stability

Method/Standard: ASTM D 942; DIN 51808 / IP 142

The reaction with oxygen may lead to deterioration of lubricating grease.

This test conducted in the Norma-Hoffman oxidation bomb evaluates resistance of lubricating greases in a closed container to oxidation under specific conditions of static exposure.

In this test, each of the five dishes in the bomb is filled with 4 g of the grease to be tested. The bomb is then sealed and pressurised to 110 psi (7.7 kg/cm^2) with oxygen and placed in a bath held at 99°C. The pressure in the bomb is recorded at prescribed intervals throughout the test. At the end of the specified test time, usually 100, 250 or 500 hr, the pressure drop is calculated and reported. The drop in pressure is the net change resulting from absorption of oxygen by the grease and the release of CO_2 from the grease. Frequently, the results of this test are reported as indicative of the oxidation stability of a grease; however, it is a static test. It is not intended for the prediction of the stability of grease under dynamic conditions (Total, 2003).

2.5.1.5 Roll Stability

Method/Standard: ASTM D 1831 (modified)

The ability of a grease to resist changes in consistency during mechanical working is named its roll stability or shear stability. A variety of laboratory tests are used to evaluate the roll stability of greases, but the two that have been standardised are the change in penetration after prolonged working in the ASTM D 217 grease worker, and the change in penetration after severe rolling in

the ASTM D 1831 roll stability worker. In the Roll Stability Test a small sample (50 g) of grease is rolled at 165 rpm for a certain time at a certain temperature. Using a steel cylinder which contains a 5 kg heavy round steel block. Worked penetration at 25°C is determined on the grease before and after rolling. Due to the small size of the sample, working and penetrations are performed on ASTM D 1403 one-quarter or one-half scale equipment.

TOTAL uses a temperature of up to 100°C to achieve a closer to practice test. Furthermore TOTAL has prolonged the duration of the test from 2 to 4 or even 100 hrs to determine the mechanical stability of their greases under very severe conditions.

In both of these tests, the change in consistency with mechanical working is reported as either the absolute change in penetration or the percent change in penetration. While both tests are used widely to indicate mechanical stability requirements, the significance has never been accurately determined. It is believed that the changes in worked penetration in these tests are an indication of mechanical stability, and are directionally indicative of the changes in consistency that a grease can undergo in service.

2.5.1.6 Oil Separation (during storage)

Method/Standard: ASTM D 1742; ASTM D 6184; IP 121 / DIN 51817; NF T 60-191

Grease must release oil slowly in service to provide effective lubrication. Some oil release resulting in free oil on the grease surface is normal in storage. However, excessive separation of oil while the grease is in storage may result in loss of the user's confidence in the product. The tendency of a grease to separate oil during storage is predicted by ASTM D 1742 / IP 121 / DIN 51817 / NF T 60-191and ASTM D 6184.

In ASTM D 1742 a sample of grease supported on a 200 mesh screen is subjected to air pressure and placed in an oven at 25°C for 24 hrs. Any oil that seeps from the grease is collected, weighed and reported as the percent by weight of oil separated.

IP 121 / DIN 51017 / NF T 60-191 are more or less the same procedure, but grease is subjected to metal weight pressure and placed in an oven at 40°C for 42 or 168 hours.

ASTM D 6184 is more or less the same procedure, but grease is subjected to metal weight pressure and placed in an oven at 100°C for 30 or 50 hours.

These tests are correlated directly with oil separation which occurs in grease pails in storage, and are directionally indicative of the separation that may be expected in other sizes of containers. It is not suitable for greases softer than NLGI. No.1, and is not intended to predict the bleeding tendencies of grease under dynamic service conditions. A maximum of 1 - 5% bleeding is considered to be normal under storage conditions (also depending on thickener).

2.5.1.7 Water washout test

Method/Standard: ASTM D 1264; DIN 51807-T2 / IP 125

The environment in which a grease must perform is important but is often neglected in grease selection. A wet environment, whether it be just moist air conditions or heavy direct water washing action, can affect many greases and is an important factor to consider when selecting grease for any service.

When water intrudes into grease lubricated equipment the grease may become softer (may even become semi-fluid) or stiffen, it may emulsify or reject water, its adhesive properties may change, and metal surface protection may become inadequate from corrosive water action (rust).

The ability of a grease to resist washout under conditions where water may splash or impinge

directly on a bearing is an important property in the maintenance of a satisfactory lubricating film.

Comparative results between different greases under the prescribed test conditions can be obtained with this test, but the results may not necessarily predict field performance.

The test uses a specific ball bearing, equipped with front and rear shields having a specified clearance.

It is packed with 4 g of the test grease and then rotated at 600 rpm for 1 hr while a jet of water at 80°C impinges on the bearing housing. At the end of this time the bearing will be removed, dried, and the percent weight of the grease lost determined. The test is generally considered to be an useful screening test for greases, that are being used where water washing may occur, such as wheel bearings wet end-bearings on paper machines, and in the steel industry.

2.5.1.8 Water spray off test

Method/Standard: ASTM D 4049

The objective of this test is to measure the resistance to spray off of a grease. A predefined grease film is put on a test plate. The test plate is exposed to a water spray (22 or 40 PSI). The result after a set time is expressed in weight loss of the plate with grease. This comparison test, can give an indication of the adhesive character of a grease on metal exposed to water sprayed under pressure on the grease (Total, 2003).

2.5.1.9 Behaviour in the presence of water

Method/Standard: DIN 51807-T1

This test is developed to test the behaviour of grease against water under predefined static circumstances. A grease sample is put on a glass strip with the help of a template. The sample

thickness is about 1 mm. The glass strip is then submerged into distilled water and put in an oven. The duration of the test is 3 hours at 40°C or 90°C.

After the test the sample on the glass strip is immediately valued with the naked eye. The scale in Table 2.2 is used for rating:

Table 2.2 Scale for effect of water on grease Total, (2003)

Scale	Effect
0	No change.
1	Slight change, change of colour, slight water adhession on the grease.
2	Medium change, grease starts to dilute visible through white/yellow slime building on the grease, troubling of the water.
3	Big change, part or complete dilution of the grease into the water, building of milky white oil in water emulsion.

2.5.1.10 Grease life in ball bearings at elevated temperature high speed and load:FAG FE 9

Method/Standard: DIN 51821

Purpose: to determine the life of lubricating greases in rolling element bearings under realistic test conditions. The rolling-element bearing fitted in the apparatus as the test piece is filled with a specified quantity of the grease under examination. The test is conducted at a selected temperature, rotating speed and axial load. The lubrication conditions in the bearing change over a long period. Bearing failure is regarded as having occurred when the motor fails to drive the bearing under test.

Table 2.3 Details of grease life test Total, (2003).

Quantity of grease	2 mg
Test temperature	Adjustable up to +250°C
Rotational speed	3,000 or 6,000 rpm
Thrust force	1,500, 3,000 or 4,500 N
Bearing failure with power consumption of driving motor	520 Watt at 6,000 rpm 320 Watt at 3,000 rpm

2.5.1.11 Grease life in ball bearings at elevated temperature

Method/Standard: ASTM D 3336

The test method covers the evaluation of the performance of lubricating greases in ball bearings operating under light loads at high speeds and elevated temperatures. The ASTM D 3336 test evaluates the performance of lubricating grease in a (20 mm) ball bearing operating under light loads at temperatures up to 177°C (usual intervals 120, 150, 177°C) and shaft speeds of 10,000 rpm. Tests are run to failure or until the required number of test hours without failure has been completed (ASTM D 3336, 2002).

2.5.1.12 Copper Corrosion

Method/Standard: ASTM D 4048; IP 112 / DIN 51811

These methods are used to detect substances in lubricating grease which could corrode copper. Since copper and copper alloys are used in bearings it is essential that greases not corrode such materials.

The tests are similar, (commonly referred to as “Copper Strip Tests”) and involve a cleaned and

smoothly polished copper strip immersed vertically in the grease sample. In the ASTM method the strip is totally immersed, in the IP method the strip is 2/3 immersed. The assembly is placed in an oven for a given time and temperature, then removed and cooled. The strip is cleaned and observed for staining or corrosion, and rated either in words and/or by a numerical rating system. With the ASTM/DIN method a comparison of the copper strip condition after test is made against a reproduction ASTM Copper Strip Standard (ASTM D 4048, 2002).

The length of test and test temperature is not specific, but should be stated when reporting results. The standard test procedure recommends 100°C, but other mutually agreed-upon test temperatures are permitted. The time may be specified as 3 hours, 24 hours or 7 days or may be established by mutual agreement (Total, 2003).



Plate 2.1.: ASTM Copper Strip Corrosion Standards

2.5.1.13 Dynamic Rust Test (EMCOR test)

Method/Standard: IP 220 / DIN 51802; ISO CD 11007

In this test up to eight 30 mm, double row self-aligning ball bearings are mounted in pillow

blocks on a common bed plate and driven at 80 rpm by a common shaft. At least two bearings are required for a test. The standard pillow blocks are plastic. Each bearing is packed with 10 g of the test grease with no grease in the housings. After assembly, the bearings are run for 30 min to distribute the grease. The housings are then opened and 10 ml of distilled water (or IP135 synthetic sea water) is added to each side of each lower housing.

After assembling the bearings are run for two 8 hrs periods followed by two 16 hrs shutdowns, and one 8 hrs period followed by one 108 hrs shutdown. At the end of this time (164 hrs) the bearings are disassembled and the outer races examined for rust and corrosion. The races are rated on a numerical scale from 0 to 5 with 0 being completely free of rust.

2.5.2 Extreme Pressure Tests

While little correlation has ever been obtained between the results of laboratory Extreme Pressure (EP) and service performance tests represent the only language that describes these properties at reasonable cost. Here after two methods are described

2.5.2.1 Four (4) Ball Weld Test

Method/Standard: ASTM D 2596

This test derives its name from the 12.7 mm (0.5 in) diameter through hardened chrome alloy steel balls of 64066 Rockwell C hardness, that are used as test specimens (Plate 2.2). It provides a method for determining the load carrying properties of lubricating grease in terms of the Load-Wear index and the Weld Point.

The three lower balls are locked firmly in a cup filled with test grease. The upper ball, which is held in a chuck, is in point contact with each of the lower balls and may be rotated against them under load at a speed of approximately 1770 rpm.

The grease sample, which is brought to a temperature of about 27°C, is subjected to a series of 10 sec tests at increasing loads until welding occurs. After each 10 sec test the scar diameters on the three stationary balls are measured and recorded and all four balls discarded.

The Load-Wear Index, in kilograms, is calculated from the scar diameters corresponding to the various loads. The weld point is reported in kilograms, definition of weld load: "weld point under the conditions of this test the lowest applied load in kilograms force (or Newtons) at which the rotating ball seizes and then welds to the three stationary balls, indicating the extreme pressure level of the lubricating grease has been exceeded". Suggested form for Recording Test Results (Kgf) 80, 100, 126, 160, 200, 250, 315, 400, 500, 620, 800.

ASTM states: "This test method, used for specification purposes, differentiates between lubricating greases having low, medium and high levels of Extreme Pressure properties. The results do not necessarily correlate with results from service".



Plate 2.2: Steel Balls used for grease testing Total, (2003)

2.5.3 Wear Prevention Tests

In testing wear prevention qualities of lubricating greases, two methods, ASTM D 2266 and DIN 51350-T5, are among the most frequently used. These methods, use four test balls in which the test parts are half-inch steel balls, in point contact, with one rotating ball. The two methods are listed in the next section.

2.5.3.1 Four (4) Ball Wear Test

Method/Standard: ASTM D 2266 / IP 239

. This test is similar in principle to ASTM D 2596, the four ball Extreme Pressure test, but the machine is much more sensitive and the applied load is limited to 40 kg (392 N) rather than the 800 kg of the Extreme Pressure machine.

The same composition and hardness steel balls as ASTM D 2596 are used. At the lighter loading, seizure or welding does not occur and the material removed from the balls is the result of wear.

The test is run for 60 min at 1,200 rpm with a load of 40 kg. The grease sample is held at 75°C. At the end of the test sizes of the wear scars on the three stationary balls are measured and reported.

The test is intended to determine the relative wear preventive characteristics of greases in sliding steel on-steel applications. It is not intended to predict wear characteristics with other metal combinations, and cannot be used to differentiate between Extreme Pressure and non Extreme Pressure greases Total, (2003).

2.5.3.2 Four (4) Ball Wear Test

Method/Standard: DIN 51350-T5

This test uses the same test rig as mentioned under test DIN 51350-T4.

The Table 2.4 shows the procedures:

Table 2.4 Test Procedures for 4 Ball wear tests Total, (2003)

Procedure	Load (N)	Running Time (min)
C	150	60 ± 0.5 m
D	300	60 ± 0.5 m
E	1000	60 ± 0.2 sec

The grease is tested in a four ball system consisting of a rotating ball (running ball) sliding with the appropriate test force (C, D or E) on three balls identical to it (standing balls) (tests are done twice). After the test duration the scar diameter of 2 sets of standing balls is measured. This then leads to the reported mm's wear Total, (2003).

2.5.4 Other physical and performance tests for grease

Other tests are also used to assess particular performance characteristic or grease property and can be summarily grouped under the following general headings:

2.5.4.1 Low Temperature Flow Pressure

Method/standard: DIN 51805

To determine the lowest usable temperature of a grease. A sample of grease at a temperature of, for example, -35°C is placed in a sample holder. Afterwards the grease is put under pressure. The pressure is increased by step until the grease is released from the sample holder. The pressure

needed to release the grease is expressed in mbar. If a grease is known to be less pumpable the results can be expressed in °C at a maximum pressure of 1,400 mbar **Total, (2003).**

2.5.4.2 Penetration at Low Temperature

Method/standard: NF T 60-171

To determine the lowest usable temperature of a grease. A sample of grease at a temperature of, for example, -35°C is placed in a sample holder. Afterwards the grease is put under pressure. The pressure is increased by step until the grease is released from the sample holder. The pressure needed to release the grease is expressed in mbar. If a grease is known to be less pumpable the results can be expressed in °C at a maximum pressure of 1,400 mbar

2.5.4.3 Apparent Viscosity (SOD)

Method/standard: ASTM D 1092

By carrying out apparent viscosity determinations at low temperatures (method ASTM D 1092) the pumpability and flow properties of a grease at low temperatures can be determined. Greases designed for extremely low (sub-zero) temperature service should not stiffen and offer excessive resistance to rotation of bearings after soaking at low temperatures (Total, 2003).

The following methods (IP 186 and ASTM D 1478) measures starting and running torque's of small lightly loaded ball bearings at temperatures down to -54°C Total, (2003).

2.5.4.4 Low Temperature Torque

Method/standard: ASTM D 1478

The test bearing is fully packed with the test grease, installed on a spindle that can be rotated at 1 rpm, and inserted in a cold box, which can be maintained at any temperature down to -54°C. The outside of the bearing housing is connected through a string assembly to a scale so that the

retraining force can be measured. After 2 hours, the motor is started, and the initial restraining force recorded. As rotation continues, torque drops, the restraining force is again recorded after running for 60 minutes. The two values are multiplied by the length of the lever arm and the products reported as the starting and running torque's, in N-m units for the grease (Total, 2003).

2.5.4.5 Low Temperature

Method/standard: torque IP 186

This method of test determines the resistance caused by the grease at sub-zero temperatures down to -73°C in an axially loaded ball-bearing rotating at 1 rpm. Although the apparatus is of a different design the method of test is basically very similar to that of ASTM D 1478. A test bearing is packed with test grease, installed on a loaded spindle, that can be rotated at 1 rpm, and installed in a sealed unit which is immersed in a fluid cooling bath. The temperature of the cooling bath is steadily reduced to the test temperature, which may be down to -73°C over 1-1 1/2 hours. After 2 hours the motor is started and the initial restraining force recorded, after running for a period of time the restraining force is again measured. The starting and running torque's are then determined and reported in mNm units (Total, 2003).

2.6 Previous Research Work

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 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 (Adhvaryu *et al*, 2005 as in Othman, 2009)

2.6.1 Review of Related Works

The first greases formulated were bio-based during 1400 B.C. which tallow was utilized to lubricate chariot wheels. From the year 1859, when petroleum grease was first manufactured it is estimated that about 98% of the petroleum grease dominated the marketplace in 2004. (Biresaw and Mittal, 2008 as in Sharma *et al* 2006).

Recently, due to increasing petroleum prices, the use of bio-based oleochemicals as lubricant fluids, metalworking fluids and greases has increased dramatically. There were many researchers working on formulating biobased 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 (Othman, 2009).

Sukirno *et al* (2009) synthesized Lithium Biogrease from waste palm oil. Evaluation of anti wear properties using the both the ball wear and gear wear tests showed that it has superior antiwear properties than mineral oil grease.

Othman (2009) also produced a Lithium biogrease from waste cooking oil. The work involved the grease synthesis and the determination of depth penetration, dropping point and oxidation stability of the biogrease.

In Nigeria, even though, some work has been done and still ongoing in the area of biofuels, little has been done in Tribology especially on biobased grease. Researches in biobased tribology are scarce but some work has been undertaken.

Idris (2012) investigated the lubricating properties of Jatropha oil, castor oil and neem oil blend. The objectives of the work included examining the physicochemical properties of these oils, determining their viscosities at various temperatures, their hydrodynamic behavior and lubrication effectiveness of each oil blend. The following tests were carried out; viscosity using Brookfield viscometer, flashpoint and fire point using Cleveland open cup method, PH value by immersion PH electrode and specific gravity using a density bottle.

Awoyale and Odubiyi produced and tested biogrease from black date (*C. schweinfurthi*). The work involved extracting the oil from black date using solvent extraction and production of the grease using calcium hydroxide ($Ca(OH)_2$) as thickener and stearic acid as additive. Penetration test, dropping point and Biodegradability tests were carried out using Oando and Texaco conventional greases as controls.

Olisakwe *et al* (2011) did a comparative study and determined that both Jatropha curcas and Thevetia peruviana seed oils were suitable base oils for the production of biogrease. The work involved extracting the respective oils and producing a Lithium grease from the oils and various blends.

Samuel *et al* (2012) produced and tested Aluminium grease produced from palm kernel oil. The work involved production of aluminium soap grease and the effects of varying both the metallic soap content and the fat content on the physical properties/characteristics like relative density, melting temperature range and solubility of the grease samples.

Samuel (2014) produced and tested Biogrease from Jatropha curcas seed oil. The work involved the extraction (Mechanically) of Jatropha seed oil and the synthesis of a sodium soap grease

from the oil. An evaluation of evaporation loss, heat stability, viscosity and oil separation were carried out for the grease.

2.6.2 Research Gap

There is a huge potential in identifying suitable TBOs for the production of biogreases and biolubricants since they are a cheaper alternative to vegetable oils coupled with the added advantage of not being used as a food source. Nigeria is richly blessed with many TBOs and desert date is one such resource. It has been characterized as a suitable feedstock for biodiesel production but no investigation has been done on its suitability or use as a bio lubricant. Literature consulted showed no reported research on the use of *balanites aegyptiaca* seed oil as base oil for biolubricants in general and biogrease in particular. The objective of this study therefore, was to investigate the suitability of using the desert date seed oil as base oil for biogrease and also study some properties of the developed biogrease in comparison with a commercially available mineral based Lithium grease. These properties are worked and uworked penetration, dropping point and anti wear characteristics. This work therefore is aimed at filling that gap and providing a window into the further exploitation of this natural resource.

2.6.3 Desert date - *Balanites aegyptiaca* (Aduwa)

Balanites aegyptiaca is a species of tree, classified either as a member of the Zygophyllaceae or the Balanitaceae see Plate 3.1. It has many common names, It is called desert date, soap berry tree or bush. It is known as lalob or heglig (hijlij) in Arabic, while in Hausa it is called Aduwa and in Swahili Mduguyu Chothani and Vaghasiya, (2013).



Plate 2.1 Balanites aegyptiaca tree

Balanites aegyptiaca is native to much of Africa and parts of the Middle East. The tree is mostly found in the Sahel-Savannah region across Africa but it can also be found in many kinds of habitat, tolerating a wide variety of soil types, from sand to heavy clay, and climatic moisture levels, from arid to subhumid. It is relatively tolerant of flooding, livestock activity, and wildfire Chothani and Vaghasiya, (2013).

The fruit is a green, egg-shaped drupe, which turns yellow as it ripens see Plate 3.2. Inside the date-like form is an oleaginous kernel from which vegetable oil can be produced. The term "Egyptian mirobalan" is used only for the unripened fruit. The oil is pale yellow in color, mild in smell and taste, and solidifies below 5° C (Burkill, 1985).



Plate 2.2: Desert date fruit

2.6.3.1 Composition of Desert date Oil

The stone of the fruit contains 44% unsaturated oil and the kernel is also rich in protein (26 to 30%) Gutti *et al*, (2012).

Owing to its composition (30% saturated fatty acids, 70% unsaturated fatty acids: oleic and linoleic), the oil has a low viscosity.

Palmitic acid: 14.73%

Stearic acid: 9.406%

Linoleic acid: 75.863%

The proportion of unsaponifiable matter is less than 1%. Gutti *et al*, (2012)

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Introduction

The development and testing of a biodegradable grease from the seed kernel of desert date fruit (see Plate 3.2) would involve extraction of the oil, determination of some of its physical properties like viscosity, testing its suitability for the formulation of biogrease by varying the percentage composition of the oil with a lithium soap thickener and testing the respective samples thus obtained against a commercially available mineral based grease.

3.2 Seed Preparation

3.2.1 Materials

1. Balanites aegyptiaca (Desert date) seeds

3.2.2 Equipment

1. Plastic Buckets
2. Plastic Mats
3. Small Hammer
4. Plastic Bowl
5. Old Car Engine Flywheel

3.2.3 Procedure For Seed Preparation

Samples of *B. aegyptiaca (Aduwa)* seeds (see Plate 3.2) were obtained from Sabon Gari market in Zaria. These were decorticated (i.e removing the hard yellow peels) by soaking in water for three days see Plate 3.3. The soft, fleshy glycoside, was then removed by vigorous rubbing by hand, to obtain the hard stony seeds. The hard *Balanites aegyptiaca* (Desert date) seeds were then sun dried for a 3-4 days to remove any moisture from the washing process. The resulting dry seeds were then crushed on an old engine flywheel using a metal hammer to obtain the oil bearing kernel (Gutti *et al* 2012), Ajayi and Folorunsho, 2013).



Plate 3.1 Desert date seeds soaked in water

3.3 Oil Extraction

3.3.1 Materials

1. Desert date seeds
2. n- Hexane.

3.3.2 Equipment

1. Electric Kitchen Blender (Eurolex)
2. Conical Flask (500ml) Pyrex England
3. Round Bottom Flask 200ml Pyrex England
4. Soxhlet reflux apparatus
5. Retort Stand
6. Water Bath BUCHI Model R10 Type:225272 (3700W)
7. Chiller CHURCHILL Instrument, Middlesex England

3.3.3 Procedure for Extraction Of Base Oil

The oil was extracted by first milling the kernels using a blender. Solvent extraction was then used to extract the oil. The milled kernels were then filled in to a thimble and immersed in n-hexane in a round bottomed flask. The flask containing the mixture of milled kernels and n-hexane was connected to a Soxhlet extraction apparatus immersed in a hot water bath,(Plate 3.4) where the pure *Balanites ae*. oil was collected in a suitable container for the production of the grease. Upon extraction of the oil from the miscella, a fresh charge was placed in the thimble and the process repeated. The procedure was then repeated, until sufficient quantity of the oil was obtained. See Plates 3.5 and 3.6. The oil collected was heated to 65°C for about 20 minutes to remove any traces of solvent.



Plate 3.2 Soxhlet apparatus in hot water bath



Plate 3.3 Oil collected from the Soxhlet apparatus and the residue



Plate 3.4 Pure Desert date seed oil

3.4 Viscosity Measurement

3.4.1 Materials

1. 7ml *Balanites aegytiaca* seed oil

3.4.2 Equipment

1. Brookfield Viscometer Model RVT, Serial No:43320
2. Magnetic Stirrer Hot plate Make: Searchtech Instruments Model; 78HW - 1
3. Mechanical Stirrer
4. 200ml Beaker
5. Thermometers



Plate 3.5 Brookfield Viscometer

3.4.3 Procedure for Determination of Viscosity

150 ml of the oil sample was poured into 800ml beaker and two thermometers immersed. The average temperature of the two thermometers was taken as the initial temperature of the oil To.

The beaker was then appropriately positioned on the Brookefield viscometer and the spindle lowered into the oil until the level mark was reached. The spindle NO 3 was selected and fixed on the viscometer. The viscometer was then left to run until oscillation. It was then stopped and the reading on the dial gauge taken which gave the corresponding value of the dynamic viscosity in centipoises at room temperature (30°C).

The sample was now placed on the magnetic hot plate and the stirrer immersed. The sample was then heated and stirred continuously, until the temperatures on the thermometers reached the

averaged value of 40°C, the sample was now quickly removed and placed under the viscometer to obtain the value of dynamic viscosity at 40°C.

This was repeated in steps of 20°C from 40°C through 100°C.

The kinematic viscosity of the oil was determined using the formula

$\gamma = \mu / \rho$

Where :

γ= Kinematic Viscosity (m^2/s)

μ= absolute or dynamic viscosity (Ns/m^2)

ρ= density (Kg/m^3)

3.5 Grease Formulation

3.5.1 Materials

1. *Balanites aegyptiaca* seed oil
2. Lithium Hydroxide
3. Stearic Acid
4. Diphenylamine (DPA)

3.5.2 Equipment

1. Digital Scale OHAUS Adventurer Pro Model AV 264 (0-250g) Accuracy 0.0001g
2. Crucible
3. Retort Stand
4. Electric Plate Heater
5. Pyrex Beaker 500ml

6. Stainless Steel stirrer
7. Electric Kitchen Blender (Eurolex)

3.5.3 Procedure for Grease Production

Since grease by definition, is a solid to semi fluid mixture of a fluid lubricant, thickener, and additives, production of grease would simply be a process that would involve preparation of the thickener and the base oil and then later additives. Thickener preparation is carried out by saponification which is a process of reacting fatty acid with metals/metallic hydroxides to produce soap and water at a temperature of about 160°C. The water produced as a by-product of the reaction is evaporated due to the very high reacting temperature of the mixture. The soap produced under this manner is referred to as thickener or thickening agent (Olisakwe *et al*, 2011).

In this study, the thickening agent used was lithium stearate (lithium soap) which is a simple soap. The mixture of the base oil and lithium stearate was cooked and blended for 30 mins at 160°C and allowed to cool to 85°C. No additive was added to the blend. The mixture was then continuously stirred until the temperature reached room temperature of 35°C. Further milling was then carried out at room temperature to obtain a homogeneous mixture.

This procedure was repeated with varying quantities of desert-date seed oil, Lithium Hydroxide and Stearic acid on percentage weight basis, and a set of grease samples were obtained.

The procedure was also repeated and upon cooling of the mixture to 85°C, the anti oxidant Diphenylamine (DPA) was added to the blend. The mixture was then continuously stirred until the temperature reached room temperature of 35°C. Further milling was then carried out at room temperature to obtain a homogeneous mixture (Olisakwe *et al*, 2011).

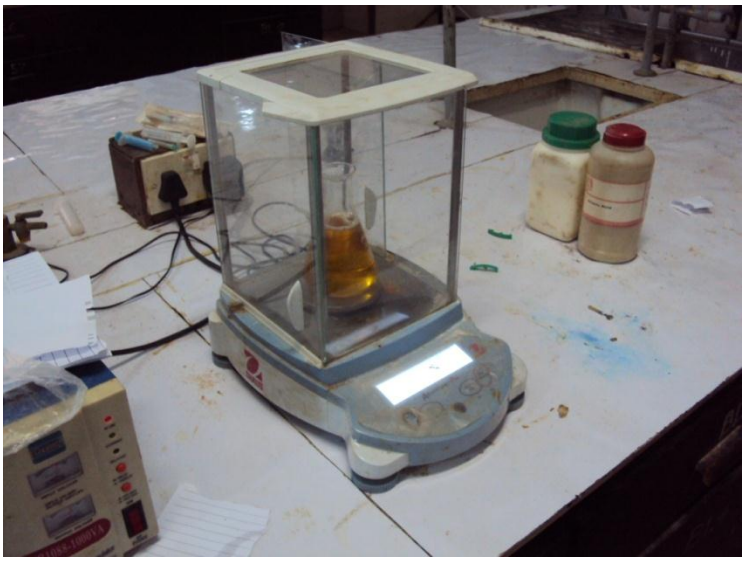


Plate 3.6 Weighing the base oil



Plate 3.7 weighing the lithium stearate



Plate 3.8 The grease production set up showing the beaker and thermometer



Plate 3.9 Adding the soap to the base oil



Plate 3.10 Biogrease samples produced

3.6 Grease Testing

3.6.1 Materials

1. Biogrease samples

3.6.2 Equipment and Instrumentation

1. Grease Worker (Mechanical)

2. Grease cup

3. Spatula

4. Penetrometer



Plate 3.11 Mechanical Grease Worker



Plate 3.12 Penetrometer



Plate 3.13 Grease cup and Grease worker



Plate 3.14 Filling the grease cup and the grease worker with the biogrease sample

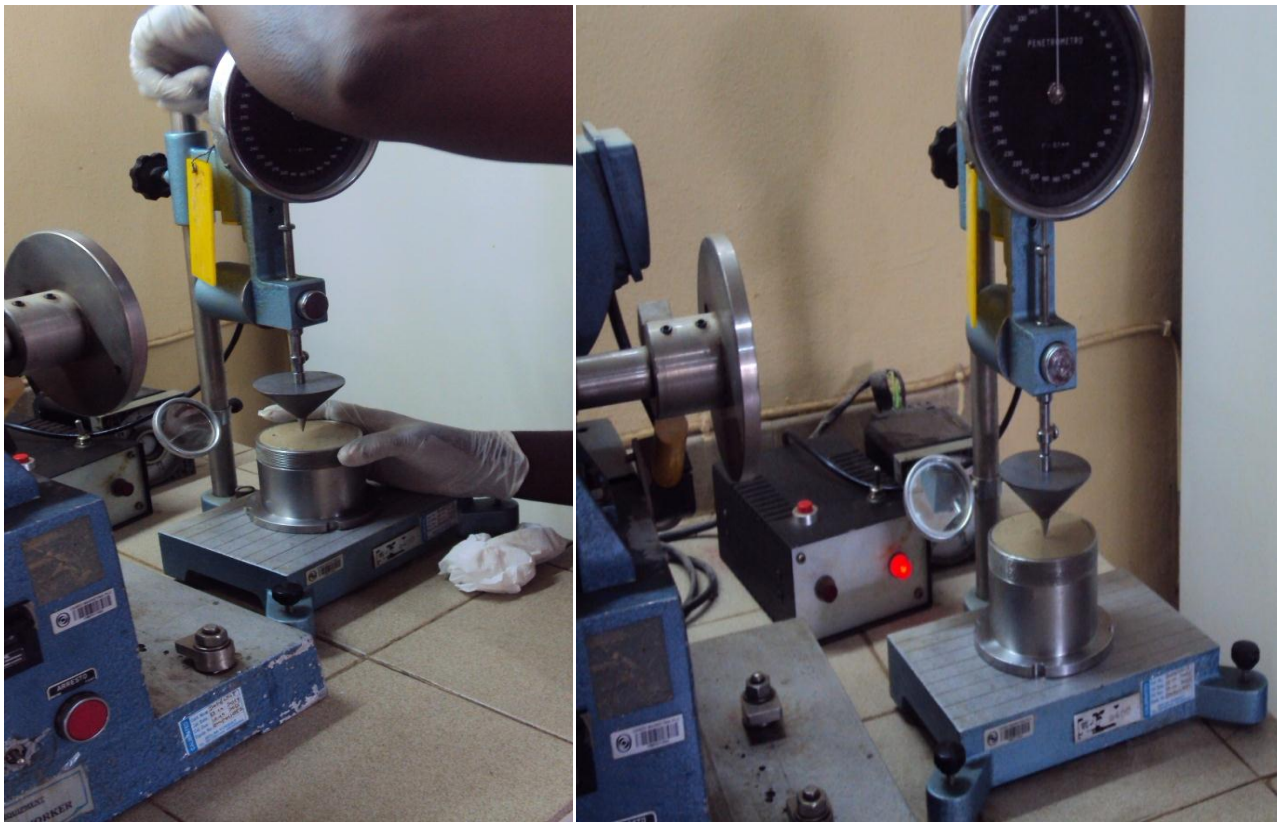


Plate 3.15 Penetrometer set with Biogrease sample

3.6.3 Procedure for Cone Penetration

For unworked penetration, the sample of the grease to be tested was collected and put into the grease cup. A spatula was used to remove the excess grease and to ensure that the grease surface was flat. The grease cup packed with the biogrease sample was placed under the cone assembly of the penetrometer with the cone set to just about touching the surface of the grease block. The cone was then released and allowed to drop freely into the grease for 5 seconds. Three determinations were made and the averaged result was to give the reported result (**ASTM D217**).

For worked penetration, the sample of the biogrease to be tested was collected and put into the grease cup and the grease worker was then tightened to the grease cup. The ….allowed the expulsion of excess grease from the grease worker. The grease worker was then placed and coupled to the Mechanical grease worker and the grease was worked for 60 double strokes. The worked biogrease sample was placed under the cone assembly of the penetrometer as in the case of unworked penetration and the cone was released and allowed to drop freely into the grease for 5 seconds. Three determinations were made and the averaged result was to give the reported result (**ASTM D217, 2002**).

3.7 Dropping Point Test

3.7.1 Materials

1. Biogrease Samples

3.7.2 Equipment and Materials

1. Mettler Dropping Point apparatus See Plate 3.18
2. Thermoneter



Plate 3.16 Mettler dropping Point apparatus

3.7.3 Procedure for Dropping Point

The biogrease sample was filled into the grease cup and prepared. The cup and thermometer arrangement was placed on the apparatus. The temperature at which the grease melted and a drop fell was noted. The procedure was repeated and the average reading noted (ASTM D566, 2002). This procedure was then repeated for each of the other samples.

3.8 Gear Wear Test

Gear wear test method was used to assess anti-wear characteristics in a pair of spur gears. In this test, a pair of gears was run at ambient temperature, for 10000 revolutions. The mass/weight loss was used to measure the rate of wear.

3.8.1 Materials

1. Biogrease Samples

2. Spur gears

3.8.2 Equipment

1. Gear wear test rig

2. Digital Scale

3. Flat Spanners



Plate 3.17 Sample spur gears used in the test



Plate 3.18 Test Rig

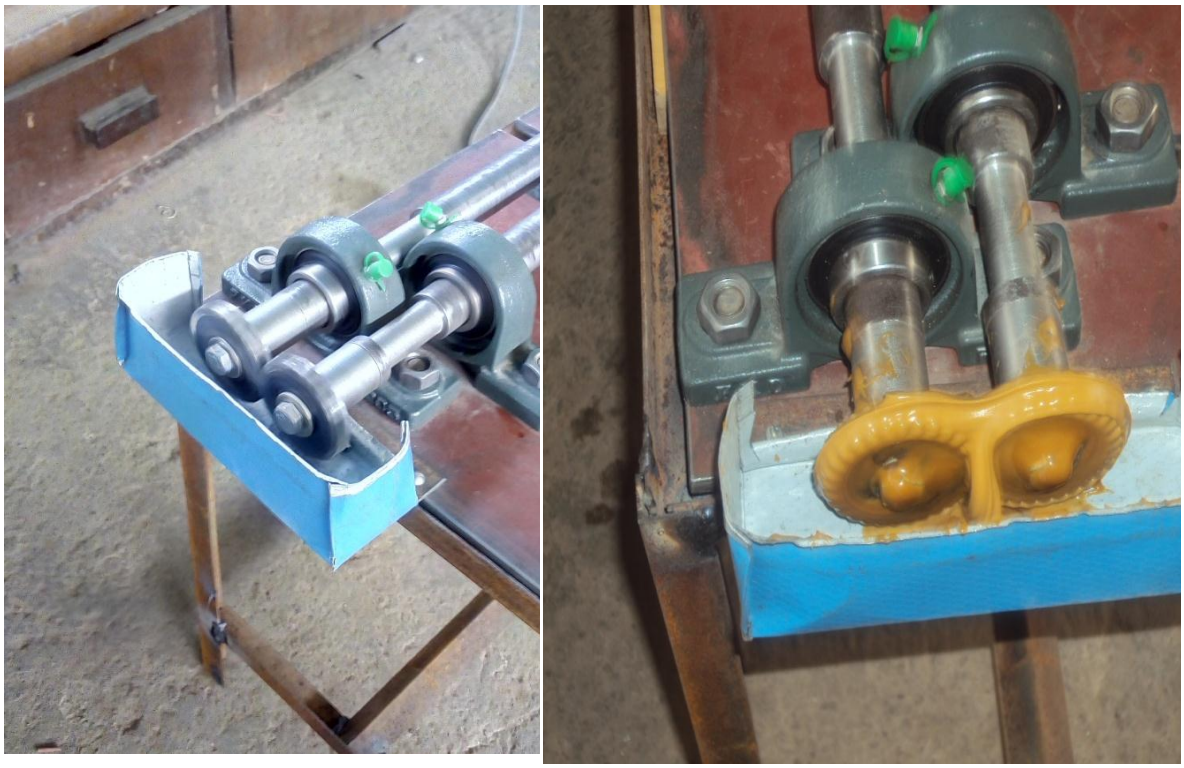


Plate 3.19 Test rig running in air and with sample C

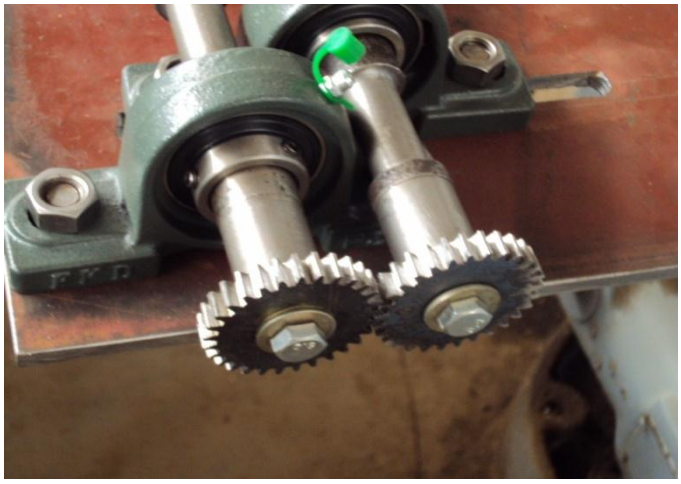


Plate 3.20 Close up of meshing gears

3.8.3 Procedure for Gear Wear Determination

A pair of calibration spur gears from a SATAM fuel dispensing pump was selected weighed, and these were then, fitted onto the two shafts of the machine. The machine was started and allowed to run for 10000 revolutions in air to establish a control value of wear. The gears were removed and weighed to obtain the mass/weight loss due to wear on the gears. The procedure was repeated but this time each pair of gears was dipped onto a sample of the grease and the machine allowed to run for 10000 revolutions. The gears were then removed, thoroughly cleaned and reweighed to obtain the mass loss due to wear.

The mass loss due to wear was calculated using the relation

$$Wg1i - Wg1f = A$$

Where

$Wg1i$ = initial weight of gear i

$Wg1f$ = final weight of gear i

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Viscosity Determination For Balanites aegyptiaca Seed Oil

The viscosity values obtained at different temperatures for desert date seed oil is as shown in Figure 4.1.

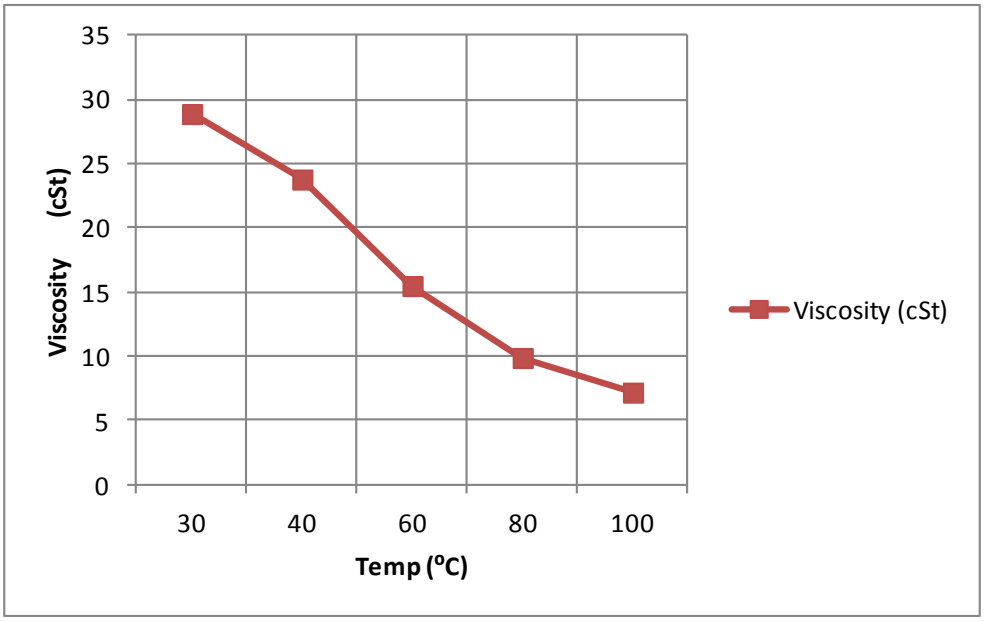


Figure 4.1 Variation of viscosity with temperature for *Balanites ae*. Seed oil.

The viscosity measurements for Desert date seed oil varied from 28.9 cSt at room temperature to about 7.2 cSt at 100°C. The graph shows a negative variation in viscosity that is a decrease in viscosity with increase in temperature. This is consistent with theory on physical properties of oils Totten *et al*, (2003)

4.2 Grease Formulation

Grease being a combination of base oil, thickener and additives ranging from about 70% Base oil to about 95% , and thickener from 0% to about 10% Yaakub,2008) grease formulation was done by varying only the base oil percentages and thickener starting from 70% base oil. The initial proposal was to have a range of samples of varying base oil/ thickener proportions starting from 70% through 95%. As shown in table 4.2.

Table 4.1 Proposed Biogrease Formulation Guide

S/No	Base Oil %	Thickener %	Total
1	70	30	100
2	75	25	100
3	80	20	100
4	85	15	100
5	90	10	100
6	95	5	100

However, upon commencement of grease production, at 85% base oil concentration, consistency of the oil did not change appreciably to qualify for being a grease. The mixture remained as an oil. Additional concentration of Base oil (90%) produced the same result. Therefore, further

increase in base oil concentration was abandoned. These resulted in having three samples with base oil concentrations of 70%, 75% and 80%.

A sample was also prepared base oil concentrations of 75% but with the additive Diphenylamine (DPA) 5%. As the minimum quantity of grease for required for the cone penetration tests was 450g, a target quantity of biogrease of 450g for each sample was therefore, proposed and the following quantities were used to obtain the samples (See breakdown in Table 4.3).

Table 4.2 Empirical Composition of Formulated Biogrease

Sample	Sample Weight =		450	g			Total	
	Base Oil (%)	Wt (g)	Thickener (%)	Wt (g)	Additive (%)	Wt (g)	%	Wt(g)
A	70	315.0	30	135.0	0	0	**100**	**450.0**
B	75	337.5	25	112.5	0	0	**100**	**450.0**
C	80	360.0	20	90.0	0	0	**100**	**450.0**
D	75	337.5	20	90.0	5	22.5	**100**	**450.0**

4.3 Consistency Tests

4.3.1 Worked and Unworked Penetration Tests

The results of these tests for the grease samples are as shown in Tables 4.4 and Table 4.5.

Table 4.3 Unworked penetration values for the biogrease samples.

S/No	SAMPLE	UNWORKED PENETRATION			AVERAGE
1	A	321	320	320	320
2	B	256	258	255	256
3	C	360	361	355	359
4	D	308	309	308	308
5	CONTROL	270	270	273	271

Table 4.4 Worked penetration values for the biogrease samples

S/No	SAMPLE	WORKED PENETRATION		AVERAGE
1	A	370	372	370
2	B	350	351	350
3	C	378	380	380
4	D	-	-	Indeterminate
5	CONTROL	280	278	279

.

The worked penetration values of the 3 biogrease samples A, B and C placed the greases under the NLGI classification of '0'. This is for greases with Worked penetration values of **W355 -385**. (Table 2.1). It was not possible to obtain the worked penetration values for the sample D because the Penetrometer could only read to a maximum value of 380, and the worked penetration values for that sample was beyond that. However even without the exact figures it could be placed at the NLGI classification of '**00**' that is **W400 – 430.** Our control sample was a NLGI 2.Also it could also be inferred that the presence of the antioxidant additive DPA might have influenced the penetration values hence the higher readings in the case of sample D.

4.3.2 Dropping Point Tests

The results for the dropping point tests are shown in Figure 4.2.

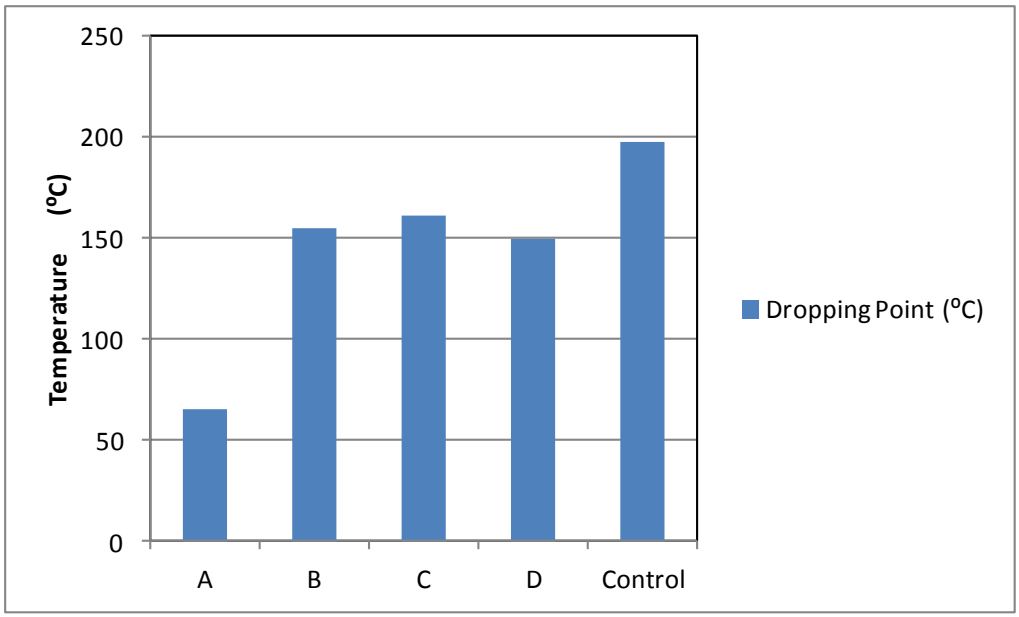


Figure 4.2 Dropping Point values for biogrease samples

The dropping point of the biogrease samples showed a variation with sample A having the highest concentration of thickener with the lowest value of dropping point of 65°C. There was a

rise in dropping point with corresponding decrease in thickener concentration with sample B having 75% thickener and a dropping point of 155°C. This trend continued with a dropping point of 161°C for a thickener concentration of 80%. The sample D also with a thickener concentration of 75% but with addition of the antioxidant DPA had a dropping point of 150°C. This could also be interpreted conversely showing an increase in dropping point with increase in base oil concentration.

The control sample had a dropping point of 198°C which was consistent with the grease data sheets showing a dropping point range of ≥190°C.

4.3.3 Gear Wear Test

The results of the gear wear test are shown in Figures 4.3, 4.4 and 4.5.

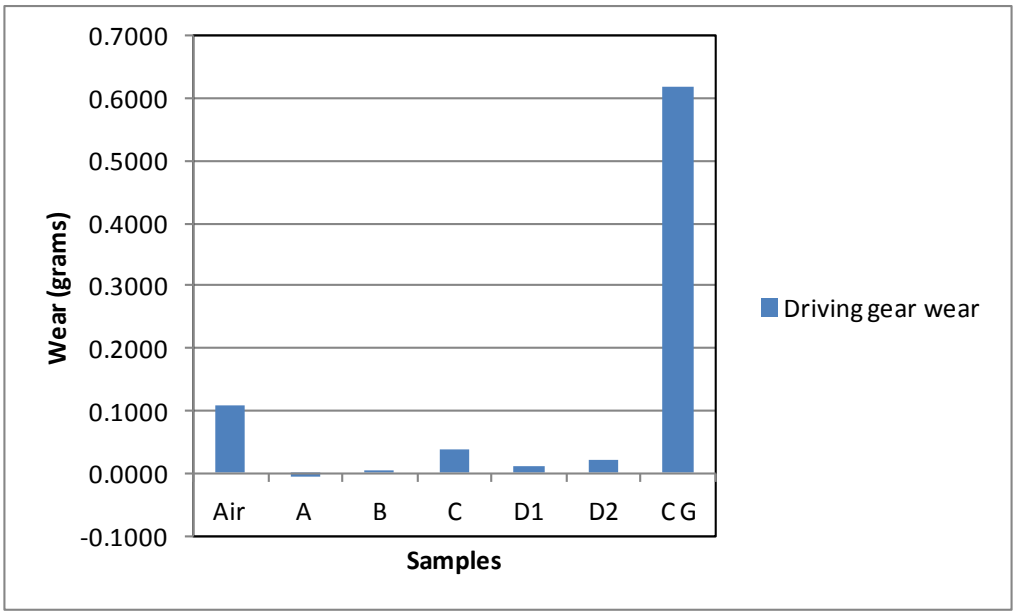


Figure 4.3 Wear values for the driving gear for each Biogrease sample

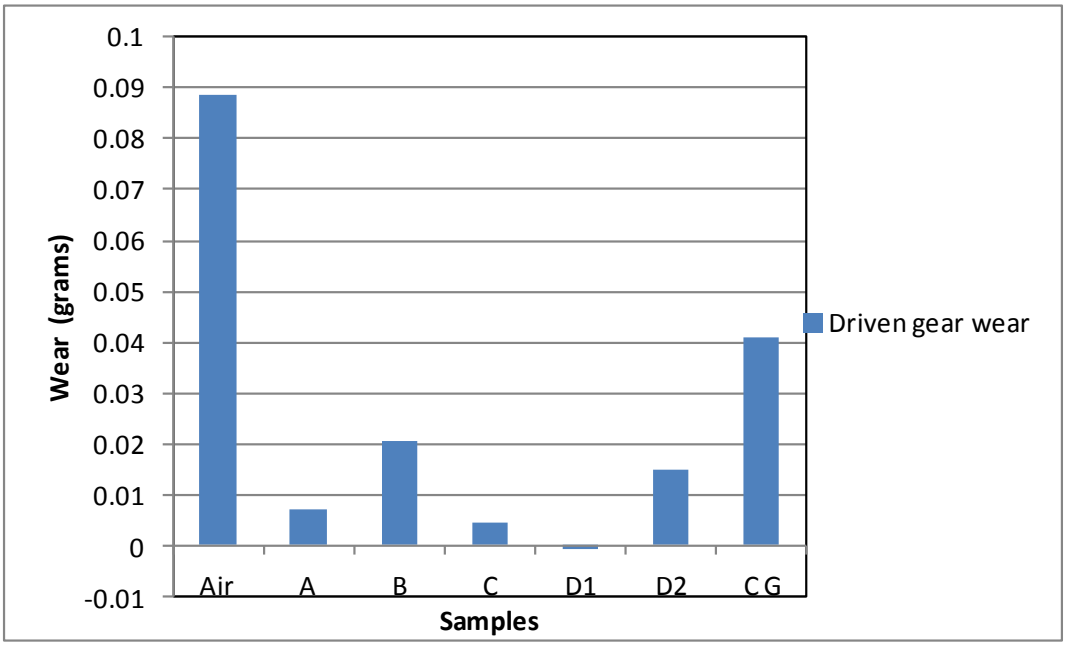


Figure 4.4 Wear values for the driven gear for each Biogrease sample

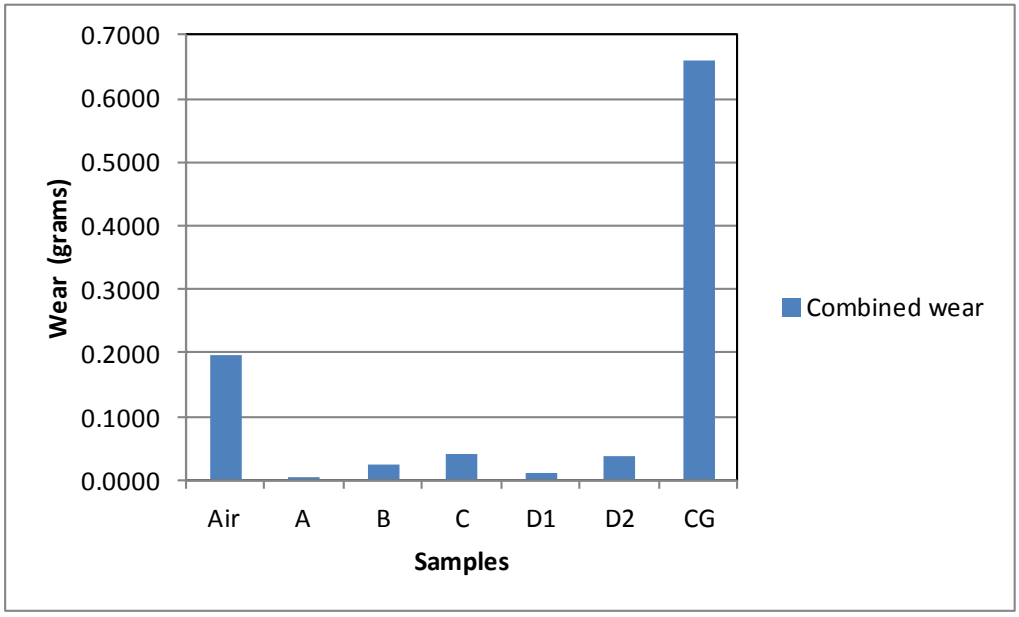


Figure 4.5 Combined gear wear values for Lithium Biogreases samples

For the mass loss due to wear test, a pair of gears chosen G0 and GX were run for 10000 revolutions in air to establish a control basis. It is expected that the maximum amount of wear would be obtained from these set of values. A value of 0.1090 g for the driving gear G0, and 0.0886 g for the driven gear GX was recorded. For the driving gears sample there was a sharp drop in the amount of wear for all the test samples of the greases, showing that the developed biogreases were all effective in preventing wear. However the amount of wear in the commercial grease sample was abnormally high. This represented a sharp deviation from the norm and could be attributed to perhaps some defect in the gear itself which made it softer that the others hence it exhibits a higher amount of wear. The sharp drop in wear as a result of the introduction of the biogrease samples was also observed for the driven gears. Here too, the amount of wear for the commercial grease sample was higher than for the samples lubricated by the biogreases. This could be attributed to the consistency values of the biogreases. They have worked penetration values placing them in the NLGI 0 classification making them more fluid than the commercial grease which is of classification NLGI 2. What it means is that the developed greases have good enough anti wear properties and are suitable for use at ambient temperature and light duty applications at low temperatures.

4.4 Costs

The summary of total costs for the production of the grease is shown in table 4.8. Based on these costs a final cost per unit for the end product was calculated.

Table 4.5 Summary of Costs for Lithium Biogrease from desert date seed oil

S/No	ITEM DESCRIPTION	QUANTITY USED	UNIT COST (N)	TOTAL COST (N)
1	Desert date	52 Kg	81.25	4,225.00
2	n-Hexane	5 litres	1,800.00	9,000.00
3	Lithium Hydrxide (LiOH)	46.6 g	35.00	1,631.00
4	Stearic Acid	551g	9.50	5,234.50
5	Labour (Nut Cracking)			800.00
6	Labour (Oil extraction)		5000	5,000.00
	Sub Total			20,890.50
	Processing Costs			
7	Plastic buckets	2	125	250.00
8	Plastic jars	6	30	180.00
9	Labour (Grease blending)		7000	7,000.00
	Testing			
10	Penetration Test	4 Samples	1000.	4,000.00
11	Dropping point	4 Samples	2000	8,000.00
	Total			**65,210.00**

From table 4.8, the total cost of desert Date seed oil based on raw material cost, solvent cost and labour paid for cracking **N19,025.00**.

Amount of oil realized (Kg) = 2.552

Therefore cost of oil per unit (Kg) = **N7,454.94**

Similarly, the total cost for the Biogrease samples = **N46,185.00**

Amount of grease produced (Kg) = 1.8

Average cost of Biogrease per unit (Kg) = **N36,227.77**

The costs are high due to a number of reasons.

1. The cost of solvent n-Hexane is quite high and has to be factored in even though it is reusable. Also the Soxhlet apparatus used was faulty and leakages affected the amount of solvent recovered.
2. Cost of laboratory scale chemicals like the LiOH and Stearic acid are also higher than industrial scale prices which also contributed to the high costs.
3. Testing cost per unit was also high since in commercial scale testing would be done on a batch basis and therefore the cost for a small test quantity would be spread over the entire bath thereby reducing he cost per unit of product.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

From the experimental results of this work, the following conclusions could be drawn

i. The Crude oil was extracted from the seed kernel of the desert date.

ii. Lithium Biogrease was developed from the extracted oil.

iii. Cone penetration, dropping point and gear wear tests were carried out on the developed grease.

iv. Comparative analysis of the developed grease with petroleum based Lithium grease (OANDO CROWN EP2 GREASE)was carried out.

v. Balanites aegyptiaca or desert date seed oil is a suitable feedstock for the production of biodegradable grease.

vi. Desert date seed oil combined with lithium stearate on a percentage weight basis ranging from 70% to 80%.

vii. Balanites aegyptiaca seed oil combined with lithium stearate yielded a grease of consistency NLGI '0'.

viii. Lithium Biogrease from Balanites aegyptiaca seed oil yielded greases with dropping point values ranging from 65^0C to 161^0C with increasing dropping point value as percentage concentration of base oil increases, peaking at 161^0C for 80% base oil.

ix. Lithium Biogrease from Balanites aegyptiaca seed oil showed good antiwear characteristics comparable or even superior to commercially available mineral grease.

5.2 Recommendations

The following recommendations are suggested.

i. A more detailed variation in the concentration of base oil and thickener for Balanites aegyptiaca needs to be carried out to obtain an optimum value.

ii. The combination of Balanites aegyptiaca seed oil with other metal soap thickeners with need to be undertaken to establish the type and characteristics of biogrease so obtained.

iii. Effect of additives on the properties, characteristics and biodegradability of the biogrease needs to be further studied in order to obtain improved and enhanced biogrease formulations.

iv. Test equipment used in Tribology research should be provided in our higher institutions to facilitate and enhance research in this area.

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