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# Lubricating Greases Based on Fatty By-Products and Jojoba Constituents

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## 1. Introduction

There has been a need since ancient times for lubricating greases. The Egyptians used mutton fat and beef tallow to reduce axle friction in chariots as far back as 1400 BC. More complex lubrications were tried on ancient axle hubs by mixing animal fat and lime, but these crude lubricants were in no way equivalent to the lubricating greases of modern times. Good lubricating greases were not available until the development of petroleum based oils in the late 1800's. Today, there are many different types of lubricating greases, but the basic structure of these greases is similar.

In modern industrial years, greases have been increasingly employed to cope with a variety of difficult lubrication problems, particularly those where the liquid lubricant is not feasible. Over the last several decades, greases making technology throughout the world, has undergone rapid change to meet the growing demands of the sophisticated industrial environment. With automation and mechanization of industry, modern greases, like all other lubricants, are designed to last longer, work better under extreme condition and generally expected to provide adequate protection against rust, water, and dust. So, greases are the important items for maintenance and smooth running of various machineries, automobiles, industrial equipments, instruments and other mechanical parts. Industrial development and advances in the field of greases have been geared to satisfy all these diverse expectations (Cann, 1997).

In general, lubricating greases contain a variety of chemical substances ranging from complicated mixtures of natural hydrocarbons in the base oils, well defined soaps and complex organic molecules as additives. Therefore, the more practical greases are lubricating oils which has been thickened in order to remain in contact with the moving surfaces, do not leak out under gravity or centrifugal action or be squeezed out under pressure. The majority of greases in the market are composed of mineral oil blended with soap thickeners. Additives enhance the performance and protect the greases and/or lubricated surfaces. Lubricating greases are used to meet various requirements in machine elements and components, including: valves, seals, gears, threaded connections, plain bearings, chains, contacts, ropes, rolling bearing and shaft/hub connections (Boner, 1954, 1976).

Developments in thickeners have been fundamental to the advances in grease technology. The contribution of thickeners has been so central to developments that many types of greases are often classified by the type of thickener used to give the required structured matrix and consistency. The two principal groups of thickeners are metal soaps and inorganic compounds. Soap-based greases are by far the most widespread lubricants.

In soap greases the metallic soap consists of a long-chain fatty acid neutralized by a metal such as lithium, sodium, calcium, aluminum, barium or strontium. A wide variety of fatty materials are used in the manufacture of base lubricating greases. In particular, lithium lubricating greases, first appeared during World War II, were made from lithium stearate pre-formed soap. Nowadays they are usually prepared by reacting lithium hydroxide, as a powder or dissolved in water, with 12-hydroxy stearic acid or its glycerides in mineral oils or synthetic oils. Whether the free acid or its glycerides is preferred depends on the relationship between cost and performance (Kinnear & Kranz, 1998; El-Adly, 2004a).

A comprehensive study of all aspects of grease technology with the corresponding literature references is beyond the scope of this short contribution. There are numerous textbooks available on this subject (Vinogradov, 1989; Klamann, 1984; Boner, 1976; Erlich, 1984; Lansdown, 1982).

Within the area of alternate sources of lubricants (El-Adly et al, 1999, 2004a, 2004b, 2005, 2009), a new frontier remains for researchers in the field of lubricating greases. Lithium greases have good multi-purpose properties, e.g. high dropping point, good water resistance and good shear stability. Alternative sources of fatty materials and additives involved in the preparation of such lithium greases will be found later in this chapter. The main objective is to explore the preparation, evaluation and development of lithium lubricating greases from low cost starting materials such as, bone fat, cottonseed soapstock and jojoba meal. The role of the jojoba oil and its meal as novel additives for such greases is also explored (El-Adly et al., 2004b).

## 2. Raw materials

The main components of lubricating greases, in general, are lubricating mineral oil, soaps and additives. The mineral oil consists of varying proportions of paraffinic, naphthenic and aromatic hydrocarbons, in addition to minor concentrations of non-hydrocarbon compounds. Soaps may be derived from animal or vegetable fats or fatty acids. Additives are added to lubricating greases, generally in small concentrations, to improve or enhance the desirable properties of the finished product. The use of these ingredients such as fats, fluids and additives, each of which consists of a number of chemical compounds, was originally dictated to a large extent by economic factors and availability. The raw materials mentioned in this chapter are, therefore, according to the following:

### 2.1 Lubricating fluid

Mineral oils are most often used as the base stock in grease formulation. About 99% of greases are made with mineral oils. Naphthenic oils are the most popular despite of their low viscosity index. They maintain the liquid phase at low temperatures and easily combine with soaps. Paraffinic oils are poorer solvents for many of the additives used in greases, and with some soaps they may generate a weaker gel structure. On the other hand, they are

more stable than naphthenic oils, hence are less likely to react chemically during grease formulation.

Characteristics	Base oil (B1)	Bright stock (B2)	Test Methods
Density, g/ml: at 15.56, °C	0.872	0.8975	ASTM D.1298
Refractive index, n <sub>D</sub> <sup>20</sup>	1.5723	1.5988	ASTM D.1218
ASTM-Color	1.0	1.0	ASTM D.1500
Kinematics viscosity, c St.			
at 40°C	50	78	
at 100°C	9	19	ASTM D.445
Viscosity index	233	225	ASTM D. 189
Dynamic Viscosity, @ 30 °C (20 rpm), cP	2100	2905	ASTM D. 189
Pour point, °C	-3	Zero	ASTM D.97
Total acid number, mg KOH/ g@72 hr	0.12	0.2	ASTM D.664
Flash Point, °C	210	290	ASTM D.92
Molecular Weight	755	890	GPC*
Predominant, molecular weight	762	898	GPC*
Polydispersity	1.1023	1.253	GPC*
Structural group analysis hydrocarbon component, wt %			ASTM D-3238
%C <sub>A</sub> (Aromatic Percentage)	19	20	
%C <sub>P</sub> (Paraffinic Percentage)	61	68	
%C <sub>N</sub> (Naphthinic Percentage)	20	12	
Mono-aromatic	14.9	13.2	Column chromatography
Di-aromatic	12.0	15.5	
Poly-aromatic	1.2	1.5	

GPC\* Gel Permeation Chromatography

Table 1. Physico-chemical properties of the lubricating fluids (Base oil B1&bright stock B2)

In this respect, two types of lube base oils are investigated as fluids part for preparing lithium lubricating greases: the first is a base mineral oil designated B1 and the second is a bright stock designated B2. The Physico-chemical properties of these oils were carried out using ASTM/ IP standard methods of analysis as shown in Table (1). Data in this table reveal that the bright stock could be classified as heavier oil than lube base oil. It may be pointed out, therefore, that the internal friction between oil layers in B2 is greater than in B1. This interpretation agrees with the data of gel permeation chromatography concerning molecular weights of B1 and B2. This is further supported by predominant molecular weights of B1 and B2 which are 762 and 898, respectively. In addition, the polydispersity (i.e., number of average molecular weight divided by mean molecular weight value,  $M_n/M_w$ ) for bright stock is 1.2530 while it is 1.1023 for base mineral oil. This indicates that B1 and B2 have higher degree of similarity in hydrocarbon constituents (cross sectional areas of molecules are similar) and morphology of structure.

The rheological properties of the above mentioned oils were studied at different temperatures using Brookfield programmable Rheometer LV DV-III ULTRA. Different mathematical model (Herschel Bulkley, Bingham and Casson models) were applied to deduce the viscoelastic parameters. It was found that the fluids under investigation had a Newtonian behavior (El-Adly, 2009).

## **2.2 Fatty material**

### **2.2.1 Cottonseed soapstock**

Soapstock is formed by reacting crude vegetable oils with alkali to produce sodium soap as a by-product, which is separated from the oil by centrifuging. Typically, soapstock accounts for 5 to 10 wt. % of the crude oil. In general, soapstock from oilseed refining has been a source of fatty acids and glycerol. These processes are no longer cost effective. Consequently, in cottonseed oil extraction facilities, the treated soapstock is added to the animal meal to increase the energy content, reduce dust and improve pelleting of food products (Michael, 1996). In general compositional information considering raw and acidulated cottonseed soapstock has been published (El-Shattory, 1979; Cherry & Berardi, 1983).

### **2.2.2 Bone fat**

The crude bone fat is produced by solvent extraction of crushed bone during the manufacture of animal charcoal. It is considered as by-product for this process. Also, it is extracted by wet rendering under atmospheric pressure from femur epiphyses of cattle, buffaloes and camels. The physical and chemical properties of the above mentioned bone fat was studied (El-Adly, 1999). It has low cost and possesses large-scale availability.

### **2.2.3 Physicochemical properties of the bone fat and soapstock**

Data in Table (2) show the physicochemical properties of the bone fat and cottonseed soapstock carried out using ASTM/ IP standard methods of analysis. Bone fat and cottonseed soapstock consist primarily of glycerides, that is, of various fatty acid radicals combined with glycerol. It is apparent from Table (2) that the saponification number for bone fat and soapstock are 180.0 and 198.0, respectively. These values were not only used as basis for figuring the amount of alkali required for a particular formulation, but also permitted speculation as to the identity of the fatty acids making up the fatty materials.

The results of gas liquid chromatography analysis of the esterified fatty acids in bone fat and hydrolyzed cottonseed soapstock are shown in Table (2). There is a wide variation in their fatty acids composition myristic, palmitic, stearic, oleic, linoleic and linolenic acid. Bone fat is composed of about 52% unsaturated fatty acids, mainly oleic acid, and 47% saturated fatty acids, being palmitic, stearic and myristic acid. However, soapstock contains more unsaturated fatty acids 71% and saturated 29%. This finding was supported by the iodine value measured for both fatty materials. The difference in their fatty constituents leads to the possibility of producing lithium lubricating grease.

Property (in mole %)	Bone Fat	Soapstock	Test method
Saponification number	180	198	ASTM D-1962
Iodine value	45	60.0	ASTM D-2075
Titer, C°	35	45.0	ASTM D-1982
Palmitic acid	23.0	27.0	Gas chromatography
Myristic acid	9.0	trace	
Oleic acid	48.0	29.0	
Stearic acid	15.0	2.0	
Linoleic acid	4.0	42.0	
Linolenic acid	trace	trace	

Table 2. Physicochemical properties of bone fat and cottonseed soapstock

2.3 Additives

The additives used in grease formulation are similar to those used in lubricating oils. Some of them modify the soaps, others improve the oil characteristics. The most common additives include anti-oxidants, rust and corrosion inhibitors, tackiness, and anti-wear and extreme pressure additives. Many studies reported detailed information about lubricating additives (Mang & Dresel, 2001; Shirahama, 1985). This chapter presents the utilization of jojoba oil and its meal as additives for the preparation of lithium lubricating greases.

2.3.1 Jojoba oil

Jojoba is known in botanical literatures as *Simmondsia chinensis* (Link) of the family Buxaceaa and as *Simmondsia californica* Nutall. The first name is the correct one, although it perpetuates a geographical misnomer. In late 1970 sperm whale was included by the US Government in the list of endangered species and imports of oil, meal and other products derived from whales were banned. At that time, sperm oil consumption in the United States was about 40-50 million pounds per year, with half that figure used in lubricant applications. No single natural, or synthetic replacement with the unique qualities of sperm whale oil has yet been found, but enough experimental evidence has accumulated in the last years that jojoba oil is not only an excellent substitute of sperm oil but its potential industrial uses go beyond those of sperm oil (Wisniak, 1994). Sperm oil is widely used in lubricants because of the oiliness and metallic wetting properties, it imparts and its nondrying characteristics that prevent gumming and tackiness in end-use formulations. It is more important as a chemical intermediate since it is sulphonated, oxidized, sulfurized, sulfur-chlorinated and chlorinated to give industrial products that were used primarily as wetting



agents and extreme pressure (EP) additives. The composition and physical properties of Jojoba are close enough to sperm oil to suggest the use of Jojoba oil as a substitute for most of the uses of sperm oil (Miwa & Rothfus, 1978). Sperm oil has been used as an extreme pressure and antiwear additive in lubricants for gears in differentials and transmissions, in hydraulic fluids that need a low coefficient of friction and in cutting and drawing oils. In some of these, sperm oil has been directly, but it is usually Sulfurized (sometimes epoxidized, chlorinated, or fluorinated). Gear lubricants (e.g., in automobile transmissions) commonly contain 5 to 25 percent of Sulfurized sperm oil (Peeler & Hartman, 1972).

Some of the first published results of sulfurized jojoba oil use a lubricant and extreme pressure (EP) additive were reported as patents (Flaxman, 1940; Wells, 1948). Wells pointed out several advantages of jojoba oil over sperm oil. Its slight odor is distinctly more pleasant than the fishy odor of sperm oil. Crude jojoba oil contains no glycerides so that the crude oil needs little or no treatment to prepare it for most industrial purposes.

In general, lubricant technology dealing with jojoba oil and its derivatives in the 70's concentrated on its replacement of sulfurized sperm oil products in such applications as industrial and automotive gear oils, hydraulic oils and metal working lubricants (Heilweil, 1988; Wills, 1985). In the 80's the lubrication industry has developed and research on jojoba has been shifting towards new derivatives with potential application to new technologies and newer areas of lubricant use. A monograph by Wisniak (1987) summarized the chemistry and technology of jojoba oil and jojoba meal.

### 2.3.1.1 Composition

The chemical composition of jojoba oil is unique in that it contains little or no glycerin and that most of its components fall in the chain-length range of C<sub>36</sub>-C<sub>42</sub>. Linearity and close-range composition are probably the two outstanding properties that give jojoba oil its unique characteristics. The oil is characterized of being a monoester of high molecular weight and straight chain fatty acids and fatty alcohols that has a double bond on each side of the ester. The molecular structure of the oil can be represented by the following general formula:



Where, m and n are between 8 to 12 (Miwa 1971, 1980; Spencer et al, 1977; Greene & Foster, 1933).

They were the first to report that jojoba nuts contain about 46-50% of liquid oil which resembles sperm whale oil in its analytical characteristics. Qualitative tests suggested that the oil might consist mainly of fatty acid esters of decyl alcohol. Shortly thereafter, detailed analysis of the chemical constituents was reported (Greene & Foster, 1933). The main components were eicosenoic and docosenoic acids and eicosanol and docosanol. Because of the problems of the high resistance of the oil to saponification, the difficulties in isolating pure fractions and the lack of convenient and reliable quantitative analytical techniques the characterization of jojoba oil was developed by Miwa (1971 & 1980).

Jojoba oil is unusually stable towards oxidation especially at high temperature. Kono et al, (1981) mentioned that the oxidative stability of jojoba oil was due, at least in part, to the presence of tocopherol and other natural antioxidants. Also, some of the antioxidants separated and identified by molecular distillation of the oil and analysis of the distillate by gas chromatography/ mass spectrometry. The,  $\alpha$ ,  $\gamma$  and  $\delta$  isomers of tocopherol are present, in varying quantities depending on the origin of the oil,  $\gamma$ -isomer being most abundant.

2.3.1.2 Physical properties

Jojoba oil is chemically purer than most natural substances. It is soluble in common organic solvents such as benzene, petroleum ether, chloroform, carbon tetrachloride, and carbon disulfide, but it is immiscible with ethanol, methanol, acetic acid, and acetone (Miwa & Hagemann, 1978). It is usually a low-acidity, light-golden fluid that requires little or no refining. It is non-volatile and free from rancidity. Even after repeated heating to temperatures above 285°C for 4 days it is essentially unchanged (Daugherty et al., 1953). Its boiling point (at a pressure of 757 mmHg, under nitrogen) rises to 418 °C but drops rapidly to a steady 398 °C (Miwa 1973; Wisniak, 1987). Neutralization of the oil is not usually required and bleaching to a water-clear fluid can be done with common commercial techniques. Some properties of the oil are listed in Table 3 (El-Adly et al, 2009). Data in Table (3) reveal that the possibilities for economic development of the oil and its suitability to produce lubricants and lubricant additives for use in the preparation of lubricating greases. This view is in agreement with a study on using of jojoba oil as oxidation, thermal and mechanical stabilities to improve the properties of lithium lubricating grease (Ismail, 2008).

Characteristics	Jojoba oil	Test Method
Density, g/ml @ 25/25, °C	0.863	ASTM D-1298
Refractive index, n <sub>D</sub> <sup>20</sup>	1.4652	ASTM D-1218
Kinematics viscosity, c St.		
at 40°C	26	ASTM D-445
at 100°C	7.5	ASTM D-445
Viscosity index	257	ASTM D- 189
Dynamic Viscosity, @ 30 °C (rpm 6), cP	58.4	ASTM D-97
TAN, mg KOH/g	2.0	ASTM D-664
Flash Point, °C	310	ASTM D-92
Iodine Value	80	ASTM D-2075
Average Molecular Weight	604	GPC
Surface tensions mN/m	24	
Oxidation stability test (min)	23	IP 229

Table 3. Physico-chemical properties of Jojoba oil (El-Adly et al, 2009)

2.3.2 Jojoba meal

A byproduct of jojoba seeds is the meal remaining after the oil has been pressed and extracted. This material constitutes about 50% of the seed and contains 25-30 % crude protein. Table (4), presents the amino acid composition (%by wieght) of deoiled meal of two varieties of jojoba meal (Verbiscar &Banigan, 1978). Basic information on the composition of jojoba meal, polyphenolic compounds, carbohydrate contents, and Simmondsin compounds have been reported (Verbiscar et al., 1978; Cardeso et al., 1980; Wisniak, 1994). On the other hand, the possibility of using the meal as fuel has already been considered (Kuester, 1984 & Kuester et al., 1985). El-Adly et.al (2004b) reported the novel application of jojoba meal as additive for sodium lubricating grease.



Amino acid	Apache 377	SCJP 977
Lysine	1.05	1.11
Histidine	0.486	0.493
Arginine	1.56	1.81
Aspartic acid	2.18	3.11
Threonine	1.14	1.22
Serine	1.04	1.11
Glutamic acid	2.40	2.79
Proline	0.958	1.1.
Glycine	1.50	1.41
Alanine	0.832	0.953
Valine	1.10	1.19
Methionine	0.186	0.210
Isoleucine	0.777	0.866
Leucine	1.46	1.57
Tyrosine	1.04	1.05
Phenylalanine	0.919	1.07
Cystine+ cystine	0.791	0.519
Tryptophan	0.492	0.559

Table 4. Amino acid composition (%) of deoiled meal of two varieties of jojoba meal (Verbiscar and Banigan, 1978)

Table (5) presents the anion and cation concentrations in jojoba meal determined through the sulphuric acid wet ashing. The anion concentrations are measured using ion chromatography (IC) model DIONEX LC20 equipped with electrochemical detector model DIONEX ED50, while the cation concentrations determined by inductively coupled plasma/atomic emission (ICP/AE) spectrometer model flame Modula spectra.

Cations	Concentration ppm	Anions	Concentration, ppm
Calcium	1178	Phosphate	12718
Lithium	1.73	Chloride	1286
Potassium	7304	Sulphate	8600
Sodium	566	fluoride	135
Magnesium	2079		
Alumminum	33.4		
Iron	124		
Copper	13.9		
Manganese	20.1		
Barium	1.51		
Zinc	29.8		
Cobalt	3.56		
Nickel	0.34		
Strontium	3.99		

Table 5. Anions and cations contents of the jojoba meal

Table (5) also reveals that the main anions in jojoba meal are phosphate (12718 ppm) and chloride (1286 ppm) but the main cations are magnesium, calcium, potassium and sodium. This indicates the possibility of using and optimizing the organometallic compounds in jojoba meal as additives for the lubricating greases.

### 3. Grease preparation and evaluation

#### 3.1 Lithium greases preparation

Lithium base lubricating greases can be prepared either by batch or continuous processes. Such products can be manufactured from either preformed soap or soap prepared *in situ*. From the standpoint of economy and versatility, the latter method is preferable and is therefore used by most manufacturers. The exception to this last statement is in the case of synthetic lubricating fluids. Preformed soaps are desirable in such case because some of these fluids such as diesters will hydrolyze in the presence of alkalies and heat (Boner, 1954, 1976). The lithium lubricating greases mentioned in this chapter were prepared using batch processing. The studied greases were prepared in two steps according to the following:

- a. Saponification process was performed on a mixture of fatty materials and fluids by alkaline slurry within the temperature range 190 to 195°C. The autoclave was charged, while stirring, with a mixture of 25% wt of light mineral oil and 14% wt of fatty materials (bone fat and soapstock). The autoclave was closed and heating started. Then about 3% wt lithium hydroxide/oil slurry is gradually pumped into the autoclave. The temperature of the reaction mixture must be raised to 190-195°C and held at this temperature for approximately 60 min. to ensure complete saponification. After completion of the saponification step, jojoba oil and or jojoba meal in different concentrations was added. A sample was then taken to examine its alkalinity/acidity. Corrections were made by adding fatty materials or Lithium hydroxide oil slurry as required reaching a neutral product i.e. complete saponification
- b. Cooling process was performed after the completion of the saponification reaction. The reaction mixture was cooled gradually while adding the rest of the base lube oil to attain the required grease consistency.

The obtained greases were tested and classified according to the standards methods, National Lubricating Greases Institute (NLGI) and the Egyptian Standards (ES). Also, the physico-chemical characteristics of all the prepared greases under investigation were determined using standard methods of analysis. These include penetration, dropping point, apparent viscosity, oxidation stability, total acid number, oil separation and four balls. In general, test methods are used to judge the single or combined and more or less complex properties of the greases. The last summary containing detailed descriptions of ASTM and DIN methods was reported (Schultze, 1962); but the elemental analysis of the greases is nowadays performed by spectroscopic methods, e.g. X-ray fluorescence spectrometry, inductively coupled plasma atomic emission, or atomic absorption spectrometry, with attention being directed mostly to methods of preparation (Robison et al 1993; Kieke, 1998). Also, Thermogravimetry and differential scanning calorimetry tools are used to evaluate of base oil, grease and antioxidants (Pohlen, 1998; Gatto & Grina, 1999).

#### 3.2 Effect of the fatty materials and fluid part concentrations on the prepared greases

The physical and chemical behaviors of greases are largely controlled by the consistency or hardness. The consistency of grease is its resistance to deformation by an applied force.

Ingredient	Symbol								Test method
		G <sub>1A</sub>	G <sub>1B</sub>	G <sub>1C</sub>	G <sub>1D</sub>	G <sub>1E</sub>	G <sub>1F</sub>	G <sub>1G</sub>	
Base oil, Wt %		79.0	79.0	80.0	-	-	-	30	
Brightstock, Wt %		-	-	-	80.0	80.0	80.0	50	
Soap stock, Wt %		18	-	8.5	17.0	-	8.5	8.5	
Bone fat, Wt %		-	18.0	8.5	-	17.0	8.5	8.5	
LiOH, Wt %		3.0	3.0	3.0	3.0	2.8-3	2.8-3	2.8-3	
Penetration									ASTM D-
	Unworked	300	300	300	290	290	290	285	217
	worked	310	310	310	300	300	300	290	
Dropping point, °C		170	173	174	174	175	177	178	ASTM D-566
Copper Corrosion 3h/100°C		Ia	Ia	Ia	Ia	Ia	Ia	Ia	ASTM D-4048
Oxidation Stability 99± 96h, pressure drop, psi		4.2	4.1	4.5	4.0	4.0	4.1	4.0	ASTM D-942
Alkalinity, Wt%		0.3	0.4	0.4	0.5	0.5	0.5	0.5	ASTM D-664
TAN, mg KOH/gm @ 72h		0.34	0.34	0.33	0.33	0.32	0.30	0.28	ASTM D-664
Oil Separation, Wt%		2.5	2.5	2.3	2.3	2.2	2.2	2	ASTM D-1724
Code grease NLGI Egyptian standard		2 LB	2 LB	2 LB	2 LB	2 LB	2 LB	2 LB	
Apparent Viscosity, cP, @ 90 °C		39600	39650	39680	39700	39710	39750	39891	ASTM D-189
Yield stress, D/cm²		60.2	61.3	62.1	62.9	63.6	64.3	65.0	
Four ball weld load, Kg		160	162	165	166	168	169	170	ASTM D-2596

Table 6. Effect of the fatty material and fluid concentrations on characterization of prepared greases

Also, it is defined in terms of grease penetration depth by a standard cone under prescribed conditions of time and temperature (ASTM D-217, ASTM D-1403). In order to standardize grease hardness measurements, the National Lubricating Grease Institute (NLGI) has separated grease into nine classification, ranging from the softest, NLGI 000, to the hardest, NLGI 6. On the other hand, the drop point is the temperature at which grease shows a change from a semi-solid to a liquid state under the prescribed conditions. The drop point is the maximum useful operating temperature of the grease. It can be determined in an apparatus in which the sample of grease is heated until a drop of liquid is formed and detaches from the grease (ASTM D-266, ASTM D-2265).

In order to evaluate the effect of fatty materials type and fluid on the prepared lithium grease properties, grease blends  $G_{1A}$ ,  $G_{1B}$ ,  $G_{1C}$ ,  $G_{1D}$ ,  $G_{1E}$ ,  $G_{1F}$  and  $G_{1G}$  have been prepared and formulated according to the percent ingredient listed in Table (6).

Data in Table (6) indicate the effect of different ratios from soapstock, bone fat, base oil and bright stock on the properties of the prepared lithium lubricating greases. It is evident from these results that the dropping point of lithium grease blend made from bone fat or soapstock alone is lower than that of lithium grease containing a mix from each both fatty materials and fluids. This clearly indicates that the most powerful thickener in the saponification process is the equimolar ratio from bone fat and soapstock. In other words, both fatty materials have synergistic effect during the saponification reaction. The mechanical efficiency of the formulated greases is according to the following order  $G_{1G} > G_{1F} > G_{1E} > G_{1D} > G_{1C} > G_{1B} > G_{1A}$ . On the other hand, the above mentioned test showed that the difference of penetration values between unworked and worked (60 strokes) greases follows an opposite order. Based on this finding, it is concluded that the most efficient lube oil in saponification is the light base oil (B1). This is attributed to the fact that lighter oil B1 is easily dispersed in fatty materials during saponification step at temperature 190°C and form stable soap texture. After completion of saponification, the bright stock (B2) is suitable in the cooling step which leads to heavier consistency and provides varying resistance to deformation. This reflects the role of the effect of mineral oil viscosity and fatty materials on the properties of the prepared grease.

It is apparent from the data in Table (6) that the oil separation, oxidation stability, total acid number and mechanical stability for the prepared grease  $G_{1G}$  are 2.0, 3.0, 0.68 and 5.0 respectively. This indicates that the best formula is  $G_{1G}$  compared with  $G_{1A}$ ,  $G_{1B}$ ,  $G_{1C}$ ,  $G_{1D}$ ,  $G_{1E}$ , and  $G_{1F}$ . Based on the above mention results and correlating these results with the apparent viscosity dropping point and penetration, clearly indicates that the suitable and selected formula for the lithium lubricating grease is  $G_{1G}$ .

### 3.3 Effect of the jojoba oil additive on properties of the selected prepared grease

To evaluate the role of jojoba oil as additive for the Selected Prepared Grease  $G_{1G}$ , different concentrations from jojoba oil were tested. In this respect, three concentrations of jojoba oil of 1wt%, 3 wt% and 5wt% were added to the selected grease  $G_{1G}$  yielding  $G_{2A}$ ,  $G_{2B}$  and  $G_{2C}$ , respectively, as shown in Table (6). Worth mentioning here, Jojoba oil ratio was added to the prepared greases after the completion of saponification process. Data in Table (7) show that the results of the penetration and dropping point tests for lithium grease prepared  $G_{2A}$ ,  $G_{2B}$  and  $G_{2C}$  produced from different ratio of jojoba oil. These results show that the difference of penetration values between unworked and worked (60 double strokes) lithium lubricating greases are in the order  $G_{2C} < G_{2B} < G_{2A}$ . This means that the resistance to texture deformation

decreases with increase of jojoba oil ratio in the prepared grease. It may be indicated also that on increasing the ratio jojoba oil additive to the prepared greases would increase binding and compatibility of the grease ingredient. As a result, the dropping point values for prepared greases  $G_{2A}$ ,  $G_{2B}$  and  $G_{2C}$  increased to 178, 180 and 183°C, respectively. Table (7) shows, in general, the positive effect of all concentrations of jojoba oil additive on the proprieties of  $G_{2A}$ ,  $G_{2B}$  and  $G_{2C}$ . In this respect, the 5%wt of additive of jojoba oil showed a marked improvements effect. Such improvements may be attributed to the unique properties of jojoba oil, e.g. high viscosity index 257, surface tension 45 mN/m and its chemical structure (Wisniak, 1987). Based on these properties and correlation with the dropping point, penetration, oil separation, oxidation stability, dynamic viscosity, consistency index and yield stress data, its clear that the suitable and selective grease formula is  $G_{2C}$ .

Ingredient & property	Symbol			Test method
	$G_{2A}$	$G_{2B}$	$G_{2C}$	
$G_{1g}$ , wt%	99	97	95	
Jojoba oil, wt%	1	3	5	
Penetration at 25°C				
Un worked	284	278	277	
worked	289	282	280	ASTM D-217
Dropping point, °C	180	182	187	ASTM D-566
Oxidation Stability 99±96h,				
pressure, drop, psi	3.5	3.2	3.0	ASTM D-942
Alkalinity, Wt%	0.16	0.14	0.14	ASTM D-664
Total acid number, mg				
KOH/g, @72h	0.20	0.18	0.16	ASTM D-664
Oil separation, Wt%	1.8	1.8	1.7	ASTM D-1724
Copper Corrosion				
3h/100°C	Ia	Ia	Ia	ASTM D-4048
Code Grease				
NLGI	2	2	2	
Egyptian Standard	LB	LB	LB	
Apparent Viscosity, cP, @				
90 °C	39891	41090	41294	ASTM D-189
Yield stress, D/cm <sup>2</sup>	75.6	78.1	80.6	
Four ball weld load, Kg	188	190	195	ASTM D -2596

Table 7. Effect of addition of Jojoba oil on properties of the selected prepared grease  $G_{1G}$

3.4 Effect of the jojoba meal additive

Because greases are colloidal systems, they are sensitive to small amounts of additives. To study the effect of jojoba meal additive on the properties of the selected grease  $G_{2C}$ , five grades of lithium lubricating greases containing different concentrations of jojoba meal additive were prepared. These concentrations included 1 wt%, 2 wt%, 3 wt%, 4 wt% and 5 wt% yielding  $G_{3A}$ ,  $G_{3B}$ ,  $G_{3C}$ ,  $G_{3D}$  and  $G_{3E}$  greases, respectively.

These greases have been prepared and formulated according to the percent ingredient listed in Table (8).

<div><div></div><div>Ingredient&amp; property</div></div>	Symbol					Test method
	G <sub>3A</sub>	G <sub>3B</sub>	G <sub>3C</sub>	G <sub>3D</sub>	G <sub>3E</sub>	
G <sub>2C</sub> , Wt %	99	98	97	96	95	
Jojoba meal, Wt %	1	2	3	4	5	
Penetration at 25°C						
Un worked	282	280	278	275	275	ASTM
worked	287	285	280	277	277	D-217
						ASTM
Dropping point, °C	188	190	192	195	198	D-566
						ASTM
Oxidation Stability 99± 96h, pressure, drop, psi	2.5	2.3	2.0	1.5	1.5	D-942
						ASTM
Intensity of (C=O) group @ 72h,	1.2	1.0	1.0	0.995	0.937	D-942
						ASTM
Intensity of (OH) group@ 72h	0.821	0.7921	0.7501	0.7023	0.6813	D-942
						ASTM
Alkalinity, Wt%	0.12	0.13	.14	0.15	0.15	D-664
						ASTM
Total acid number, mg KOH/g @ 72 h	0.15	0.15	0.14	0.12	0.12	D-664
						ASTM
Oil separation, Wt%	1.8	1.8	1.7	1.7	1.6	D-1724
						ASTM
Copper Corrosion 3h/100°C	Ia	Ia	Ia	Ia	Ia	D-4048
Code grease						
NLGI	2	2	2	2	2	
Egyptian Standard	LB	LB	LB	LB	LB	
						ASTM
Apparent Viscosity, cP, @ 90 °C	41820	42032	42232	42611	42652	D-189
Yield stress, D/cm²	80.6	82.5	85.0	86.4	86.6	
						ASTM
Four ball weld load ,Kg	235	240	245	250	250	D-2596

Table 8. Effect of addition of jojoba meal on properties of the selected prepared grease G<sub>2C</sub>



Data in this table reveal that all concentrations of the JM exhibit marked improvements in all properties of the investigated greases compared with the corresponding grease  $G_{2C}$  without jojoba meal. In addition, the difference of penetration values between unworked and worked for greases  $G_{3A-3E}$  decreased markedly by increasing jojoba meal content in the range of 1wt to 3wt%. Further increase of the jojoba meal concentration up to 4 and 5% by wt shows almost no difference. Parallel data are obtained concerning dropping point, dynamic viscosity, oil separation and total acid number of greases  $G_{3A-3E}$ . Such improving effect, as mentioned above, could be attributed to the high polarity of jojoba meal constitutes, which result in increasing both the compatibility and electrostatic forces among the ingredients of the prepared greases under investigation. Based on the improvement in the dynamic viscosity, consistency, dropping point and oil separation of the addition jojoba meal to the selected grease  $G_{2C}$  (Table 8), a suggested mechanism for this improvement is illustrated in the Schemes 1& 2. This suggested mechanism explains the ability of jojoba meal ingredients (amino-acids and polyphenolic compounds) to act as complexing agents leading to grease  $G_{3D}$  which is considered the best among all the investigated greases. This agrees well with previous reported results in this connection (El-Adly et al, 2009).

The aforementioned studies on the effects of fatty materials, jojoba oil and meal reveal that the selective greases are  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$ , respectively.

### 3.5 Evaluation of the selected greases ( $G_{1G}$ , $G_{2C}$ and $G_{3D}$ )

#### 3.5.1 Rheological behavior

Lubricating grease, according to rheological definition, is a lubricant which under certain loads and within its range of temperature application, exhibits the properties of a solid body, undergoes plastic strain and starts to flow like a liquid should the load reach the critical point, and regains solid body like properties after the removal of stress (Sinitsyn, 1974).

Rheology is the cornerstone of any quantitative analysis of processes involving complex materials. Because grease has rather complex rheological (Wassermann, 1991) properties it has been described as both solid and liquid or as viscoelastic plastic solids. It is not thick oil but thickened oil. The grease matrix is held together by internal binding forces giving the grease a solid character by resisting positional change. This rigidity is commonly referred to as consistency. When the external stress exceed the threshold level of sheer (stress or strain)-the yield value-the solid goes through a transitional state of plastic strain before turning into a flowing liquid. Consistency can be seen the most important property of a lubricating grease, the vital difference between grease and oil. Under the force of gravity, grease is normally subjected to shear stresses below the yield and will therefore remain in place a solid body. At higher level of shear, however, the grease will flow. Therefore, it is the utmost important to be able to determine the exact level of yield (Gow, 1997).

The rheological measurement of the selected greases is tested using Brookfield Programmable Rheometer HADV-III ULTRA in conjunction with software RHEOCALC. V.2. All Rheometer functions (rotational speed, instrument % torque scale, time interval, set temperature) are controlled by a computer. The temperature is controlled by connection with bath controller HT-107 and measured by the attached temperature probe. In this respect, the rheological behavior of the selected greases  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$  are determined at 90°C and 120 °C. Figures 1 and 2 afford nearly linear plots having different yield values. Also, they indicate that the flow behavior of greases at all temperatures obey plastic flow. This is due to

operative forces among lithium soap, lubricating fluid, jojoba oil and its meal. Also, the variety in fatty acids (soapstock and bone fat compositions) lead to the soap particles will arrange themselves to form soap crystallites, which looks a fiber in the grease. These soap fibers are disposed in a random manner within a given volume. This packing will automatically ensure many fiber contacts, and as a result, an oil-retentive pore network is formed, which is usually known as the gel network. When a stress is applied to this network, a sufficient number of contact junctions will rupture to make flow possible. The resistance value associated with the rupture is known as yield stress. Therefore yield stress can be defined as the stress value required to make a grease flow (Barnes, 1999).

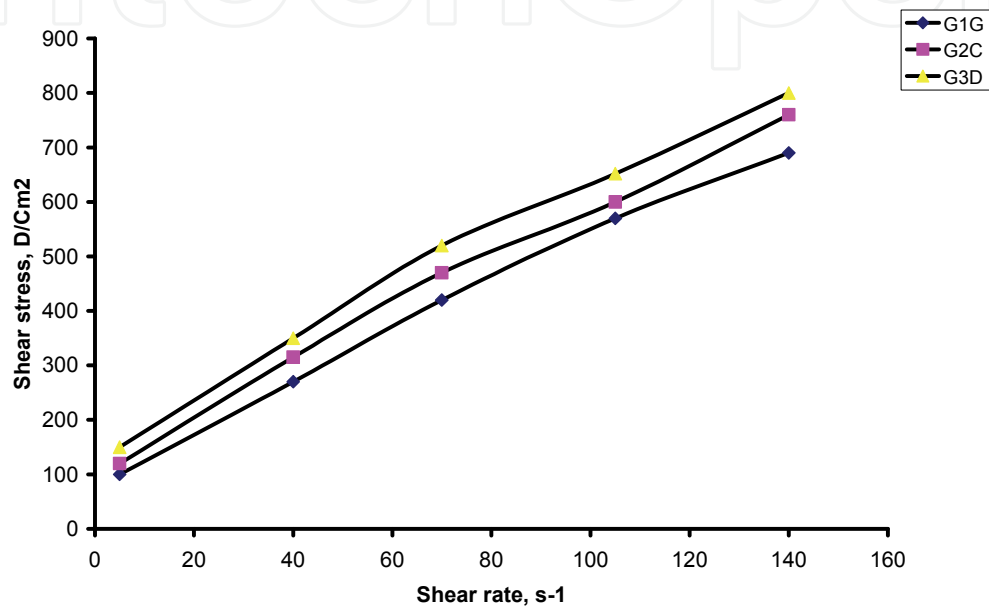


Fig. 1. Variation of shear stress with shear rate for  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$  at 90°C

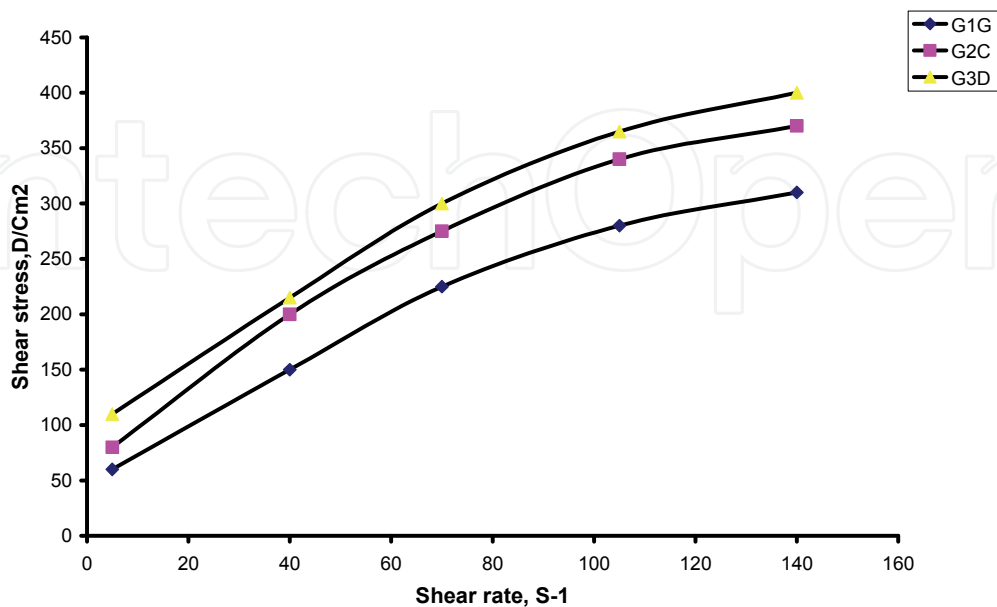
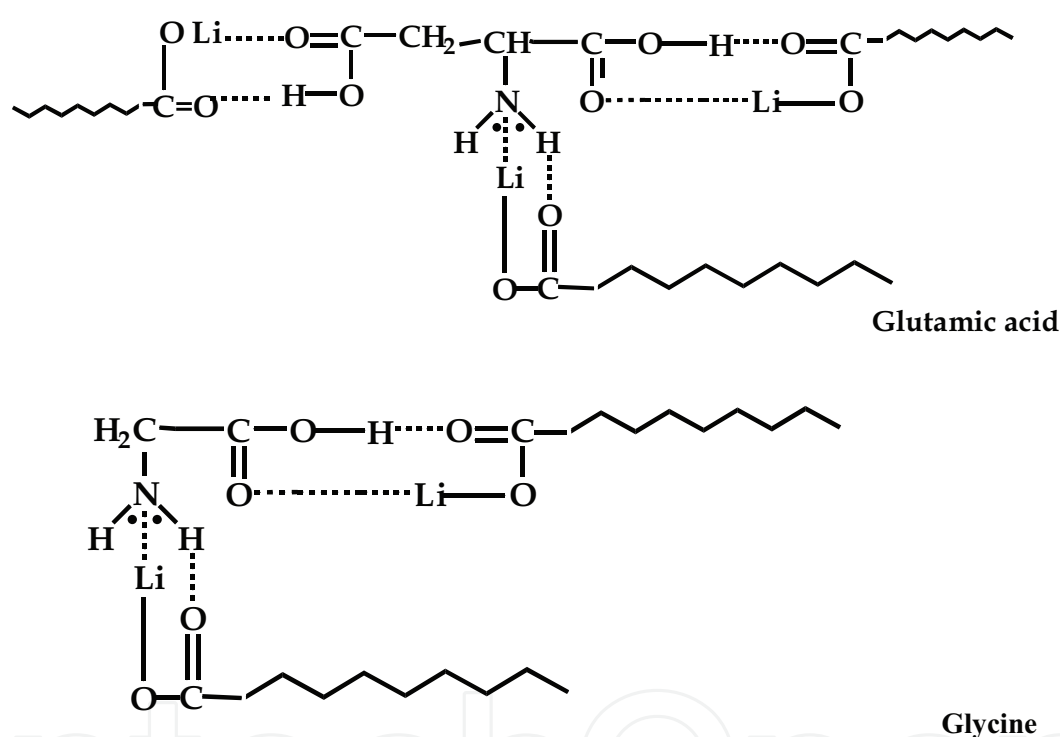


Fig. 2. Variation of shear stress with shear rate for  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$  at 120°C

In this respect, Rheological data apparent viscosity and yield stress (Tables 6, 7 & 8), for the selected greases show improvement and reinforcement in the order  $G_{3D} > G_{2C} > G_{1G}$ . This is attributed to the ability of jojoba meal to enhance the resistance to flow for  $G_{3D}$ , due to the action of the jojoba meal containing amino acids which act as chelating compounds, columbic interactions and hydrogen bonding, with Li-soap Scheme (1& 2). Also, according to the basic information on the composition of the jojoba meal (Verbiscar, et al., 1978; Cardeso, et al., 1980; Wisniak, 1994), amino acids, wax ester, fatty materials, polyphenolic compounds and fatty alcohols in jojoba meal could be acting as natural emulsifiers leading to increase in the compatibility among the grease ingredients. There is evidence that soap and additive have significant effects on the rheological behavior.

The flow and viscoelastic properties of a lubricating grease formed from a thickener composed of lithium hydroxystearate and a high boiling point mineral oil are investigated as a function of thickener concentration (Luckham & Tadros, 2004).



Scheme 1. The role of amino acids as complexing agent with texture of lithium soap grease

### 3.5.2 Extreme-pressure properties

Extreme pressure additives (EP) improve, in general, the load-carrying ability in most rolling contact bearing and gears. They react with the surface to form protective films which prevent metal to metal contact and the consequent scoring or welding of the surfaces. The EP additives are intended to improve the performance of grease. In this respect, the selected greases are usually tested in a four ball machine where a rotating ball slides over three stationary balls using ASTM-D 2596 procedure. The weld load data for the selected greases  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$  are 170, 195 and 250 Kg, respectively. These results indicate that the selected grease containing jojoba oil and jojoba meal  $G_{3D}$  exhibit remarkable improvement in extreme pressure properties compared with grease without additives  $G_{1G}$  and grease  $G_{2C}$  with jojoba oil alone. This may be attributed to the synergistic effect of the complex

combination among Li-soap, amino acids, and polyphenolic compounds scheme (1 &2), in addition to the role of anion ( $\text{PO}_4^{3-}$ ,  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$  and  $\text{F}^-$ ) and cation ( $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Co}^{2+}$  and  $\text{Ni}^{2+}$ ) in jojoba meal. These chemical elements are in such a form, that under pressure between metal surfaces they react with the metal to produce a coating film which will either sustain the load or prevent welding of the two metals together. This view introduces the key reasons for the improvements of the load-carrying properties and agrees well with the data previously reported by El-Adly et al (2004).

On other hand, it has been found that some thickening agents used in grease formulation inhibit the action of EP additives (Silver & Stanly 1974). The additives most commonly used as anti-seize and anti-scuffing compounds are graphite and molybdenum disulphide.

### 3.5.3 Oxidation stability

The oxidation stability of grease (ASTM D-942) is the ability of the lubricant to resist oxidation. It is also used to evaluate grease stability during its storage. The base oil in grease will oxidize in the same way as lubricating oil of a similar type. The thickener will also oxidize but is usually less prone to oxidation than the base oil. So, anti-oxidant additive must be selected to match the individual grease. Their primary function is to protect the grease during storage and extend the service life, especially at high temperatures.

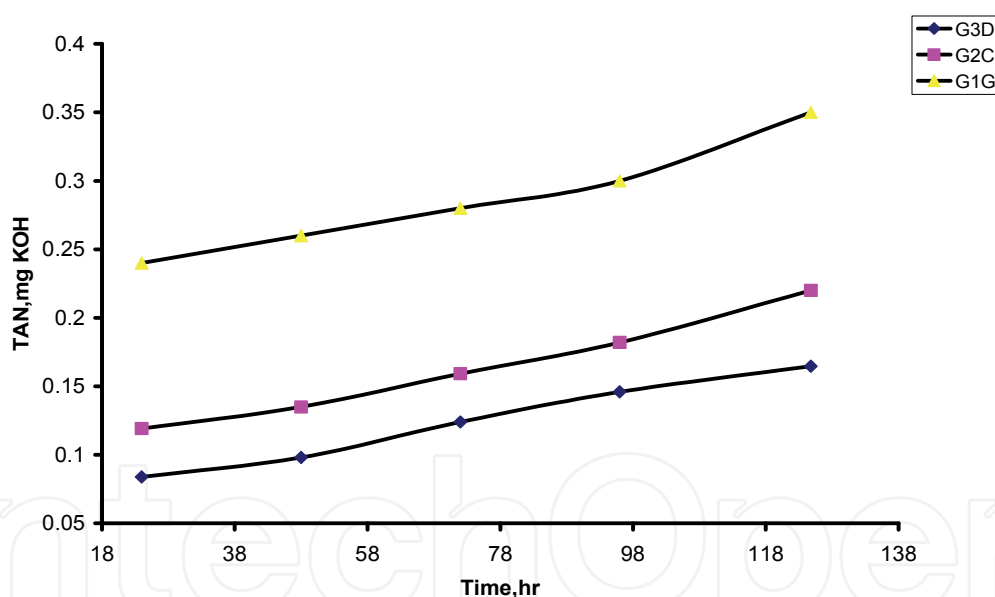
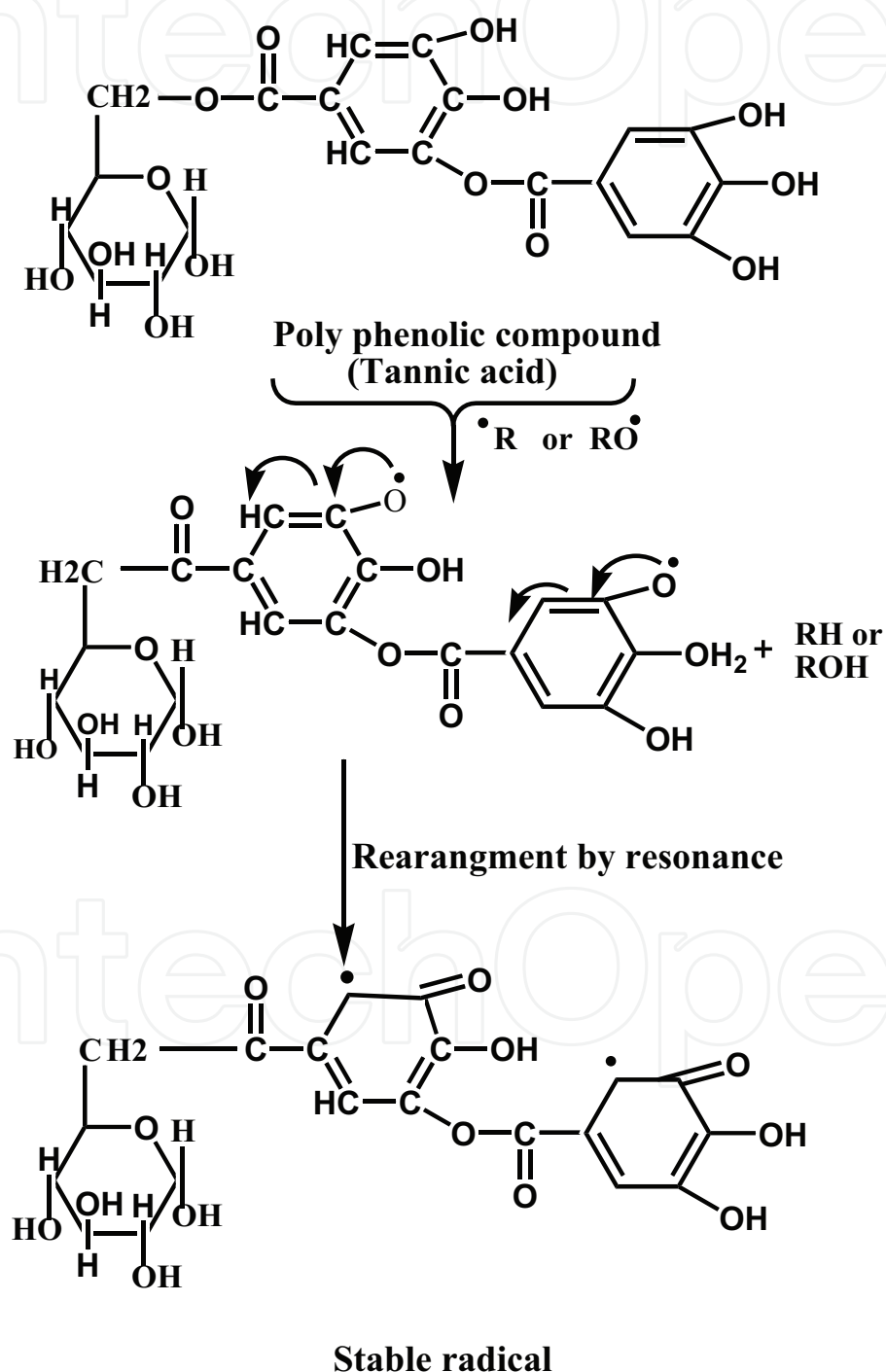


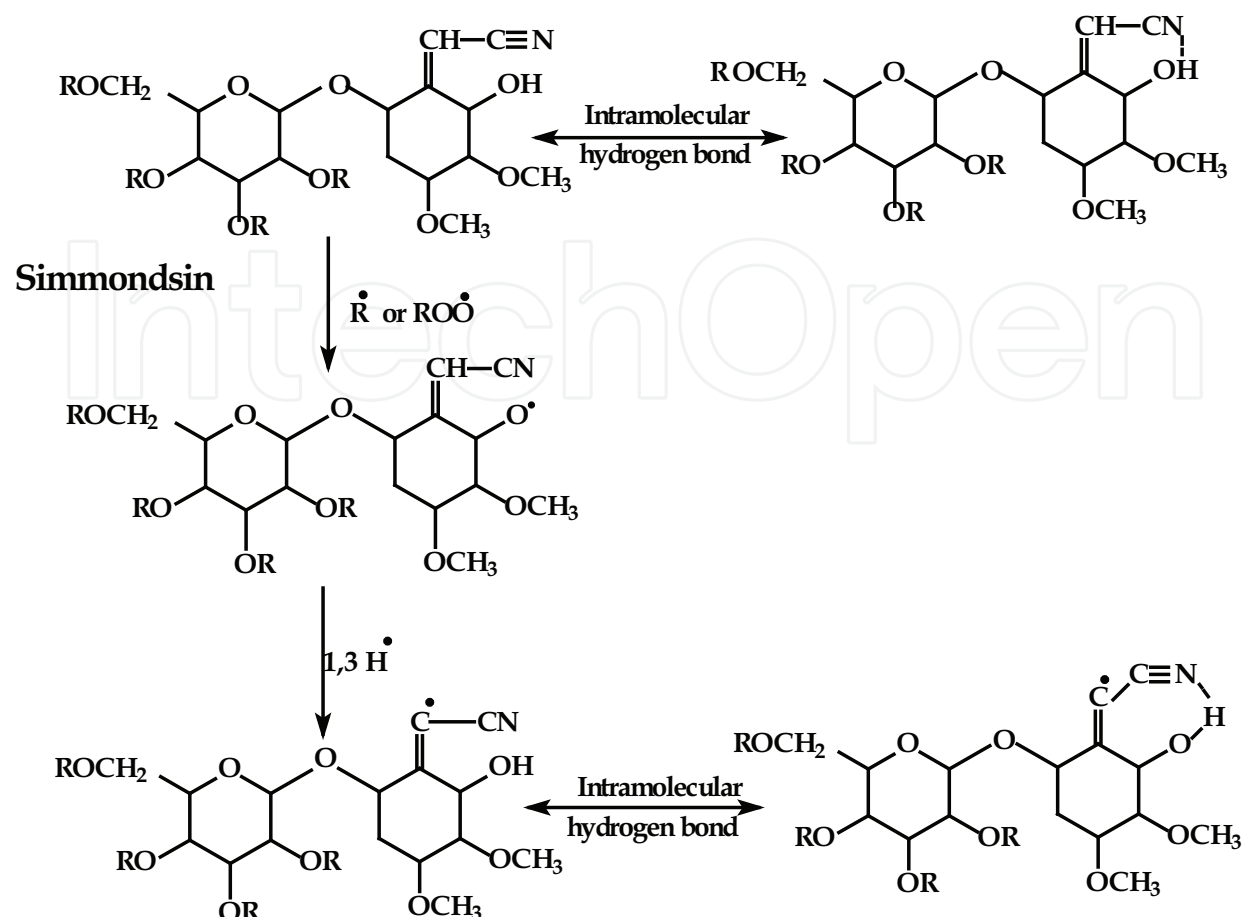
Fig. 3. Effect of deterioration time on Total Acid Number for selected greases

Oxidative deterioration for the selected greases  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$  are determined by the total acid number at oxidative times ranging from zero to 120 hours Figures (3). In addition, pressure drop, in psi. at 96 hour for greases  $G_{1G}$ ,  $G_{2C}$  and  $G_{3D}$  are 4.0, 3.0 and 1.5 psi respectively. These results give an overview on the efficiency of the jojoba meal and jojoba oil in controlling the oxidation reactions compared with the grease without additive  $G_{1G}$ . Jojoba oil in conjunction with jojoba meal additive proves to be successful in controlling and inhibiting the oxidation of the selected grease  $G_{3D}$ . Inhibition of oxidation can be accomplished in two main ways: firstly by removal of peroxy radicals, thus breaking the oxidation chain, secondly, by obviating or discouraging free radical formation. A suggested

mechanism for this inhibition is illustrated in the Schemes (2 & 3). The efficiency of jojoba meal ingredients as antioxidants is here postulated due to the presence of phenolic groups and hyper conjugated effect. Accordingly, Simmondsin derivatives and polyphenolic compounds which are considered the main component of jojoba meal include in their composition electron rich centers, which act as antioxidants by destroying the peroxides without producing radicals or reactive oxygenated products.



Scheme 2. The role of Polyphenolic compounds as antioxidant for prepared lithium grease



Scheme 3. The role of the Simmondsin as antioxidant for prepared lithium grease

#### 4. Future research

Base oils used to formulate greases are normally petroleum or synthetic oils. Due to growing environmental awareness and stringent regulations on the petroleum products uses, research and development in the area of eco-friendly grease is now gaining importance. Since biodegradable synthetic ester lubricant is higher in cost, vegetable oils are drawing attention economically as biodegradable alternates for synthetic esters. Looking forward into the next decade, the need for more advanced science in grease technology is essential. The design of special components is becoming increasingly complicated and machines are becoming much smaller and lighter in weight and are required to run faster and withstand heavier loads. To be able to develop the optimal lubricants for these new conditions, the mechanism behind grease lubrication must be further studied and understood. There will be an increased specialization in both products and markets and the survival of individual lubricants companies will depend on their ability to adapt to changing conditions. Not only machines but also new materials will affect the development of greases. Biogreases (El-Adly et al 2010) and nanogrease have better lubricating properties such as, wear protection, corrosion resistance, friction reduction, heat removal, etc. In this respect, anti-friction, anti-wear and load-carrying environment friendly additives are prepared from non-traditional vegetable oils and alkyl phenols of agricultural, forest and wasteland origin (Anand, et al, 2007).



## 5. Conclusion

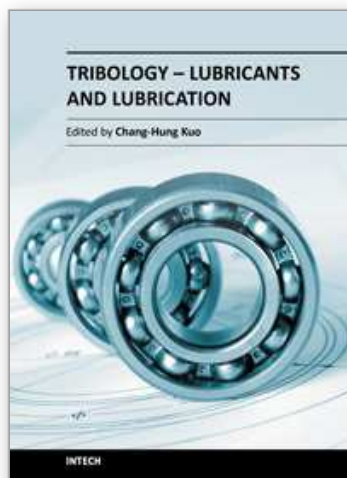
Lubricating grease is an exceptionally complex product incorporating a high degree of technology in all the related sciences. The by-products, soapstock, bone fat, jojoba meal, produced from processing crude vegetable oils are valuable compounds for lubricating greases. Such byproducts have varieties of chemical compounds which show synergistic effect in enhancing and improving the grease properties. Advantages of these byproducts include also their low cost and large scale availability. Research in this area plays a great role in the economic, scientific and environmental fields.

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## **Tribology - Lubricants and Lubrication**

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In the past decades, significant advances in tribology have been made as engineers strive to develop more reliable and high performance products. The advancements are mainly driven by the evolution of computational techniques and experimental characterization that leads to a thorough understanding of tribological process on both macro- and micro-scales. The purpose of this book is to present recent progress of researchers on the hydrodynamic lubrication analysis and the lubrication tests for biodegradable lubricants.

### **How to reference**

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