INFLUENCE OF SOME ADDITIVES ON PROPERTIES OF GREASES

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CERTIFICATION

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Abstract

This work aims to study the influence of some additives on the properties of soap greases, such as lithium, calcium, sodium, lithium-calcium by adding first: additives including graphite, molybdenum disulfide, carbon black, corrosion inhibiter and extreme pressure, second: base stock oils 40, 60, 150.

Various additives have been added to greases to obtain the best percentages that improve the properties of grease such as load carrying, wear resistance, corrosion resistance, drop point and penetration.

The result showed the best weight percentages to all types of grease which give good properties are 1.5% extreme pressure additive, 3% graphite, 1% molybdenum disulfide, 2.5% carbon black.

On the other hand, the best weight percentage for corrosion inhibiter is 1% to lithium-calcium grease, 2% to lithium grease and 3% to sodium grease. It was concluded that there is no need to add corrosion inhibiter to calcium grease.

For adding stock oils (40, 60, 150) to greases, a decrease in grease properties has been noticed when adding 20% of base stock oil 40, 30% of base stock oil 60, and no significant change being observed of base stock oil 150.

Nomenclatures

Symbols	Notations
1a	Slight tarnish (light orange) on copper strip corrosion test.
2a	Moderate tarnish on copper strip corrosion test.
1b	Slight tarnish (dark orange) on copper strip corrosion test.
cSt	Cent-stock (mm^2/s).
G	Elastic modulus.
Kg_f	Kilogram force.
N	Newton.

Abbreviations

ASTM	=	American Society for Testing and Material.
BC	=	Before Christ.
EP	=	Extreme Pressure.
IP	=	Institute of Petroleum.
NLGI	=	National Lubricating Grease Institute.
PANA	=	Phenyl alpha-naphthyl amine.
rpm	=	Revolution per minute.
SUS	=	Saybolt Universal Seconds.
VI	=	Viscosity Improver.
V.I	=	Viscosity Index.

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Chapter One

Introduction

Grease is a complex multi-phase material whose way of functioning needs to be clarified because of its growing use in modern machines [1].

True grease consists of oil and/or other fluid lubricant that is mixed with another thickener substance such as a soap to form a solid.

Greases generally cannot satisfy the requirements of high performance lubricants without using the benefit of modern additive technology. Additives are natural or synthetic chemical substances that can improve lots of different parameters of lubricants. They can boost existing properties, suppress undesirable properties and introduce new properties in the greases. Also, additives have a very big influence on the performance of lubricants that make it possible to fulfill new performance levels. The most common additives include corrosion inhibiter, anti wear and extreme pressure (EP) additives [2].

Solids, often called filler, are some times added to lubricating greases in concentrations varying from a fraction to several percent. These solids are most often inorganic materials such as asbestos, graphite, metal oxides, and metal powders or flakes metal sulfides, etc. [3].

The choice of lubricants for a specific application is a question that must be resolved by matching the characteristics of either oils or greases, and with service requirements and considering the relative advantages of each [4]. The answer is quite simple. It is always best to use oil as a lubricant whenever possible. Unfortunately, or perhaps fortunately for the grease makers, the ancillary equipment necessary for oil is often too large or too costly even to be considered. Oil may require complicated devices, or means of circulation for example pumps, and sometimes means of cooling.

All of these require space, often more than is available, and it is then that grease comes to rescue [5].

In general, grease lubrication is the best choice for:

1. Equipment that runs intermittently or is kept in storage for long time periods.
2. Extreme operating conditions (high temperatures, pressures shock loads, and low speed).
3. Badly worn equipment (thicker grease films work well in worn and enlarged clearances).
4. Where it is difficult or inconvenient to access for frequent oil lubrication, such as sealed-for-life electrical motor bearings [6].

Anti-wear and Extreme Pressure (EP) additives improve, in general, the load-carrying ability in most rolling contact bearings and gears. Extreme Pressure additives react with the surface to form protective films which prevent metal to metal contact and the consequent scoring or welding of the surfaces. The additives most commonly used as anti-seize and anti-scuffing. Fillers are sometimes used as fine solids in grease formulations to improve grease performance. Typical fillers are graphite, molybdenum disulphide, carbon black and others [7].

Satisfactory lubricating greases for general use should have the ability to prevent corrosion, particularly rusting. This action is very often due to water or salt water which breaches the grease film. Additives, which adhere to the metal surface and have polarity which resists penetration of water, are necessary if rusting is to be preventing [8].

A number of tests were developed and standardized by the American Society for Testing and Materials (ASTM) and the Institute of Petroleum (IP) which described the properties of performance characteristics of lubricants [9]. Users can identify required performance criteria by means of such specifications and check incoming stocks to assure product compliance with these criteria. Non–standard testing is often undertaken to evaluate grease characteristics under specific application or operating environments [4].

Grease has a wide number of characteristics and features, because of which it is used in a variety of industrial applications. The important properties, which affect the characteristics of grease, are amount and type of thickener, physical characteristics and oil viscosity, additives and low or high temperature performance.

The aim of the present work is to study the influence of some additives on properties of (lithium, calcium, sodium and lithium-calcium) soap greases by adding:

1. Additive which include graphite, molybdenum disulfide, carbon black, corrosion inhibiter and extreme pressure additive.
2. Base stock oils which include 40, 60, and 150.

Chapter Two

Literature Survey

2.1 Historical Background:

Grease-like lubricants were already known to the Sumerians and used by them in their wheeled vehicles from 3500 to 2500 BC [10]; it has also been stated that as early as 1400 BC the Egyptians used greases made from olive oil and or tallow and lime to lubricate the axles of their chariots [11], although only pig fat was reported for that purpose by ancient authors such as Dioscurides and Plinius Secundus [2]. Probably the first grease of the industrial age was patented by Partridige in 1835; it was a calcium grease, also made from olive oil or tallow [12]. Greases based on mineral oils and thickened by soaps were probably first proposed in 1845 by Raecz [13], and a sodium grease made with tallow was patented by Little in 1849 [14].

Aluminum soap-based greases are usually produced with pre-manufactured aluminum soaps, usually aluminum stearate. The first greases of this type were probably introduced by Lederer in 1933 [15].

Prior to 1940 nearly all the grease requirements were met with uninhibited mineral oil thickened with calcium, sodium, or aluminum soaps. Later, during the 1940's, the need of aircraft for improved low temperature performance resulted in the use of diesters as the fluid component of low-temperature aircraft greases.

A major advancement was made possible in 1942 by Earle's discovery of lithium soap thickened greases. This led to the development of water-resistant greases that had high melting points and superior low-temperature properties [16].

In 1945 the British patent 570,500 pointed to the rust preventative properties are improved by incorporating 0.2 to 5 percent of zinc salt of a naphthene sulfonate in a lubricating grease [17]; both a soap and a fatty acid are used for this purpose, namely, 0.05 to 5 percent of an oil-soluble alkali metal salt of a soap-forming fatty acid or 0.1 to 5 percent of a dehydrated sulfated castor oil or the alkali metal of alkaline earth metal salts thereof, together with a normally liquid, oil-soluble free fatty acid in amount at least equal to that of the soap or castor oil compound [18].

Brophy and Zisman in 1950 stated that a close relationship has been demonstrated between the wear-prevention and rust-inhibitive properties of amphipatic polar compounds. Good polar rust inhibitors are always wear preventives and vice versa; but the molecular structure for optimum rust inhibition need not be the same as for optimum wear prevention. They also observed that if amphipathic polar additives are to give wear prevention at temperatures above 150 to 200°C, they must be able to react chemically or combine with the bearing metal [19].

Brophy and Zisman in 1951 mentioned that fatty acids are much more effective as rust inhibitors than compounds which are devoid of acid hydrogen atoms such as alcohols, ketones, esters, and amides, or groups able to coordinate with the metal or its oxides. Also it was stated that the polyvalent metal soaps of fatty acids and derivatives are found more useful as rust preventives than the acids but this may be due to the fact that acid is supplied through hydrolysis, as it is needed, for coating exposed areas of metal [20].

Nelson in 1952 thickened a lubricating oil with 10 to 25 percent of high molecular weight wax acids to form a grease like product which was said to act as a rust-preventive. Also, the addition of approximately 6 percent of acids, derived from the oxidation of petroleum fractions, to "Bentone" –thickened lubricating greases in order to prevent corrosion when the lubricant is in service [21].

Macdonald and Dreher in 1953 stated that amines, amine salts, metallic sulfonates, and naphthenates, esters and nonionic surfactants have been suggested as rust preventives. They consider that such materials effective because of their affinity for the metal surfaces or because they lower the water-grease interfacial tension. Attention is devoted to the fact that response of additives as rust preventives is dependent upon the type of lubricating grease to which they are added [22].

Calhoun in 1962 reported that the tendency of molybdenum disulfide to decrease the wear of greases was shown by results of laboratory tests made at Rock Island Arsenal. However, the extreme pressure properties of greases were increased by the addition of this agent [23].

Devine and others in 1964 found that when a lubricating grade of molybdenum disulphide was used "as an additive to diester lubricating greases substantially increased load carrying capacity and functions to reduce wear in the high load regions" resulted. Further, such an addition produced no adverse effect on storage stability, oil separation or rust preventive properties of the lubricants. It was noted that field experience with the compositions verified the laboratory findings [24].

Another summary of experiments in 1968 shows the effect of variables in formulating greases containing molybdenum disulphide by Caruson and

Gerard. Since the percentage of the additive suggested is about three but benefits were obtained below this proportion; the optimum particle size was found to vary with the application; suggestion was made that the acidity value of MoS_2 should be considered in formulating grease. While no specific data are given, it is said: "MoS_2 performance can be affected by other additives in a grease formulation: the effect can be positive, even synergistic, enhancing the MoS_2 performance, or it can be negative, diminishing the MoS_2 performance". They mentioned that the oxidation stability of non-inhibited grease can be improved by the addition of MoS_2 and that "all" MoS_2 greases investigated can be inhibited to pass stringent corrosion test [25].

Sinitsyn in 1974 suggested a new definition of lubricating grease incorporating the rheological properties "a grease 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 fluid should the load reach the critical point, and regains solid-body properties after the removal of the stress" [26].

Tarunendr Singh in 2000 showed that the blends of bis (1,5-diaryl-2,4-dithiomalonamido) dioxomolybdenum complexes in lithium base grease are evaluated for their extreme pressure activity in a "four-ball test" using 12.7 mm diameter alloy steel ball specimen. The additive, bis (1,5-di-pmethoxyphenyl-2,4-dithiomalonamido) dioxomolybdenum and bis (1,5-di-p-chlorophenyl-2,4-dithiomalonamido) dioxomolybdenum exhibited lower values of wear scar diameter at higher load and higher value of weld load, flash temperature parameter and pressure wear index as compared with lithium base grease without additives. The greases fortified with the developed additives, prevent rusting and corrosion of bearings while grease containing no additives did not pass these tests per the standard tests. These greases have also better oxidation protection as compared to the grease have no additive. The topography and tribochemistry of the wear-scar surface are carried out by means of Scanning Electron Microscopy and Auger Electron Spectroscopy techniques, respectively [27].

Khudher in 2002 found that different percentages of powdered date stones were added to lithium base grease; the range of addition was 5-20%, the behavior of date stones addition on the wear is noticed to be similar to that of molybdenum disulfide and carbon black. It was noticed that the addition of date stones produces a significant improvement in the properties of the prepared EP lithium grease. Comparing the effect of date stones with that of molybdenum disulfide, it is noticed that 5% of date stones minimized the wear down to 0.425 mm and maximized the welding up to 800^+ kg_f which is equivalent to the effect of 10% molybdenum disulfide and much effective than carbon black [28].

Couronne and others in 2003 found that the correlation between tribological behavior of grease and its composition and structure. A tribological investigation was conducted to various 20 lubricants. The following parameters were varied: base oil, soap and presence of additives. To ensure efficient control of grease composition, greases containing the same type of soap were manufactured from the same concentrated soap sample.

Film thickness measurements showed that the thickener microstructure is not the determining factor for the formation of a thick lubricant film. Nevertheless, the soap-base oil interaction is an essential parameter. The composition of a grease influences oil bleeding, mechanical stability, and rheological behavior. The elastic modulus (G) seems to be the only parameter directly linked to tribological behavior [1].

2.2 Lubricating Grease Description:

The American Society for testing and Materials (ASTM) defined lubricating grease as a solid or semi-fluid lubricant consisting of a thickening agent in a liquid lubricant. To improve certain properties and functions, additional components are sometimes included, called additives [29].

Grease is a lubricant which has been thickened in order that it remains in contact with the moving surfaces and not leak out under gravity or centrifugal action, or be squeezed out under pressure [30].

Grease is a mixture of a fluid lubricant, a thickener, and additives. The fluid lubricant that performs the actual lubrication can be petroleum (mineral oil), synthetic oil, or vegetable oil [6]. Common thickeners are the fatty acid soaps of lithium, calcium, sodium, aluminum, and barium in concentration of 8-25 wt % [31].The fatty acids that are employed in forming the soaps usually are oleic, palmitic, stearic, and other carboxylic acids derived from tallow, hydrogenated fish oil, castor and, less often, wool grease and rosin [32].The thickener gives grease its characteristic consistency. 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 enhance performance and protect the grease and lubricant surfaces [6].

2.3 Function of Lubricating Grease:

The importance of lubricating grease to our industrial economy is readily shown by a listing of the reasons for employing this product and the advantages obtained by its uses. Some of these advantages are:

1. Less frequent application is necessary with lubricating grease than with lubricating oil and consequently the over all cost of both the lubricant and the labor of serving are reduced.
2. Lubricating grease acts as a seal against the entrance of dirt and dust. It likewise acts as a seal against leakage of material being handled through valves.
3. Dripping and spattering is almost eliminated when a machine is properly grease lubricated. When lubricating grease is used, equipment may be operated in a vertical position without leakage under normal conditions.
4. Less expensive seals are required for a grease lubricated-bearing. In general, seals which are effective in preventing oil leakage have the disadvantage of increasing friction and power consumption.
5. Lubricating grease will insure some lubrication even when a bearing is neglected for a long period.
6. If the proper lubricating grease is used it will usually cling to bearing surfaces better than lubricating oil. Therefore, with grease lubrication there is less danger of the rusting which might occur through drainage of lubricating oil from bearing surfaces when a bearing is not in use for long periods.
7. Certain lubricating greases solve the problem of lubrication without corrosion in the presence of water.

8. Lubricating grease has the advantage of minimizing starting friction of journal bearings [33].
9. In some locations, lubricating greases will reduce noise and vibration, for instances, by cushioning grease-lubricated gear teeth.
10. Lubricating grease is generally preferable under extreme operating conditions such as high temperatures, extreme pressures, low speeds, shock loading or in bearings operating intermittently or reversing frequently.
11. Where machine parts are badly worn, lubricating grease is almost the only means of providing lubrication.
12. Most lubricating greases will operate over a wider temperature range than will one particular lubricating oil.
13. Lubricating Greases are available at reasonable prices considering service rendered [34].

The only basic disadvantage of grease, compared with oil, is that it is less efficient as a coolant while it naturally cannot be used as flushing agent for a circulating system [35].

Greases are most often used instead of fluids where a lubricant is required to maintain its original position in a mechanism, especially where opportunities for frequent relubrication may be limited or economically unjustifiable. This requirement may be due to the physical configuration of the mechanism, the type of motion, the type of sealing, or to the need for the lubricant to perform all or part of any sealing function in the prevention of lubricant loss or the entrance of contaminants. Because of their essentially solid nature, greases do not perform the cooling and cleaning functions associated with the use of a fluid lubricant [36].

2.4 Nature of Lubricating Greases:

The technology of grease manufacture is largely concerned with means of economically and homogeneously dispersing the thickener agent in the liquid phase. Fundamental knowledge on the nature of the grease system has increased rapidly over years and has studies with electron microscope, in particular, have contributed greatly to understanding.

All solids will act as a thickening agent in a liquid lubricant and form grease depends on its ability to remain suspended and to exert inter-particle forces, which will keep the system in relative equilibrium. The nature of these forces depends in turn on the type of thickening agent-employed [37].

A physical examination of soap thickened grease shows it to be of fibrous nature. The fiber structure will depend predominantly upon the type of soap used and the method of manufacture [38].

Crystal's soap consists of layers of double molecules placed end-to-end, hydrocarbon-to-hydrocarbon, and carboxyl group to carboxyl group. These molecular arrangements have been agreed to by investigator and indicate the distribution of molecules in soap fibers Fig. 2-1 [39].

The length and diameter of individual fiber will control the characteristic of the final grease structure [40]. The fiber length may be short which results in smooth buttery texture. Alternatively, the fiber length may be long which will result in a stringy type texture [38].

The thickeners in non-soap greases are usually particulate rather than fibrous in nature and the structure particles are extremely fine [41].

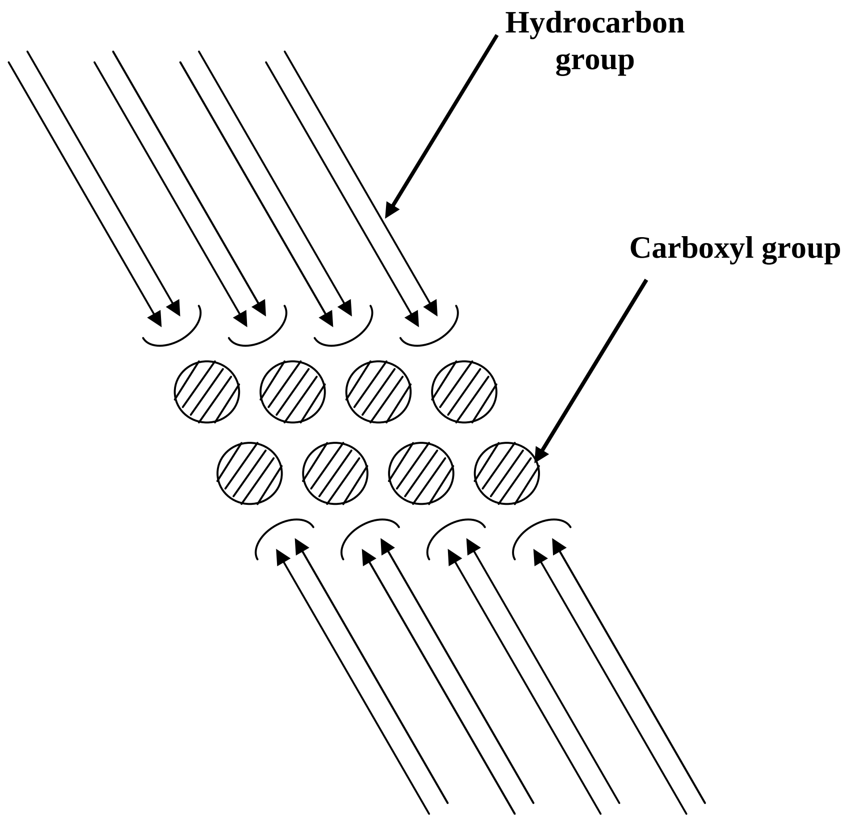


Figure 2-1 The distribution of molecules in soap fibers**.**

2.5 Rheological Characteristics:

The general definition of rheology is the science that deals with the deformation of flow of materials. Similarly, it may be stated in general terms that the primary objective of lubrication is to separate rubbing surface by a layer of lubricant in order to prevent excessive friction and wear. It is clear that the lubricant during lubrication process will be subjected to quite high shearing stresses and velocity gradients. The rheological properties of the lubricants are, therefore, likely to have an important effect of its action [42].

Greases are plastic non-Newtonian lubricants, i.e. there is a minimum value of applied stress called the yield stress or yield value, which is required to cause flow. While at higher stress they behave as viscous fluids, the viscosity falling rapidly with increasing stress and tending finally to the viscosity of the fluid component [41].

A factor, which greatly influences grease structure, is amounting of shear it undergoes in a bearing. Effect of shear, or working of the grease, is to cause break down of the structure with the result that stiff grease may assume the properties of lower consistency grease. Greases should therefore be of good mechanical stability [38]. As the rate of shear increases, the apparent viscosity falls progressively as a result of break up of the aggregates. Under very severe conditions of shear and turbulence, as in the track of ball- or roller- bearing, the individual crystalline fibers may themselves break into smaller ones [37].

2.6 Composition of Grease:

2.6.1 Fluid Lubricant:

Fluid lubricants used to formulate grease are normally petroleum (mineral oils) or synthetic oils. With growing environmental concerns, vegetable oils and certain synthetic oils are also being used in applications requiring oils and environmentally acceptable oils. They describe the characteristics that each type of oil brings to grease [6].

Base oils (liquid phase) are the major components in grease formulations and, as such, exert considerable influence on behavior of grease. In formulating grease, a base-oil viscosity is usually chosen that is similar to that which would normally be chosen if the equipment were oil lubricated [43].

In general, higher viscosity base oils are used when increased loads or duties are expected to be imposed on the grease and higher temperature or low speed and it may be necessary to include extreme pressure additives and also corrosion inhibitors.

On the other hand, a light neutral oil might be chosen to formulate a grease lubricant for high-speed, lightly load bearing and lower temperature [38].

2.6.2 Thickeners:

The thickener may be any of a number of materials that include soaps, such as those formed by the saponification of a fatty material with an alkali; non soap chemical compounds, such as those based on a urea derivative; and inorganic materials, such as those made from certain clays (i.e., montmorillonite). The success of a thickener to form a grease depends on one or more of the following:

1. The solvency of the fluid medium for the thickener and the solubility of the thickener in the fluid.
2. The attractive forces between the thickener and the fluid.
3. The capillary forces that hold the fluid within the network of thickener particles (or fibers).
4. The mechanical inter locking of the fiber network of the thickener itself.

For a thickener system to be effective in a grease, it must have the proper fiber structure [44].

The followings are the main types of thickeners:

2.6.2.1 Soap Base Thickener:

This type is dispersed in its base fluid, it gives grease its physical character. Soap thickeners not only provide consistency to grease, they affect desired properties such as water and heat resistance and pumpability.

The principal ingredients in creating a soap are a fatty acid and an alkali. Fatty acids can be derived from animal fat such as beef tallow, lard, butter, fish oil, or from vegetable fat such as olive, castor, soybean, or peanut oils [6].

Most fatty materials contain both saturated and unsaturated acid in the range C_{12} to C_{18} chain length and in some case (especially fish oil) to C_{20} and C_{22}. The type of fatty material affects the melting point of the soap and the degree of un-saturation may affect grease texture [41].

The most common alkalies used are the hydroxides from earth metals such as aluminum, calcium, lithium, and sodium. Soap is created when a long-carbon-chain fatty acid reacts with the metal hydroxide. The metal is incorporated into the carbon chain and the resultant compound develops a polarity. The polar molecules form a fibrous network that holds the oil. Thus, a somewhat rigid gel-like material "grease" is developed.

The name of the soap thickener refers to the metal (calcium, lithium, etc.) from which the soap is prepared [6].

2.6.2.2 Non Soap Base Thickener:

It is probably sufficient to define these as any greases made with gelling agents other than metal soaps. The distinguishing characteristic, and that of most significance, is insolubility of the gelling agent in the liquid phase at all temperatures [37].

Non-soap thickening agents were developed for greases needing superior, high temperature performance characteristics [45].

Probably the most important and technically interesting of non-soap greases are these used for aircraft and spacecraft. Non-soap gelling agents, who are either used commercially or have potential value, are essentially inorganic, that includes carbon black, silica and organophilic clays. The organic non-soap gelling agents include sodium octadecyl terephthalamate, aryl-subtituted ureas, copper phthalocyanines and indanthrene blue [37].

The upper limits for the thickeners are determined by phase transformations of thickener, which cause modification in the grease structure [46]. However, the presence of mineral oil limits their upper temperature use to approximately 200°C [38].

2.6.3 Additives:

Chemical substances added to a petroleum product to impart or improve certain properties [47]. 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 corrosion inhibiters, extreme pressure, antiwear, and viscosity index (VI) improver.

In addition to these additives, boundary lubricants such as molybdenum disulfide 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 [48].

The general rule is to only use an additive if it is necessary, the reason being that they increase production cost [49]. Table 2-1 gives the basic qualities that can be improved by additives [50].

Table 2-1 Added qualities due to additives [50].

Additive	Purpose
extreme pressure additive	Increase load carrying capacity, protection against micro-welding.
Anti-wear additive	Reduction of wear in the mixed friction regime.
Friction modifiers	Reduction of friction losses, imposed stick-slip and noises, neutralization of acid aging products.
Corrosion inhibitors	Protection against corrosion attack on metallic materials.
Oxidation inhibitors	Retard oxidation, reduction of surface layer and sludge formation.
Solid lubricants	Improvement of load carrying capacity, reduction of stick-slip.

The types of additives that are added to grease help determine how it will be used. Low-speed applications (e.g. hinge pins and chassis point) don't require the same types of additives as high-speed application (e.g. wheel bearings and universal joints).

Low speed, or light loaded, applications require additives that prevent rust and corrosion. Heavily-loaded applications require extreme pressure additives and may benefit from the additive molybdenum disulfide. Molybdenum disulfide helps protect components like hinge pins and bearing surfaces against the damaging effects of excessive shock loads. Water-resistance applications require greases with extra polymers to guard against water washout [51].

2.7 Properties of Lubricating Grease:

2.7.1 Influence of Base Oil on Properties of Grease:

The fluid (liquid phase) has the major influence on flow properties, particularly at low temperatures, but is of greatest significance in its effect on life at high temperatures. Thus, not only because it constitutes 80-95 % of the average grease but also for its contribution to performance, the liquid phase merits as much attention as the gelling in formulating a grease [37].

The most important property of any lubricant is viscosity. The viscosity of oil is a measure of its internal resistance to flow. The higher the viscosity the thicker the oil and the thicker the film of the oil that clings to a surface. Depending upon the service for which it is used, the oil needs to be very thin and free-flowing or thick with a high resistance to flow [52].

Yet, a general conclusion is that greases containing low viscosity oils will show less thickening at low temperatures than will those containing higher viscosity oils [36].

Compatibility of lubricating oils as the characteristic of lubricating oils to be mixed together without significant degradation properties or performance [53]. Compatibility of oils with other ingredients, most often the gellants, may determine the choice of a fluid or use of certain oil blends.

The critical solution temperature above which soaps are completely miscible in all proportion with specific fluids influences the nature of the thickener particles formed upon cooling. This in turn influences certain characteristics of lubricating greases.

The importance of synthetic lubricating oils stems from the fact that properties, not obtainable with derivatives of petroleum, can be secured and this contributes desirable characteristics to greases. Such properties, depending upon specific fluids may include utility over a wider temperature range and greater resistance to oxidation than in most natural fluids [36]. Synthetic oil is consisting of chemical compounds, which were not originally present in crude oil (petroleum) but were artificially made (synthesized) from other compounds.

Synthetic oil could be made to be a substitute for petroleum or specially made to a substitute for lubricant oil such as conventional (or mineral) motor oil refined from petroleum [54].

It is quite common practice to blend lubricating oils of different viscosities to arrive at composite fluids which will have the characteristics desired in a finished lubricating grease.

Most oils used in greases will fall in the range of oils used to blend motor oils.

Economy, provided specifications are correct, often dictates what oil blends to employ in lubricating greases. When it comes to using other fluids as blends in greases, like synthetics, most often they should provide special properties and also be compatible [36].

2.7.2 Influence of Soap Gellants on Properties of Grease:

2.7.2.1 Complex Soap:

A complex soap is formed by the reaction of fatty acid and alkali to form a soap and the simultaneous reaction of the alkali with a short-chain organic or inorganic acid to form a metallic salt (the complexing agent).

Basically, a complex grease is made when a complex soap is formed in the presence of a base oil. Common organic acids are acetic or lactic, and common inorganic acids are carbonates or chlorides. A complexing agent made from a salt of the named metal is the additional ingredient in forming a complex grease.

The dropping point of a complex grease is at least 38 $^{\circ}$C (100 $^{\circ}$F) higher than its normal soap thickened counterpart, and its maximum usable temperature is around 177 $^{\circ}$C (350 $^{\circ}$F). Complex soap greases are limited to this temperature because the mineral oil can flash, evaporate, or burn above that temperature.

Generally, complex greases have good all-around properties and can be used in multipurpose applications. For extreme operating conditions, complex greases are often produced with solid lubricants and use more highly refined or synthetic oils [6].

2.7.2.2 Age Hardening:

However, almost all greases exhibit some hardening if left undisturbed after being sheared. This is actually a different phenomenon and is known as *Thixotropy*.

Thixotropy (in lubricating grease) that property which is manifested by decrease in consistency, or softening, as a result of shearing, followed by an increase in consistency, or hardening, beginning after shearing is stopped. (Thixotropic age hardening is a relatively prolonged process proportional to ageing time and is seldom, if ever, complete, whereas the apparent viscosity increase which occurs in non-Newtonian systems with decreasing shear rate is instantaneous and fully reversible. Lubricating grease is both thixotropic and non-Newtonian) [55].

Age hardening and thixotropy may differ only in degree but certain conclusions relative to the two seem evident. The smaller the proportion of thickener in a grease composition, the less should be the hardening [37].

2.7.3 Influence of Additives on Properties of Grease:

2.7.3.1 Antioxidants:

Oxidation inhibitor is natural anti-oxidants among the most important additives used in greases. Since the oxidation of greases is a problem of particular importance, due to the higher operating temperature [32].

Greases are apparently oxidized in different ways statically (as in storing) and dynamically (as in service), no doubt because of the different temperature involved [41].

In general, chemical compounds effective inhibiting oils against oxidation are also effective in grease [37].

The steps involving in the oxidation are Initiation, Propagation and Termination.

In the initiation stage, a hydrocarbon is thermally cleaved and a free radical formed. This is a highly reactive and unstable species that immediately combines with oxygen to form an even less stable species, which, in turn, reacts with a second molecule of hydrocarbon to form a hydro-peroxide and also to regenerate the initial radical. The hydro-peroxide may then decompose to form two radical species. Thus, for every one radical initially formed, there are three capable of initiating further reactions. For this reason, oxidation reactions, once stated, become exceedingly rapid. The final products of oxidation are gums, lacquers and acidic materials [56].

Oxidation inhibitors function by preferentially combining with the peroxides or radical species, there by terminating the free radical chain reaction. Chemical compounds typically used to inhibit oxidation include hindered phenols, aromatic amines, heterocyclic nitrogen compounds, and zinc di-alkyl di-thiophosphate and di-thiocarbonates [57].

A hindered phenols and aromatic amines are widely used in greases formations. Aromatic amines such as phenyl alpha-naphthyl amine (PANA), phenyl beta-naphthyl amine (PBNA), di-octyl di-phenyl amine and pheothiazianes are effective at high temperatures but generally provide little or no inhibition at low temperature [58].

The nature oxidation resistance of greases and the effect of any given oxidation inhibitor depends on the type of thickener, the relative acidity or basicity of the system and the nature of the base oil component [59].

The synthetic oil has been reported to be very resistant to heat and thermal oxidation [60].

2.7.3.2 Corrosion and Rust Inhibitor:

The corrosion of iron and steel bearing components caused by the presence of atmospheric oxygen and water can lead to serious malfunctions.

Protection of metal surfaces from rusting is specially important in steel rolling-mill applications where large volumes of cooling water impinge on the bearing and where function grease lubricants are expected to absorb much of that water without loss in performance [41].

The chief destructive mechanism is the atmospheric rusting of iron. Rusting is an electrochemical process and proceeds in the presence of air-providing oxygen and water. Small differences in electrochemical potential are usually present on iron surface and these sets up local anodes and cathodes [38].

Provided corrosion and rust inhibitor completely coat the metal, most lubricant greases are there competent corrosion preventive [61].

Two types of corrosion inhibitors are used commercially: oil-soluble material, such as lead soaps, molybdenum disulfide, and water-soluble compounds protect by strong absorption on the metal. Sodium nitrite is corporate as dispersion of very small crystals, to avoid roughness in bearing: it dissolves in any free-water present in the bearings and thus protects the metal [56].

2.7.3.3 Extreme Pressure / Anti Wear Agents:

The addition of an appropriate extreme pressure (EP) additive to the lubricant increases mechanical efficiency and diminishes wear and destructive heating reducing friction and avoiding surface damage of sliding surfaces whilst increasing the load carrying capacity [62].

Extreme-pressure agent functions under boundary conditions where metal surfaces are in intimate contact. As the surfaces move against one another, collision of surface aspirates produces localized temperature rises, which activate the EP agents. Distinct chemical compounds form and immediately plate out on the metal surface as a thin film. Sulfide, chloride and phosphate films shear more than the metal itself: consequently, less frictional heat is generated and the potential for severe welding is reduced [56].

Molybdenum disulfide has the advantage of durability under severe operating conditions such as temperature up to 400°C, which can break down the organic components of lubricating system. The MoS_2 component retains its lubricity under such conditions to prolong bearing life [63].

2.7.3.4 Viscosity Modifiers:

Viscosity modifiers are generally oil soluble organic polymers. Chemical structure and molecular size are the most important elements of the molecular architecture of viscosity modifiers. Many types of viscosity modifiers are available and choice depends on the particular circumstances and requirements [56].

One such is polyisobutylene having a molecular weight of a bout 800 and a viscosity of approximately 3000 SUS at 210 °F. This is marketed as a mineral oil solution. Other V.I. improvers include volatilized paraffin wax, unsaturated polymerized esters of fatty acids and monohydric alcohols, and condensation products of olefin and diolefin hydrocarbons [34].

Viscosity modifiers consist of aliphatic carbon to carbon backbones. The major structural differences are in the side groups, which differ chemically and in size. These variations in chemical structure are responsible for various properties of viscosity modifiers such as, oil-thickening ability, viscosity-temperature dependency and oxidation stability [56].

The action, no doubt, is a healing of the particle attachments of the gellants which were broken, or separated, by shear. If the thickener were of a fibrous nature, orientation rather than random arrangements might have taken place [36].

2.8 Manufacture of Lubricating Grease:

Grease manufacture is essentially the mixing of soaps with lubricating oils. To make grease the soap is dispersed in the oil as fibers of such a size that it may only be possible to detect them by microscopy. The fibers form a matrix for the oil, and the type, amount, size, shape, and distribution of the soap fibers dictate the consistency, texture, and bleeding characteristics, as well as the other properties of grease.

Grease may contain from 50 % to 30 % soap, and although the fatty acid influences the properties of grease, the metal in the soap has the most important effect. For example, calcium soaps from smooth, buttery grease that is resistant to water but is limited in use to temperatures under about 95 °C (200 °F).

The soaps may be combined with any lubricating oil, from a light distillate to a heavy residual oil. The lubricating value of the grease is chiefly dependent on the quality and viscosity of the oil. In addition to soap and oil, grease may also contain various additives that are used to improve the ability of the grease to stand up under extreme bearing pressures, to act as a rust preventive, and to reduce the tendency of oil to seep or bleed from a grease. Graphite, mica, talc, or fibrous material may be added to grease that is used to lubricate rough machinery to absorb the shock of impact; other chemicals can make a grease more to oxidation or modify the structure of the grease [64].

The following presumes the preparation of simple soap thickeners, batchwise. Here the manufacture may consist of:

1. Dispersion of the necessary saponifiable material in a portion of the necessary oil.
2. Addition of the saponification agent frequently dissolved or suspended in water.
3. Heating to promote soap formation.
4. Dehydration of the soap mass.
5. Additional heating to complete soap dispersion, generally in the less than the total required oil.
6. Crystallization of the soap melt (in many cases where soap is formed in situ in oil, the crystal structure will result without further treatment).

7. Cooling, frequently with further oil addition.
8. Introduction of additives.
9. Homogenization and / or milling; and, optionally deaeration.

Vessels heated by direct firing or hot-oil circulation and are generally coupled with water-jacketed finishing vessels (kettles).

The kettles are fitted with stirrers who should give a good wall-scraping action to assist heat transfer and good circulation to avoid dead spots [36].

2.9 Types of Greases:

2.9.1 Calcium Soap Grease:

Calcium soap grease is one of the earliest known greases and is water resistant and mechanically stable. Calcium soap grease usually has a low melting point (dropping point; typically ~ 95°C / 200°F). Anhydrous calcium soaps (usually calcium 12-hydroxystearate) are somewhat more temperature resistant, having a dropping point of about 150°C (300°C). Anhydrous calcium grease finds the greatest usage (when made with low-viscosity base oil) in operations where a wide range of climatic conditions is essential.

Calcium grease is resistant to water; it has smooth texture, its chief use is for plain bearing and low-speed rolling bearings, and it has a high roll stability. The water content of calcium grease usually varies between 0.4 % and 1.0 %, is present in the form of water of crystallization, and has a stabilizing effect. High temperatures cause a loss of water and a consequent weakening of soap structure, and therefore the use of this grease is limited to a maximum temperature of about 60 °C (140 °F) [64].

2.9.2 Sodium Soap Grease:

Sodium (soda) soap grease is fibrous in structure and is resistant to moderately high temperature but not to water. Sodium soap grease has a high dropping point (~ 175 °C / 350 °F) than calcium grease. However; it tends to emulsify in the presence of water but has inherent rust protection properties. This grease is used for rolling bearings at higher temperatures and speeds than normal conventional calcium soap grease [64].

2.9.3 Aluminum Soap Grease:

Aluminum soap grease is translucent and can be produced with aluminum soaps made in situ. In the process, the preformed soap (usually aluminum distearate) is dissolved in hot oil in a mixing grease vessel, and the hot mixture is poured into pans to cool. The cooling rate affects the final consistency. The final product is smooth, transparent grease with poor shear stability but excellent oxidation and water resistance. Aluminum soap grease is water resistant and adhesive but tends to have poor mechanical stability and so is not suitable for rolling bearings [64].

2.9.4 Lithium Soap Grease:

Lithium soap grease is normally smooth in appearance but may exhibit a grain structure. This type of grease is resistant to water and to the highest normal service temperatures and, because of the variety of types, is often used as multipurpose grease. Lithium grease offers both the water resistance of calcium soap grease and high-temperature properties of sodium soap grease. Grease prepared with lithium 12-hydroxystearate has a dropping point of about 190 °C (375 °F) [64].

2.9.5 Mixed Soap Grease:

A variety of grease gellants, consisting of mixtures of soaps of two different metals have been suggested and often employed commercially. Such blends are not confined to soaps alone but may be a soap and an inorganic gellant or a soap and any other type of thickener. In rare instances more than two different gellants may be present [65].

Mixed soap greases are generally manufactured by saponifying the fatty material with mixed alkalis derived from different metals. One of the soaps usually predominates and determines the general character of the grease, while the other modifies the structure in some way. This results, for example, in changes in texture and improved mechanical stability [66].

Mixed soap grease (e.g., the sodium / calcium soap grease) is manufactured for uses that include high-speed rolling bearings [64].

2.9.6 Complex Soap Grease:

Complex soap grease is formed when two dissimilar acids are attached to the same metal molecules, thus restricting complexes to only polyvalent metals [67].

This type of grease is some times referred to as salt-soap thickened greases. The salt most often used is calcium acetate but other salts have been included, like chlorides, nitrates, nitrides, phosphates, silicates and no doubt others [36].

Aluminum complex grease has excellent water wash-off and spray –off characteristics as well as high-temperature resistance and is widely used in steel mill applications and automotive components subject to these conditions [64].

Calcium complex thickened greases normally require a greater proportion of thickener than do products containing normal soaps. Dropping points of such compositions are high, water resistance satisfactory, shear stability fair but there is a tendency to harden upon storage [36]. Calcium complex grease has inherent extreme pressure properties and provides good friction and wears performance.

Lithium complex grease performance is generally like that of lithium 12-hydroxystearate grease except it has a dropping point approximately 50 $^{\circ}$C (90 $^{\circ}$F) higher. Lithium complex grease provides good low-temperature performance and excellent high-temperature life performance in tapered roller bearings [64].

2.9.7 Non Soap Grease:

Two non soap greases are worthy of mention. One is organic, the other inorganic.

2.9.7.1 Polyurea:

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 isocyanates, 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 $^{\circ}$C (-4 to 350 $^{\circ}$F) and has long life. Water-resistance is good to excellent, depending on the grade. 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 [6].

2.9.7.2 Organo-Clay:

Organo-clay is the most commonly used inorganic thickener. Its thickener is a 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 (350 °F). 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 (500 °F). 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 [6].

The special interest of organophilic-clay greases is bentonite base grease. It is high-temperature applications. This grease does not pass through transition and so do not normally show a drop point by standard test. It can be made with a variety of liquid lubricants. The quantity of thickening agent required for a given consistency can be according to the process used [37].

2.10 Tests Methods:

The more important characteristics of greases and test methods for evaluating them are discussed under separate headings below. Some tests are used primarily for control purposes to ensure uniformity of products, while others are intended to predict performance. Grease testing is essentially empirical and the results have to be considered in the light of practical experience [66].

2.10.1 Consistency:

The most important feature of a grease is its rigidity or consistency. A grease that is too stiff may not feed into areas requiring lubrication, while a grease that is too fluid may leak out. Grease consistency depends on the type and amount of thickener used and the viscosity of its base oil. A greases 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 D217 (standard test method for cone penetration of lubricating grease) and D1403 (standard test method for cone penetration of lubricating grease using one-quarter and one half scale cone equipment) methods measure penetration of unworked and worked greases. To measure penetration, a cone of given weight is allowed to sink into a grease for 5 seconds at a standard temperature of 25 $^{\circ}$C (77 $^{\circ}$ F). The depth, in tenths of a millimeter, to which the cone sinks into is the penetration. A penetration of 100 numbers or grade numbers, ranging from 000 to 6, corresponding to specified ranges of penetration numbers. Table 2-2 lists the National Lubricating Grease Institute (NLGI) grease classifications a long with a description of the consistency of each classification [6].

Table 2-2 NLGI grease classification [6].

NLGI Number	ASTM worked penetration 0.1 mm (3.28 *10^{-4} ft) at 25°C (77°F)	Consistency
000	445-475	Semi fluid
00	400-430	Semi fluid
0	355-385	Very soft
1	310-340	Soft
2	265-295	Common grease
3	220-250	Semi hard
4	175-205	Hard
5	130-160	Very hard
6	85-115	solid

2.10.2 Dropping Point:

Drop point is an indicator of the heat resistance of grease. As grease temperature rises, penetration increases until the grease liquefies and the desired consistency is lost. Dropping point is the temperature at which grease becomes fluid enough to drip. The dropping point indicates the upper temperature limit at which a grease retains its structure, not the maximum temperature at which a grease may be used. A few greases have the ability to regain their original structure after cooling down form the dropping point [6]. The more recent method is ASTM D2265 (standard test method for dropping point of lubricating grease over wide temperature range) has particular advantages when testing the modern high drop point grease [68].

2.10.3 Corrosion and Rust-Resistance:

This denotes the ability of grease to protect metal parts from chemical attack. The natural resistance of a grease depends upon the thickener type. Corrosion-resistance can be enhanced by corrosion and rust inhibitors [6].

Greases should not corrode any metals (normally steel or cuprous alloy) with which they come in contact [41]. As greases are employed under adversely of conditions, it is not possible here to specify either the temperature or the duration of the test, but it is recommended that the temperature be not higher than 20°C below the drop point of sample [37].

2.10.4 Oxidation Stability:

This is the ability of a grease to resist a chemical union with oxygen. The reaction of the grease with oxygen produces insoluble gum, sludges, and lacquer-like deposits that cause sluggish operation, increased wear, and reduction of clearances. Prolonged high-temperature exposure accelerates oxidation in greases [6]. Standard test method for oxidation stability of lubricating greases by oxygen bomb method in which a shallow trays of grease are exposed to oxygen at 99°C (210 °F) and 110 psi (7.7 kg/cm^2). The fall of pressure with time indicates the rate of absorption of oxygen by the grease [69].

2.10.5 Mechanical Stability:

The ability of a grease to with stand a large degree of mechanical working without changing its consistency unduly can be important during the initial clearing stages in a bearing or in certain applications, for example, in a bearing assembly under vibrating conditions. The effects of mechanical instability can lead in some cases to the grease becoming fluid and losing its sealing properties [64].

2.10.6 Oil Separation:

Grease will often separate oil during storage and, if too much oil separates, the grease could harden to the extent that lubrication performance will be affected. Oil will be released from a grease at varying rates depending on the gel structure, the nature and viscosity of the lubricating fluid, and the applied pressure and temperature [64].

2.11 Grease Applications:

The particular virtues of grease that make it preferred to oil in many cases, and especially for rolling bearings are ease in application and simplicity in use .

While oil can carry away heat from the lubrication point and in general will lubricate rolling bearings more efficiently, it needs good seals and relatively elaborate means of application such as circulation system. Grease, on the other hand, can be used in simpler housing designs because it can be retained easily and therefore seals against contamination and require much less attention and maintenance.

For simplicity reasons plain journal bearings not operating under critical conditions are commonly grease-lubricated. Naturally enough the less critical conditions can be the cheaper products, i.e. the conventional calcium soap grease which are mostly used for plain bearings and low-speed rolling bearings.

For rolling bearings operating at higher temperatures and speeds the choice can be made from the remaining types of grease depending on surface conditions; the more severe conditions require high-quality inhibited greases. These latter have a multi-purpose characters and are increasingly used in many plants with benefits in stock holding and freedom from misapplication, in place of the variety of cheaper greases that would otherwise be needed. Extreme temperature, say from 150 C upwards, call for greases made from non-soap thickeners. They are also needed for critical mechanisms in nuclear power plants where soap greases can not withstand conditions involving exposure to nuclear radiation.

It is important to use the right amount of grease in bearing, especially for high speeds. Too much can cause churning and over heating, which may result in the grease breaking down and running out of the bearing; too little can result in dry running and damage to the bearing.

Best practice is to pack the bearing full and leave enough free space in the covers to accommodate excess grease working out from the bearing during the initial running. This can generally be achieved by packing the covers to between about two-thirds and three-quarters of the total capacity.

If external contamination is severed the bearing speed is fairly low, effective sealing can be maintained by fully packing. The mechanism whereby grease lubricants rolling bearings is somewhat controversial.

It is accepted that under settled conditions the bulk of grease within bearing and housing is stationary and that only a very small amount of grease is actually circulating on the moving parts, this being rapidly degraded by the mechanical action of the rolling elements.

Some consider that this alone does the job of lubrication and that the soap itself plays a valuable part. Others consider that the lubrication is done by slow oil release from the stationary bulk of grease. It seams probable that both mechanism of lubrication may operate to generate a greater or lesser extent, depending on the nature of the grease and the application conditions, practically temperature [41]. Table 2-3 show detailed listing of greases applications [74].

Table 2-3 Service Type of Lubricating Grease [74].

Designation or service	Desirable qualities	Product normally used
Axle grease	Cheap, satisfactory for rough, slow-moving machinery	Calcium-rosin oil soap base. Almost passé.
Ball and roller bearing grease	Long service life, stable	Lithium, sodium, sodium-calcium, calcium complex, arylureas of polyureas.
Brick grease	Quite hard or stiff with a high melting point so that consumption is low	Most often sodium or sodium calcium products.
Chassis grease	Adhesive, resistant to washing effect by water. Oil viscosity SSU preferably 700 at 100 F or above	Any thickener base except water soluble ones.
Cup grease	Water resistant, low torque	Calcium with low-viscosity oil.

Farm grease	Adhesive, water resistant	Same as chassis grease.
Graphite grease	Clings to metal well	Cup or chassis grease containing graphite.
EP grease	Resists heavy pressures or shock loads. Frequently used in steel mills	Calcium or lithium products plus EP additives.
Pipe thread lubricants	Seals irregularities in threads yet permits joints to be broken readily	Often calcium-base greases plus fillers.
Plug valve lubricants	Resistant to solvent action. High melting point in most cases	Special products which may be high in proportion of both soaps and fillers. One product may serve for steam, another for solvent, and another for chemicals, etc.
Gasoline-resistant grease	Resistant to gasoline-type solvents	See plug-valve lubricants.
Steel-mill grease	For high pressures, water resistance	See EP grease.
Steering gear grease	Resistance to wiping action of worm gears	Soft greases often with EP additives or load soaps included.
Textile greases	Less frequent application than oils and yet of low torque. Should wash out of fabric or thread	Calcium-or sodium-base semi-fluid greases made with low viscosity, high-colored oils.
Tool-joint grease	Protect threads and permit ease of uncoupling	Similar to pipe-thread lubricants.

Track-roller grease	Adhesive, stringy, water resistant, and soft enough to feed readily	Aluminum, calcium, or lithium base product with high-viscosity oils.
Water pump grease	Melting-point above 210 F, water resistant, stiff	No.4 NLGI grade cup grease.
Wheel-bearing grease	High melting point, work stable	Complex calcium, lithium, or sodium base of No. 2 or 3 grades.

Chapter Three

Experimental Work

3.1 Materials:

3.1.1 Lubricating Greases:

The greases used in the experimental work are produced in Al-Daura refinery, as follows:

1. Lithium soap grease is produced by using lithium soap as a thickener. It is used in multiple industrial purposes, even in places subjected to vibration, for it is mechanically stable.
2. Calcium soap grease is produced by using calcium soap as a thickener which is a good water resistant and thus it is used in applications subjected to high humidity and a vast amount of water. It is used in automobile (chassis and wheel bearings) and many other low-speed general industrial purposes where the temperature does not exceed 70 $^{\circ}$C.
3. Sodium soap grease is produced by using Sodium as a thickener. Because it contains fibers, its main characteristic is its adhesion with oiled surfaces forming a strong barrier preventing solid particles, which cause wear, to enter to the bearing. It has good pressure resistance ability and is used for temperature not exceeding 120 $^{\circ}$C. Sodium greases are not allowed to be used when water is exist or in cases of high humidity because it is water soluble.

Table 3-1 shows the main characteristics of these greases used in the experimental work according ASTM methods.

Table 3-1 Characteristics of used lubricating greases.

Specifications	Lithium soap	Calcium soap	Sodium soap
Worked penetration (mm^{-1})	270	273.8	334.2
Drop point (^{o}C)	202	100	150
Copper corrosion test (24 h at 100 ^{o}C)	2a	1a	2a
Four-ball weld load (kg_f)	400	160	250
Four-ball wear test (wear scar diameter mm)	0.31	0.65	0.566
Texture	Soft	Soft	Fibrous
Color	Brown	Yellow	Green

3.1.2 Additives:

3.1.2.1 Extreme Pressure Additive:

HiTEC 343 is supplied from Ethyl Petroleum Additives Limited, London Road, Berkshire, England. Its properties are shown in Table 3-2.

Table 3-2 Properties of HiTEC 343 extreme pressure additive [71].

Property	Specification
Appearance	Bright Clear and Amber Liquid
Viscosity @ 100 ^{o}C, mm^2/s	9.0
Density @ 15.6^{o}, g/ml	1.082
Phosphorus, %wt	1.17
Sulfur, %wt	36.1

3.1.2.2 Graphite:

Graphite is supplied from a German company. Its general properties are shown in Table 4-3.

Table 3-3 Properties of graphite additive [70].

Property	Specification
Formula	C
Color	Black
Crystalline form	Hexagonal
Melting point (˚C)	4200

3.1.2.3 Molybdenum disulfide MoS_2:

Molybdenum disulfide is supplied from Al-khalil investment institution (Iraq) with 99% purity. Its general properties are shown in Table 3-4.

Table 3-4 Properties of molybdenum disulfide additive [70].

Property	Specification
Formula	MoS_2
Color	Black
Crystalline form	Hexagonal
Melting point (˚C)	1185

3.1.2.4 Carbon Black:

Carbon black is supplied from Al-Najaf Tiers Factory , Iraq.

3.1.2.5 Corrosion Inhibiter:

The corrosion inhibiter used is called Paranox-15. It is a zinc dialkyldithiophosphate (ZnDTP) with primary and secondary alkyl groups taken from Al-Daura refinery.

3.1.3 Base Oil:

Oil-base stocks oil are furnished by Al-Daura Refinery were used in the present investigating. These mineral oils base stock are: stock oil 40, 60 and 150.

These stocks have been selected on the basis that they are widely used in commercial production of lubricating oil and greases. Table 3-5 illustrates specifications of the used base stock oil.

Table 3-5 Specifications of base stock oils [73].

Specifications	Stock 40	Stock 60	Stock150
Viscosity (cSt) at 40 °C at 100 °C	 13-17 3-3.5	 60-90 8-10	 460 minimum 30-35
V.I	95 minimum	95 minimum	93 minimum
Flash Point (°C)	165 minimum	220 minimum	260 minimum
Pour Point (°C)	-12 maximum	-6 maximum	-3 maximum
Color	0.5	1.5	3

3.2 Experimental Procedure:

1. A 100 ml beaker was used.
2. A digital balance was set to zero when the beaker was empty.
3. The grease was put in the beaker; the additive was then added such that the balance reads 100 gm in each experiment.
4. The beaker was put on a hot plate whose temperature is 70-80 °C. The mixture was then stirred for 15 minute to insure that all parts of the mixture become homogenous.
5. The mixture was tested.

6. In the case where a mixture of greases was to be tested, the mixing weight percentage was 90% lithium grease and 10% calcium grease according to the U.S Patent 2,566,793 and the additive was then added and the tests were carried out.

3.3 Test Methods:

3.3.1 Four-Ball Welding Test (ASTM D-2596):

This test was aimed, to find the force required to cause metal surfaces to weld after subjected to friction under high pressure, using lubricating grease to be tested.

3.3.1.1 Apparatus:

1. Four-ball extreme pressure lubricant tester as shown in Fig. 3-1.
2. Timer, graduated in tenths of a second.

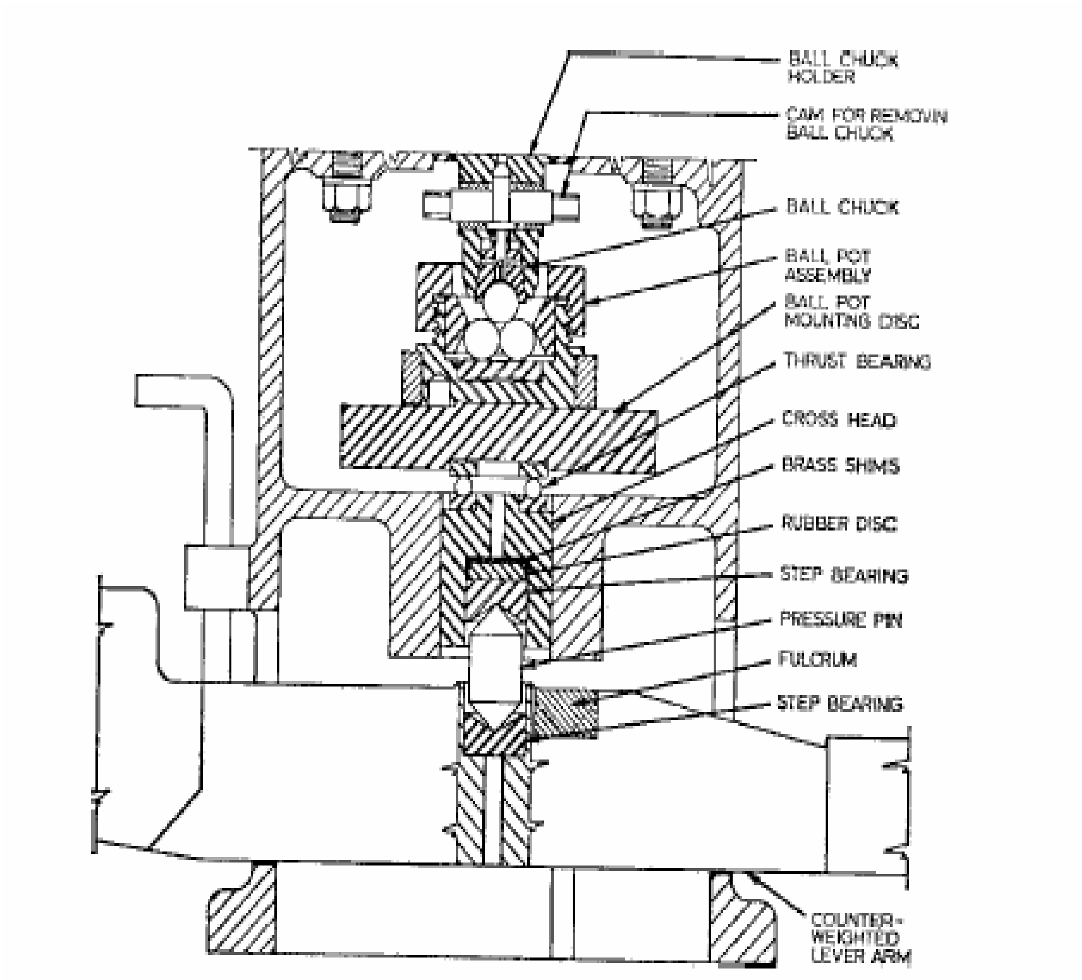


Figure 3-1 Sectional view of four-ball EP tester**.**

3.3.1.2 Procedure:

1. The four balls, ball pot and chuck assembly were thoroughly cleaned with kerosene.
2. The ball pot was filled completely with the lubricating grease to be tested, avoiding the inclusion of air pockets. The three steel test balls were embedded in the grease. The lock ring was carefully placed over the three balls and screwed down the lock nut securely. The excess grease was scraped off and pushed onto the lock nut.

3. One ball was pressed into the ball chuck and the chuck was mounted into chuck holder.
4. The ball pot assembly was installed on the test apparatus in contact with the fourth ball. The mounting disk was placed between ball pot and thrust bearing.
5. The weight tray and weights were placed on the horizontal arm in the correct notch for a base test load of 80 kg_f. The lever arm was released and the test load was gently applied to the balls, and the ball pot assembly and mounting disk were made certain to be centered.
6. The motor was started and run for 10 s.
7. If welding does not occur on the check run, then the test was repeat at the next higher load until welding is verified.

3.3.2 Four-Ball Wear Test (ASTM D-2266):

The aim of this test is to find the ability of metal surfaces to wear after rubbing one another, using lubricating grease in certain temperature and specific load.

3.3.2.1 Apparatus:

1. Four-ball wear tester as shown in Fig.3-2.
2. Microscope capable of measuring the diameters of the scars produced on the three stationary balls to an accuracy of 0.01 mm.

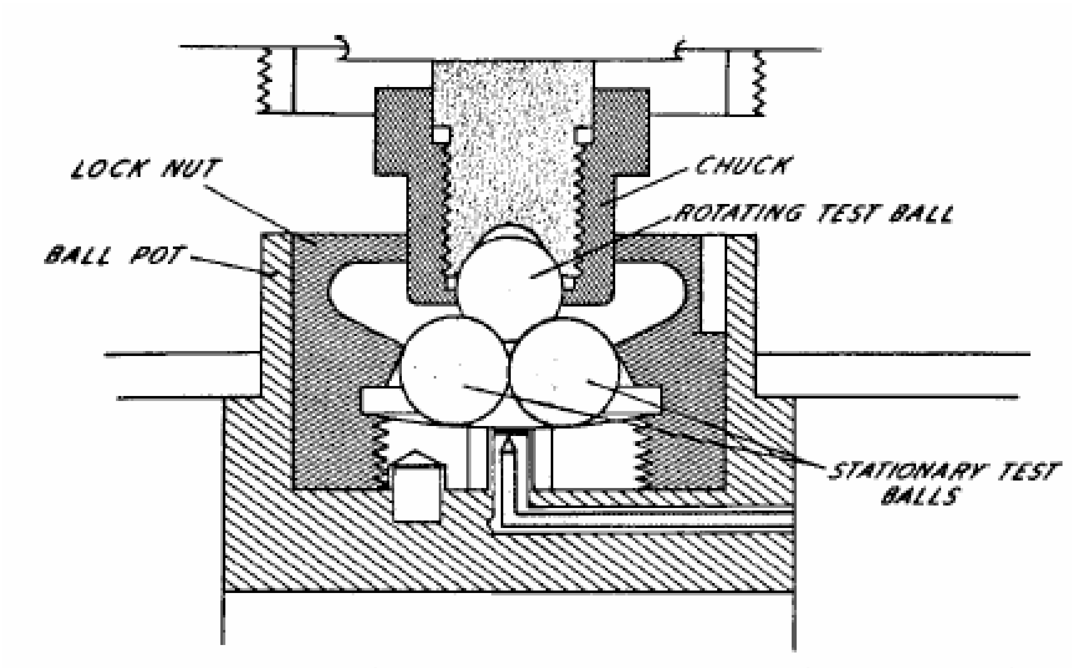


Figure 3-2 Falex corporation (Roxanna) four-ball wear test arrangement.

3.3.2.2 Procedure:

1. The four test balls, clamping parts for the upper and lower balls and the grease cup were thoroughly cleaned using a cleaning fluid (kerosene).
2. One of the clean test balls was inserted into the ball chuck. The ball chuck was inserted into spindle of the test machine and tightened according to the equipment manufacture's directions.
3. A small amount of the grease was placed in the ball cup sufficient to the fill the void space between the three balls to be inserted in the ball cup and the balls was locked in position by hand tightening the locknut into the ball cup using the wrench supplied by the equipment manufacture.
4. The test balls located in the ball chuck and ball cup were coated completely and thoroughly with the test grease and then the ball cup was filled with grease and leveled off with the top surface of the locknut.

5. The ball cup assembly containing the three test balls and grease specimen were placed on the test machine.
6. After reaching the desired test load, the temperature controller turned on and the controller was set to maintain 75 ± 2˚C.
7. When the desired temperature was reached, the timer and the drive motor were simultaneously started, previously set to 1200 ± 60 rpm.
8. After the drive motor has been on for 60 ± 1 min., the heaters and drive motor were turned off and the ball cup and three-ball assembly were removed.
9. The three lower balls were taken and the diameter of the affected areas caused by friction was measured by using the provided microscope. The result was multiplied by 0.05.

3.3.3 Detection of Copper Corrosion Test (ASTM D-4048):

This test method covers the detection of the corrosiveness to copper of lubricating grease.

3.3.3.1 Apparatus:

1. Beaker: tall form, made of glass and of 100 ml capacity.
2. Copper strip: commercial cold-rolled strip not less than 0.5 mm thickness, cut into pieces 75 mm long 13 mm wide.
3. Oven.

Figure 3-3 Instrument for copper strip corrosion measurement.

3.3.3.2 Procedure:

1. The beaker was filled with the sample to within 6 mm of the top and, with the aid of spatula, the surface was leveled and smoothed. A freshly prepared copper strip was coated with thin film of the sample for 48 mm from one end and that end vertically inserted to a depth of 50 mm into the sample contained in the beaker. The surface of the sample was pressed into contact with the strip and leveled again with the spatula. The beaker and its contents were placed in an oven controlled to the test temperature.
2. When the specified period of time has elapsed, the beaker was removed from the oven and allowed to cool to room temperature. The copper strip was withdrawn, wiped with cotton wool, and washed with kerosene. The procedure described above was repeated of each test was obtained.

3. The corrosiveness was reported in accordance with one of the classifications listed in Table 3-6.

Table 3-6 Copper strip classifications

Classification	Designation	Description[A]
Freshly polished strip[B]		
1	Slight tarnish	a. Light orange, almost the same as freshly polished strip. b. Dark orange.
2	Moderate tarnish	a. Claret red. b. Lavender. c. Multicolored with lavender blue or silver, or both, overlaid on claret red. d. Silvery. e. Brassy or gold.
3	Dark tarnish	a. Magenta overcast on brassy strip. b. Multicolored with red and green showing (peacock), but no gray.
4	corrosion	a. Transparent black, dark gray or brown with peacock green

		barely showing. b. Graphite or lusterless black. c. Glossy or jet black.

[A] The Copper Strip Corrosion Standard is a colored reproduction of strips Characteristic of these descriptions.

[B] The freshly polished strip is included in the series only as an indication of the appearance of a properly polished strip before a test run; it is not possible to duplicate this appearance after a test, even with a completely non corrosive sample.

3.3.4 Dropping Point Test (ASTM D-2265):

This test covers the determination of the dropping point of lubricating grease; this point is being the temperature at which the first drop of material falls from the cup. So the dropping is the temperature, at which the grease passes from a semisolid to a liquid state, under the conditions of the test.

3.3.4.1 Apparatus:

Dropping point assembly manufactured by KOEHLER instrument, New York, as shown in Fig. 3-4, consisting of the following:

1. Grease cup: chromium-plated brass cup.
2. Test tube: thin walled, soft glass test tube with rim.
3. Support ring.
4. Thermometer: maximum working temperature up to 300 ^{o}C with temperature controller.
5. Aluminum block oven.

Figure 3-4 Apparatus for drop point measurement.

3.3.4.2 Procedure:

1. The grease cup was filled with lubricating grease by pressing into the grease to be tested then excess grease was removed and flushed with the top edge of the cup with a spatula.
2. A hollow space was made in the lubricating grease such that light can be seen all the way through.
3. The support ring was placed down the cup to monitor the formation of the drop.
4. The grease cup was placed in a test tube.
5. A thermometer was inserted inside the grease cup.
6. Then the assembly was put in the oven.
7. The oven was turned on and the fall of grease drop was monitored.
8. The temperature at which the first drop of grease falls from the cup was recorded.

3.3.5 Worked Penetration Test (ASTM D-217):

The test method covers the procedure for measuring the consistency of lubricating grease by penetration of the standard cone.

3.3.5.1 Apparatus:

The apparatus was manufactured by the Normal Analis Company, France as shown in Fig. 3-5.

1. Penetrometer: the instrument shall be capable of indicating depth in tenths of a millimetere.
2. Standard penetrometer cone: is suitable for all penetrations.
3. Grease worker: comprising a grease cup, cover, and plunger assembly constructed for either manual or mechanical operation.
4. Water bath: capable of controlling the bath temperature at 25±0.5 $^{\circ}$C and designed to bring the assembled grease worker to test temperature conveniently.

Figure 3-5 Apparatus used for penetration measurement.

3.3.5.2 Procedure:

1. Sufficient specimen was transferred to the cup of the clean grease worker to fill it heaping full; avoiding the inclusion of air by packing with the spatula. The cup was jarred from time to time as its being packed to remove any air inadvertently entrapped.
2. The assemble worker was placed in the water bath maintained at 25 $^{\circ}$C for 1 hr.
3. The worker was removed from the bath then the grease was subjected to 60 full double strokes of the plunger, completed in about 1 min, and the plunger , the cup was returned as much of the grease cling to the plunger as can readily be removed.
4. The cup was placed on the penetrometer table, making certain that it cannot teeter.
5. The cone shaft rapidly released, and allowed to drop for 5.0 ± 0.1 s.
6. The penetration was read and recorded from the indicator.

Chapter Four

Results and Discussions

4.1 Introduction:

In this chapter, the results of using HiTEC 343 extreme pressure additive were discussed, as well as its impact on bearing pressure for the four greases used in this study. Wear resistance, drop point and worked penetration were also considered.

The effect of other additives, such as graphite, molybdenum disulfide and carbon black on grease properties were discussed in order to show which one of them is best to use from the point of lubrication, availability and cost.

On the other hand, the effect of adding corrosion inhibitor on the four types of greases, the best adding percentage at which to get the best results, i.e, to prevent corrosion of the copper strip were also discussed.

Finally, adding base stock 40, 60 and 150 were discussed and there impact on bearing pressure, wear resistance, drop point and worked penetration.

4.2 Effect of Extreme Pressure (EP) Additive:

A HiTEC 343 extreme pressure additive was selected in the present study, because of importance of adding good properties of load carrying to the lubricating grease, so it is extensively used in Al- Daura Refinery. It is a chemical additive.

Figure 4-1 shows the relation of extreme pressure additive percent on four-ball welding test at different types of grease. All data regarding the test carried out in this work are shown in appendix. The colors used in plotting the graphs are similar to the colors of the greases.

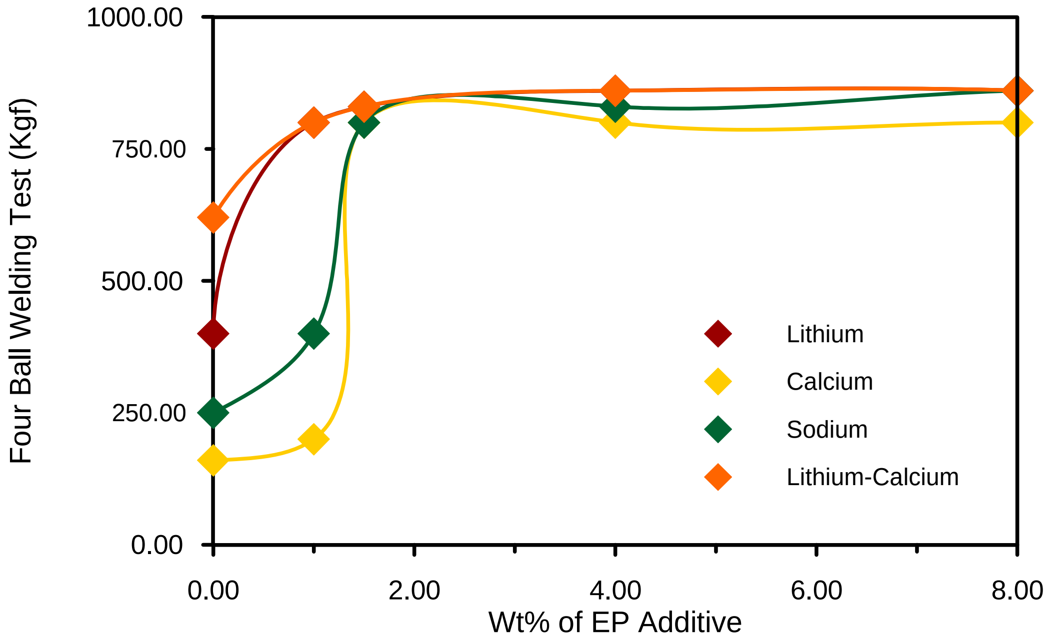


Figure 4-1 Effect of EP additive on four ball welding test at various types of greases.

From this Fig. it has been found that when adding 1% of this additive to lithium grease an extreme pressure characteristic was attained, for the grease is originally of good characteristics. A load of 800 Kg_f was attained. After 1% of extreme pressure additive was added, the four-ball welding did not show any change, when calcium grease was used, a little change in extreme pressure characteristics was noticed at this percentage, but when adding 1.5% a remarkable change in characteristics was noticed. This means that, using 50% more additive than when lithium is used. This applies to sodium as well, where extreme pressure changed from 250 to 400 kg_f. Four-ball welding increased to 800 kg_f at 1.5%. After this percentage of addition, the load remained almost constant despite the change in weight percent of the additive.

On the other hand, when two greases were mixed, an improvement in extreme pressure characteristic was noticed as well as less quantity is added. Mixing two greases improves characteristics basically and increases load up to 620 Kg_f, and with 1% additive the load becomes 800 kg_f.

Figure 4-2 shows a comparison between the effect of various weight percent of additives on four-ball welding in lithium grease and lithium-calcium grease.

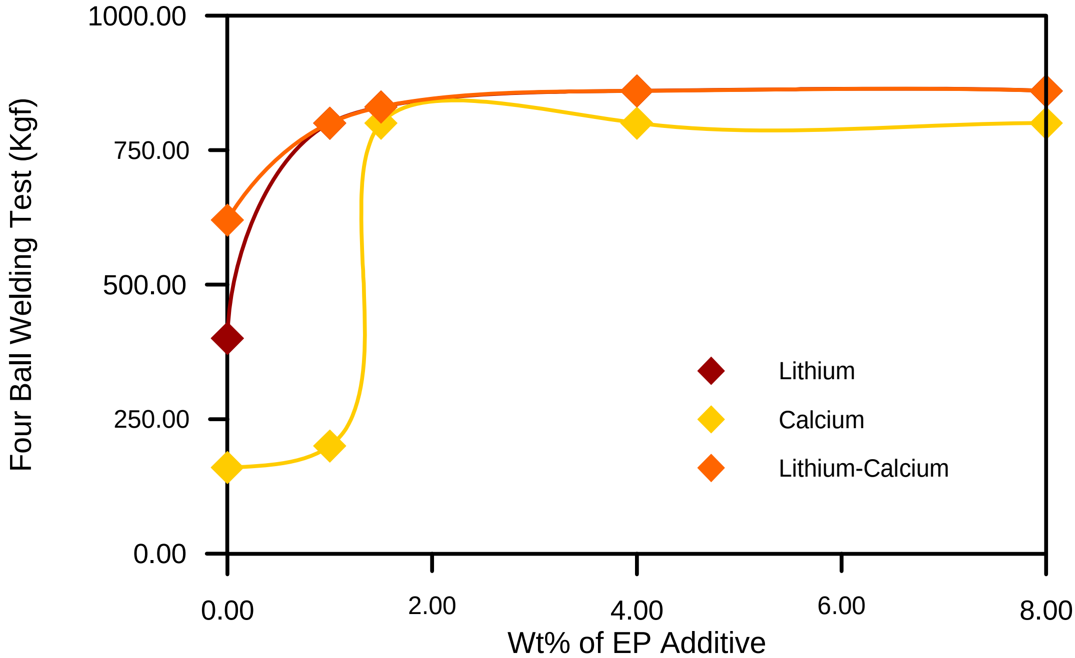


Figure 4-2 Comparison of EP additive effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

From the economic point of view, lithium grease is better than lithium-calcium grease because the latter needs mixing equipment, i.e., more power consumption and consequently higher cost.

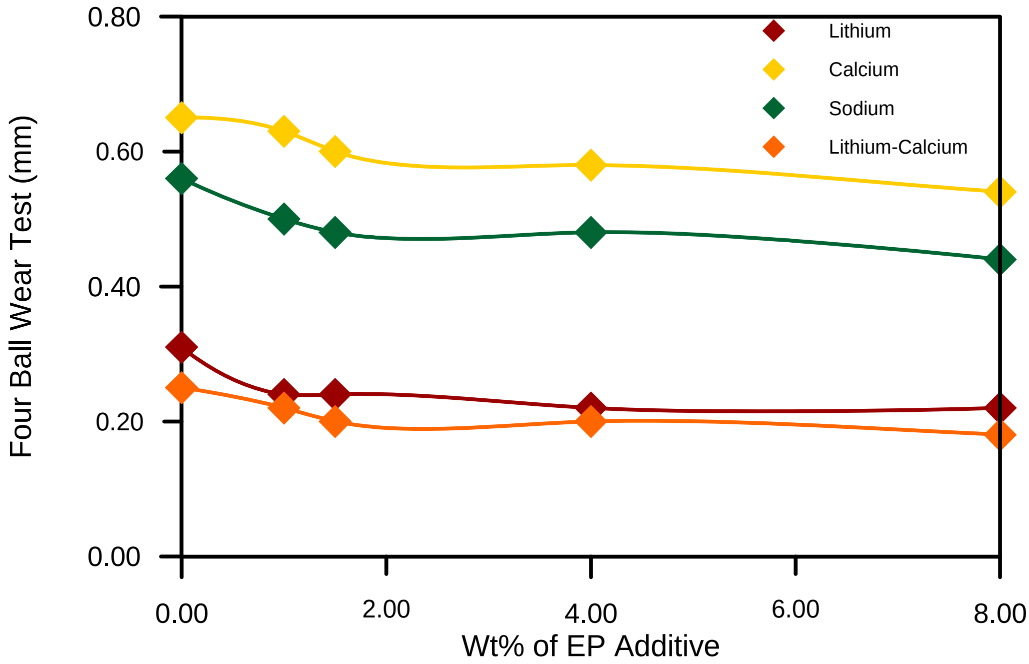


Figure 4-3 Effect of EP additive on four-ball wear test for various types of greases.

Figure 4-3 shows the effect of extreme pressure additive on wear test for different types of grease.

To reduce wear, a 1% of extreme pressure additive is added to lithium grease. This has more effect than that on any other grease, where the reduction was from 0.31 mm to 0.24 mm, i.e., 0.07 mm while, when calcium grease is used, the wear changed from 0.65 mm to 0.63 mm, i.e., 0.02 mm less. This means that calcium grease is less affected by this percent. In the sodium grease, wear is changed from 0.56 mm to 0.5 mm, i.e., 0.06 mm less.

In the mixture of lithium-calcium grease, the wear is changed from 0.25 mm to 0.22, i.e., 0.03 mm less.

Figure 4-4 shows a comparison between the effect of various weight percent of additives on wear in lithium grease and lithium-calcium grease.

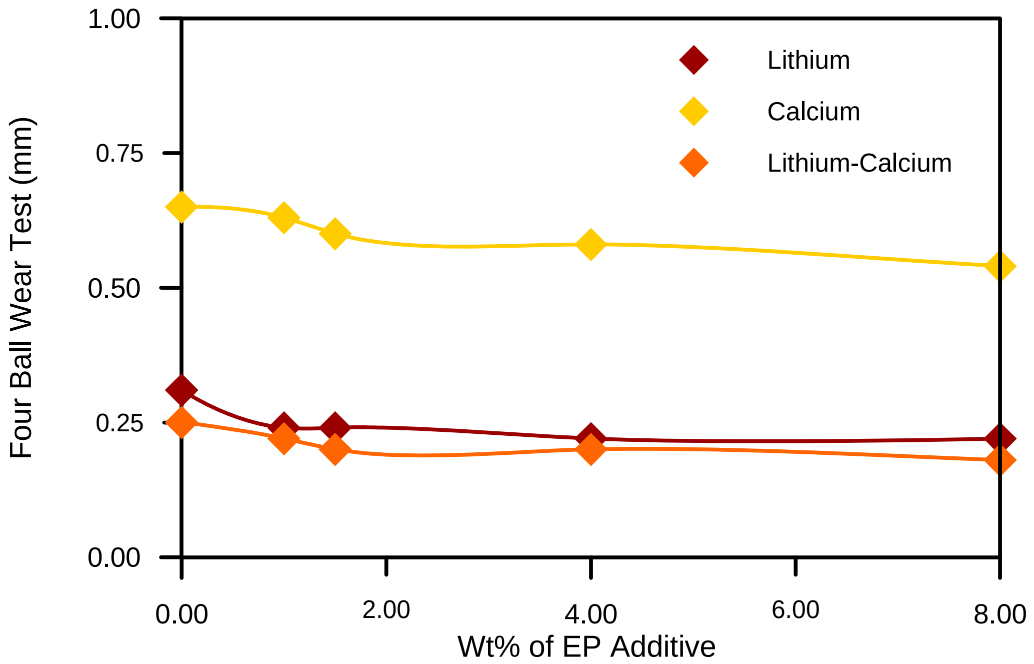
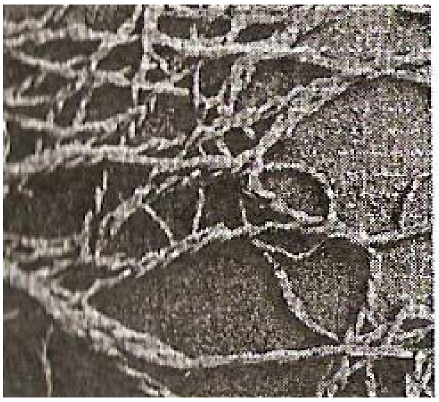


Figure 4-4 Comparison of EP additive effect on wear test in lithium, calcium and lithium-calcium grease.

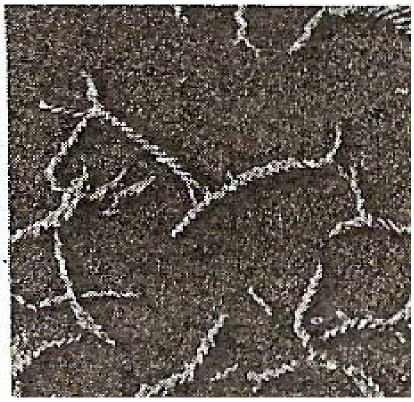
It can be recognized from Figure 4-4 that wear has been reduced slightly more by adding this additive to lithium-calcium grease than adding it to lithium grease alone. This slight reduction is not enough to make us abandon the use of lithium grease.

Figure 4-5 shows clearly that lithium grease retained a good texture for lubricating despite of the addition of which by showing a fixed drop point temperature higher than any other grease.

The drop point temperature remained constant despite the increase in additive in lithium grease. This is a good indicator that other characteristics remained unchanged in this type and the other types of greases, except for calcium grease where drop point decreased slightly because of soap fiber length that holds the structure of the grease, the calcium fiber length is small and thus affects the structure of the grease. Some of these additives may have a softening effect upon the lubricating grease. Therefore, calcium grease is considered a weak grease [34].



Lithium base



Calcium base

Figure 4-5 Fibers in lubricating greases [34].

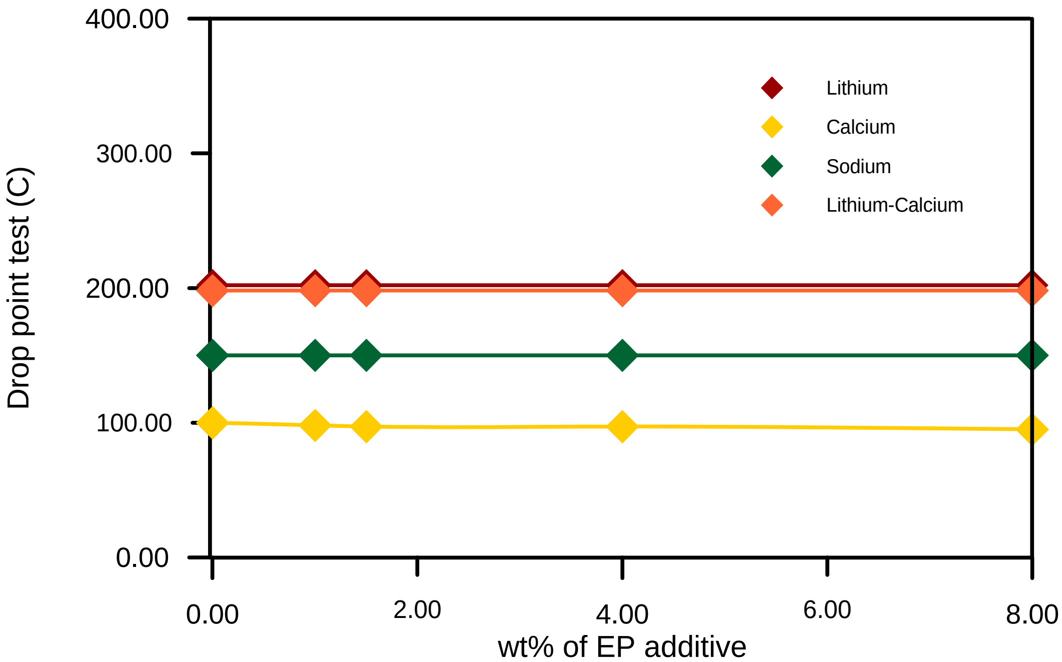


Figure 4-6 Effect of EP additive on drop point test at various types of greases.

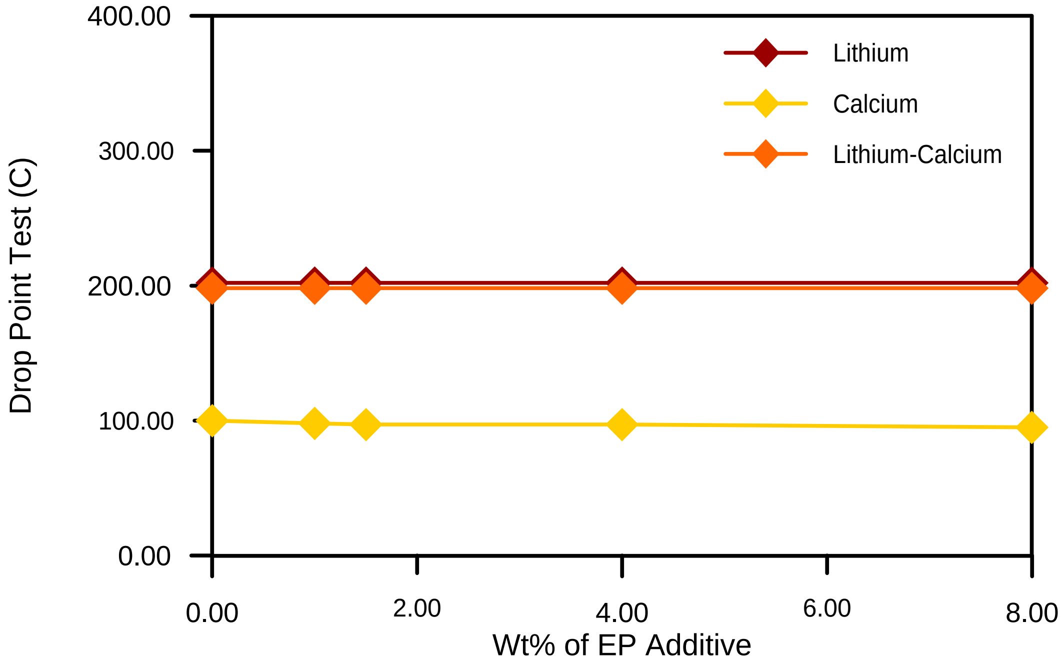


Figure 4-7 Comparison of EP additive effect on drop point test in lithium, calcium and lithium-calcium grease.

Figure 4-8 clarifies the effect of extreme pressure additive percent on worked penetration test for different types of grease.

The worked penetration was compared using different percentages of this additive. The worked penetration of lithium grease increased from 270 to 284.6 1/10mm, of calcium grease from 273.8 to 293.4 1/10mm, of sodium grease from 334.2 to 355.3 1/10mm, and of lithium-calcium grease increased from 271.5 to 279.5 1/10mm as shown in Figure 4-8 . The slight increase in worked penetration for all types of greases means that the basic texture of greases do not change significantly i.e., shear stability is maintained.

Figure 4-9 shows the comparison of extreme pressure additive effect on worked penetration test in lithium, calcium and lithium-calcium greases.

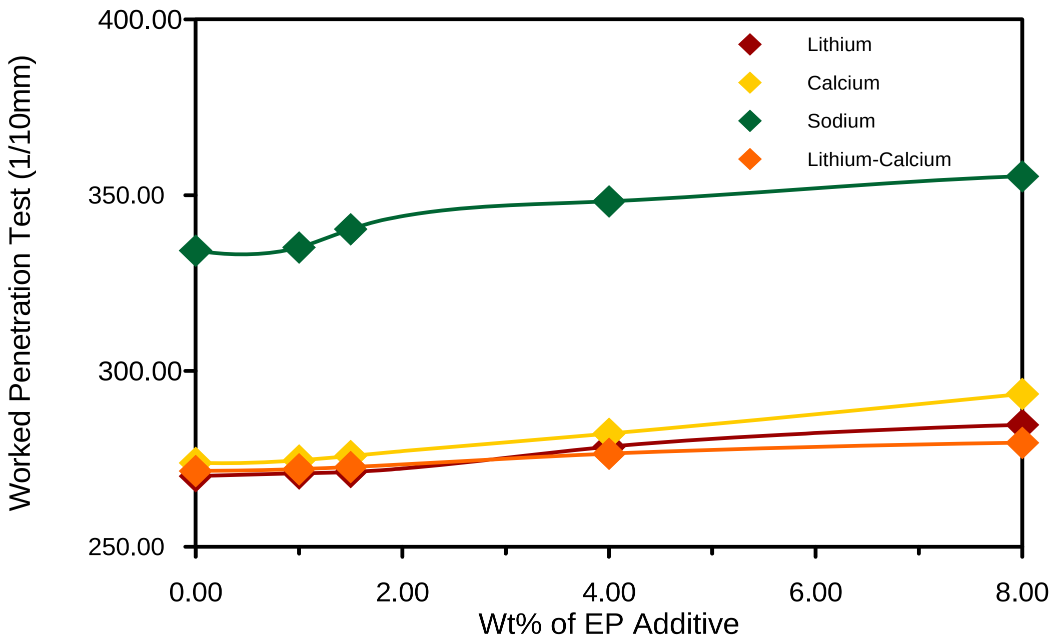


Figure 4-8 Effect of EP additive on worked penetration test at various types of greases.

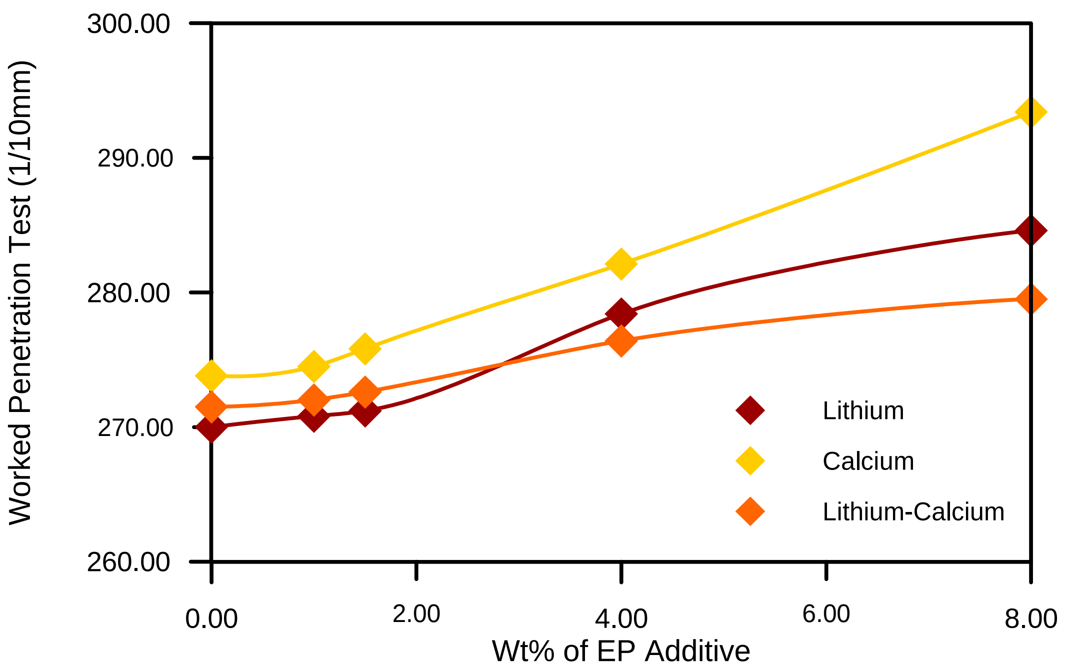


Figure 4-9 Comparison of EP additive effect on worked penetration test in lithium, calcium and lithium-calcium grease.

4.3 Effect of Graphite Additive:

Graphite is one of the most widely used fillers and that found applications in numerous types of lubricating greases [34]. It is widely used by a vast number of companies such as Aeroshell, Crown, and Al- Daura Refinery to produce extreme pressure grease. This additive is classified as a physical additive.

Fig. 4-10 clarifies the effect of graphite additive on four-ball welding for different types of grease, and Fig. 4-11 shows the comparison between lithium, calcium greases as separated greases and when mixed as a mixture of lithium-calcium grease.

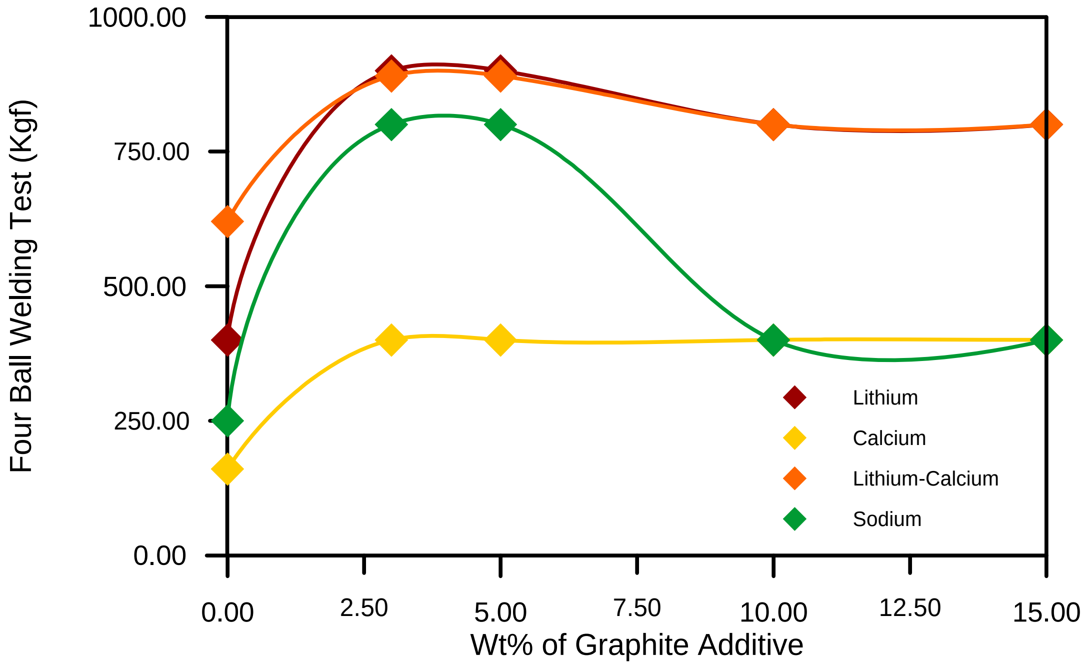


Figure 4-10 Effect of graphite on four-ball welding test.

In general, when graphite is added, the bearing load will increase. As shown in Fig. 4-10, the best additive percentage ranges between 3 - 5%, where the load increased from 400 kg_f to 800^+ kg_f in lithium grease, from 160 kg_f to 400 kg_f in calcium grease, from 250 kg_f to 800 kg_f in sodium grease and from 620 kg_f to 800^+ kg_f in lithium-calcium grease.

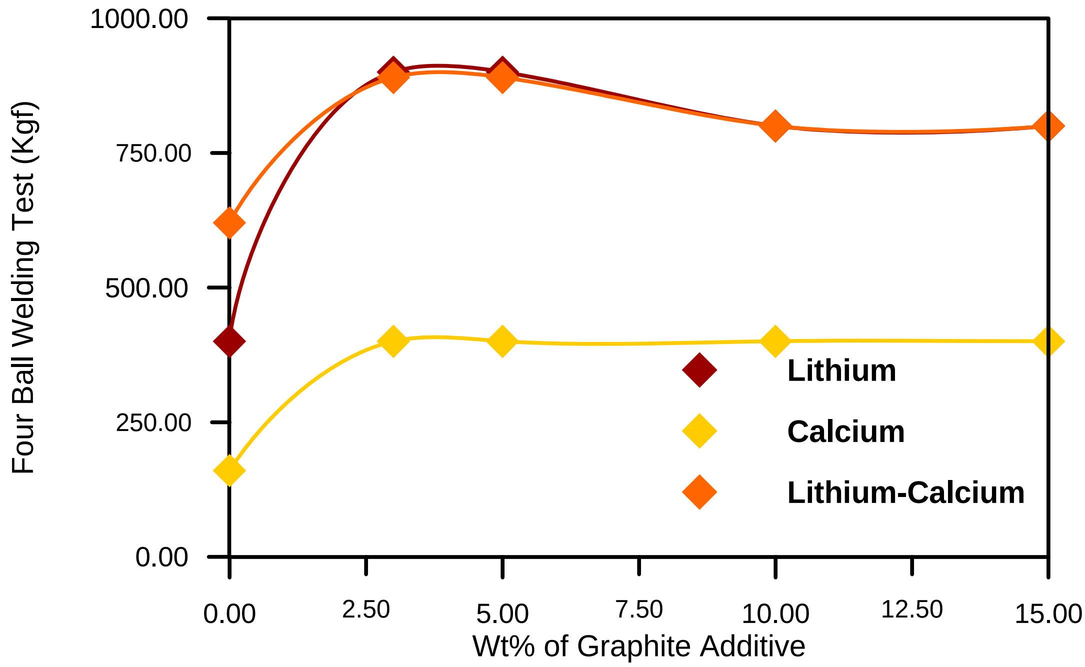


Figure 4-11 Comparison of graphite effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

When adding graphite, the wear has decreased in all kinds of greases as shown in Fig. 4-12. The best percentage of added graphite is 5% for lithium grease and 15% for calcium, sodium and lithium-calcium grease. From the economic point of view, the lithium grease is the most economical grease.

On the other hand, Fig. 4-13 shows the comparison of graphite additive effect on wear test in lithium, calcium and lithium-calcium grease. It has been noticed that the response of lithium grease to wear resistance additive is higher than that of the calcium grease. This proves that lithium grease is better. There was no big difference between lithium grease and lithium-calcium grease and thus lithium grease is better and there is no need to mix these two grease for wear resistance purposes.

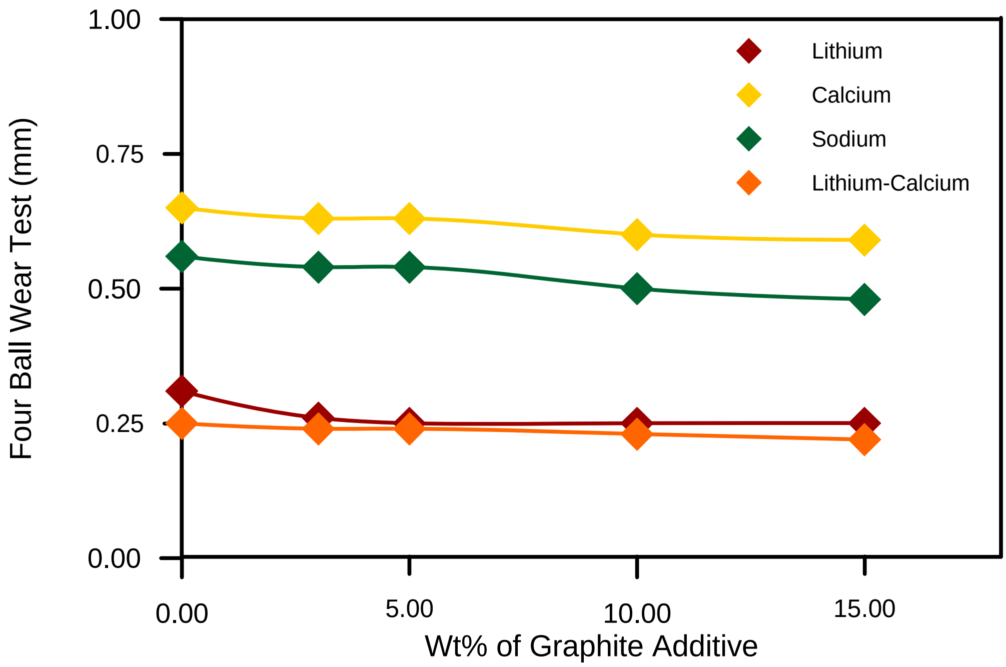


Figure 4-12 Effect of graphite additive on four-ball wear test.

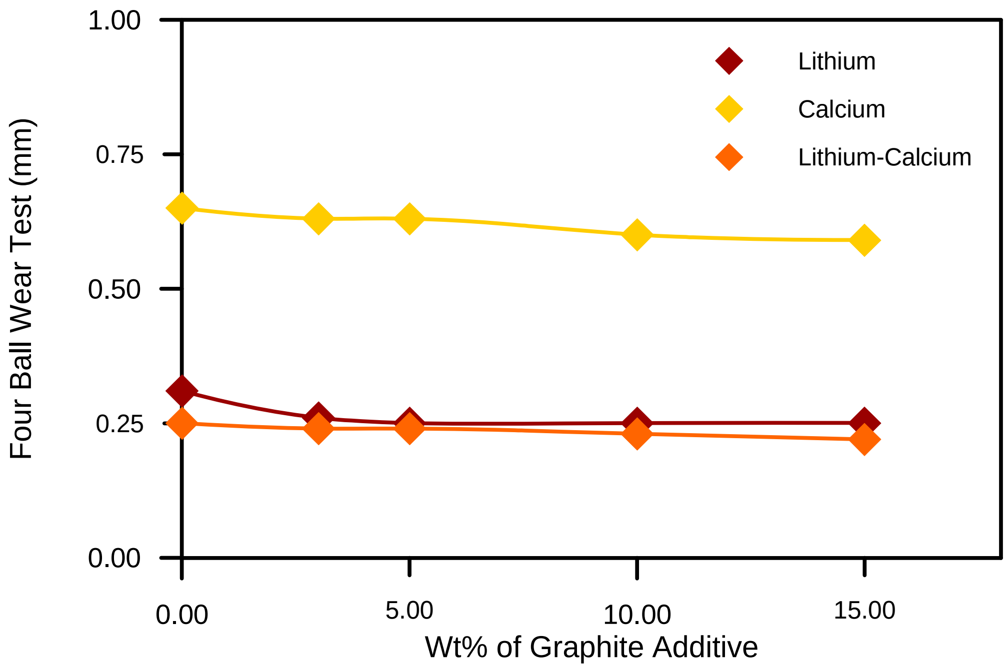


Figure 4-13 Comparison of graphite additive effect on wear test in lithium, calcium and lithium-calcium grease.

Fig. 4-14 demonstrates the effect of graphite additive on drop point test. The lithium grease has shown a highest drop point till the percentage of added graphite reached 15%, and so did the other greases, which means that adding graphite does not change the structure of grease.

Graphite can be added safely to all kinds of greases, as shown in figure 4-16. The work penetration has shown a little change when graphite is added, which emphasize that it is suitable for extreme pressure and works with all kinds of soap thickeners. When graphite is added to lubricating grease the filler has a little influence upon the consistency of the product. As the amount of filler is increased, the effect becomes evident [34].

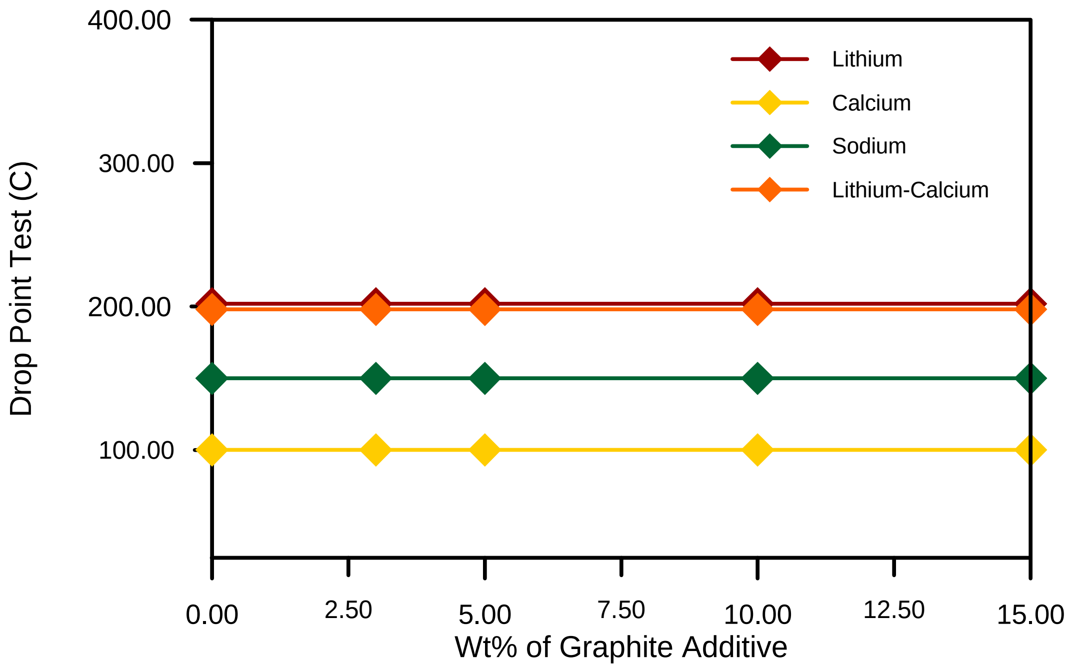


Figure 4-14 Effect of graphite percent of additive on drop point test.

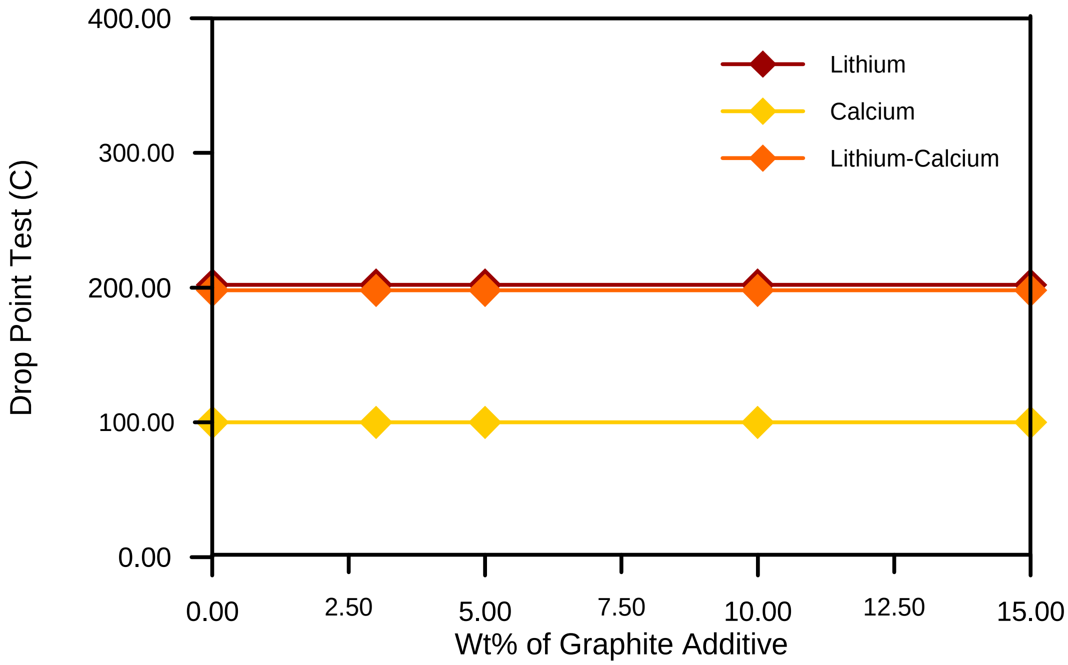


Figure 4-15 Comparison of graphite percent additive effect on drop point test in lithium, calcium and lithium-calcium grease.

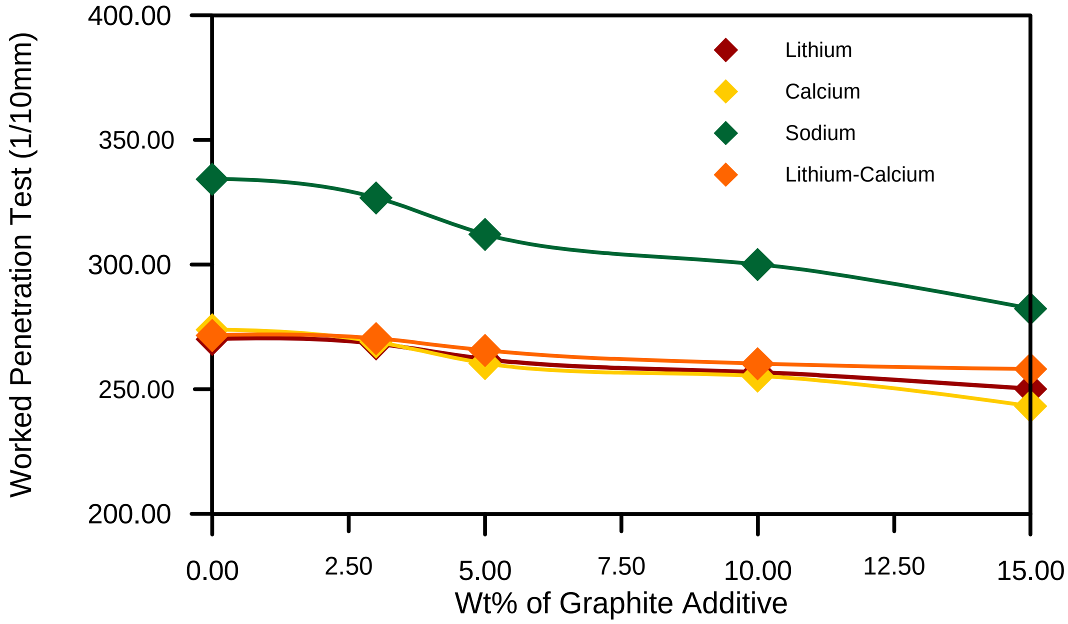


Figure 4-16 Effect of graphite additive on worked penetration test.

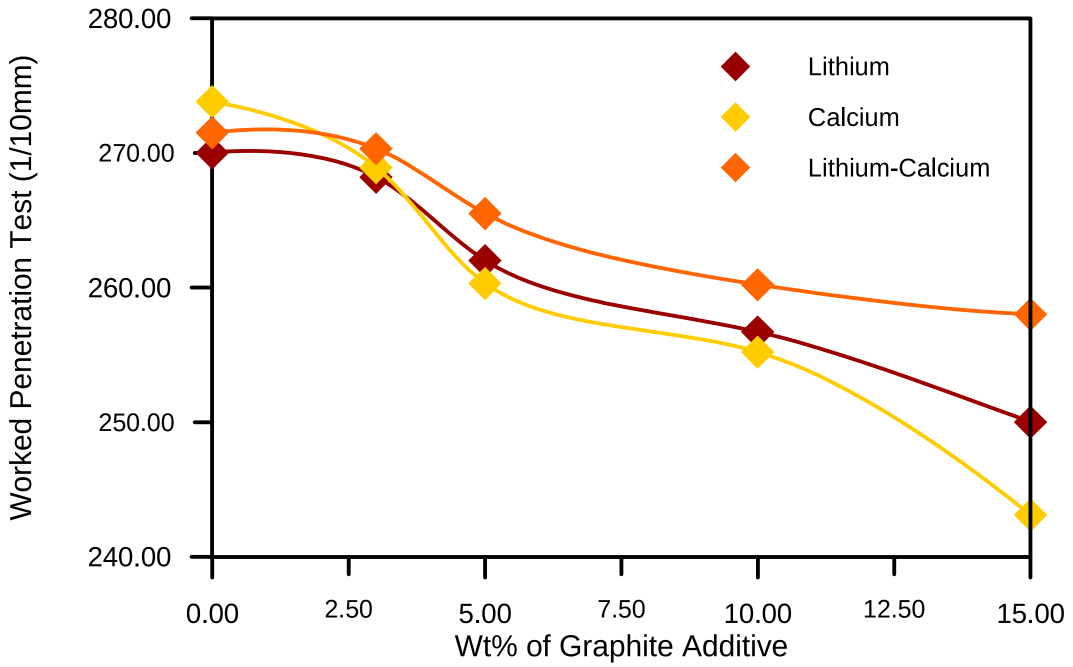


Figure 4-17 Comparison of various weight percent of additive effect on worked penetration test in lithium, calcium and lithium-calcium grease.

4.4 Effect of Molybdenum Disulfide Additive:

Molybdenum disulfide (MoS_2) can be classified as a chemical additive which is selected for study on the basis of its international use in commercial products in recent years by various companies such as Aeroshell, Enoc, Crown, Mobil, Lbrizol and Al-Daura Refinery. For suitable uses, the molybdenum disulfide should be not less than 99% pure. Thus, to improve grease characteristics, the molybdenum disulfide additive known for crystal structure (helps in sliding and lubricating) is selected for study.

Figure 4-18 shows the results of a variety of addition percentages on the four-ball welding test on greases.

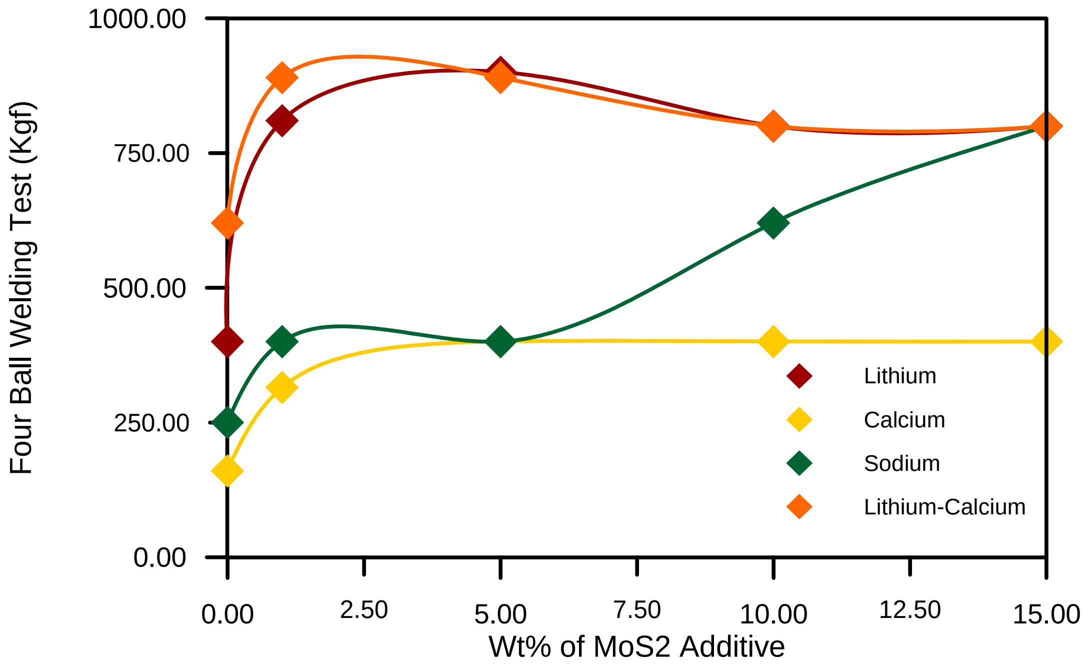


Figure 4-18 Effect of molybdenum disulfide percent additive on four-ball welding test.

It is noted that when adding different percentages of molybdenum disulfide (1-15 %) to lithium, calcium, sodium and lithium-calcium grease to become suitable to prevent seizure under conditions of high temperatures, heavy loading or extended periods of operation. Four-ball welding reading will increase at 1% molybdenum disulfide. It becomes more than 800 kg_f in lithium grease, 315 kg_f in calcium grease, 400 kg_f in sodium grease and 800^+ kg_f in lithium-calcium grease.

Figure 4-19 shows the comparison between the effect of various weight percent of molybdenum disulfide additives on four-ball welding in lithium grease and lithium-calcium grease.

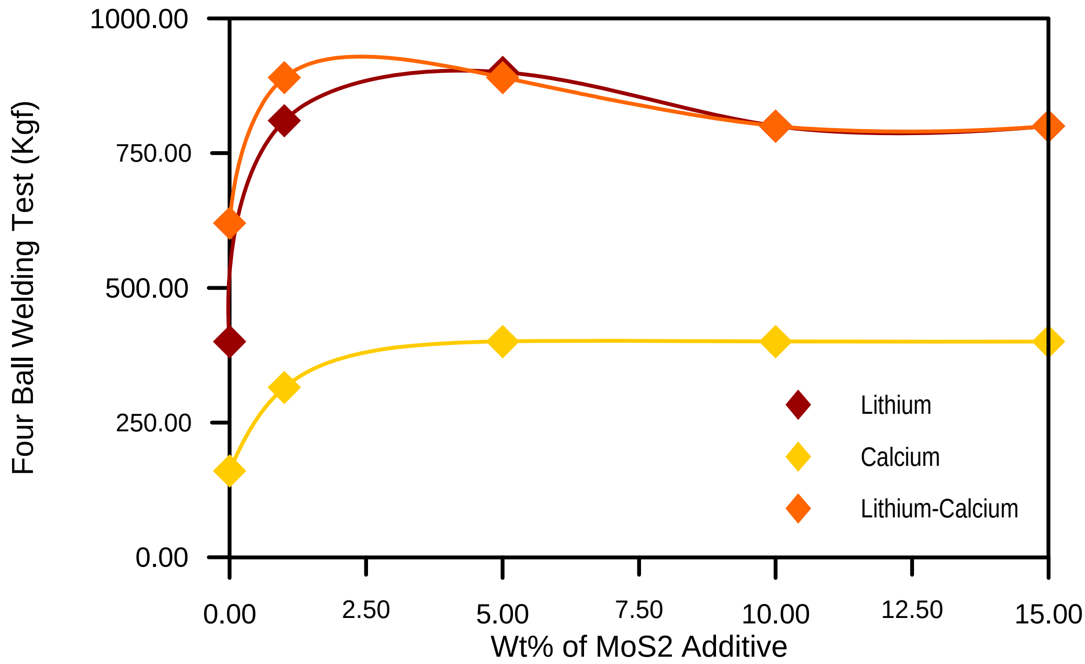


Figure 4-19 Comparison of molybdenum disulfide percent additive effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

The load carrying characteristic in molybdenum disulfide is higher than that of graphite. This is so because of their structure. These additives have a "layer lattice" structure in which the atoms in each layer or "basal plane" are located at the corners of regular hexagons. In graphite, Figure 4-20 the interatomic distance within the basal plane is 1.24 Ǻ. compared with a figure of 3.35 Ǻ for the interlayer separation.

Each "layer" in molybdenum disulfide consists of three planes–a central molybdenum core sandwiched between sheets of sulfur atoms Figure 4-21. The distance between the molybdenum atoms within a layer is 3.16 Ǻ compared with 3.08 Ǻ between the sulfur layers. The distance between adjacent layers of molybdenum atoms is 6.16 Ǻ [37].

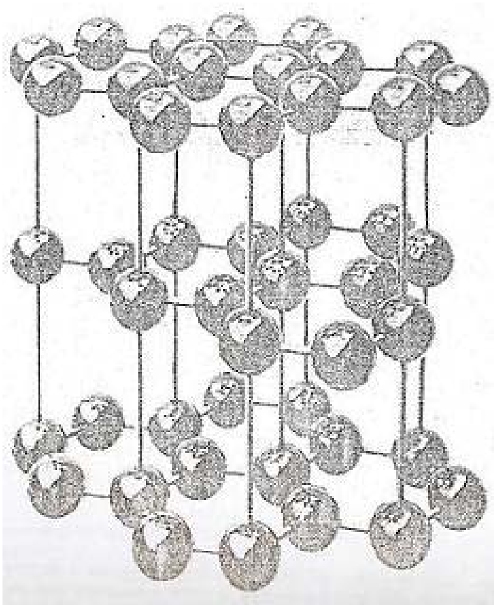


Figure 4-20 Graphite lattice (hexagonal form) [37].

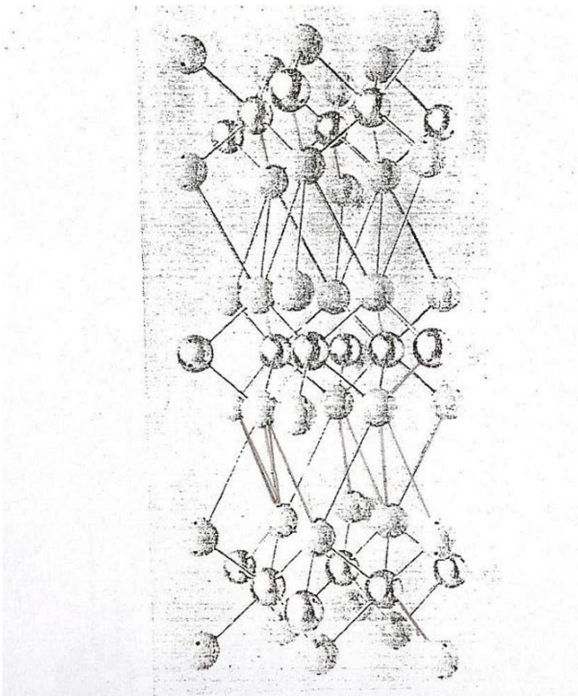


Figure 4-21 Molybdenum disulfide lattice [37].

Figure 4-22 clarifies the effect of MoS_2 on wear when different types of grease were used.

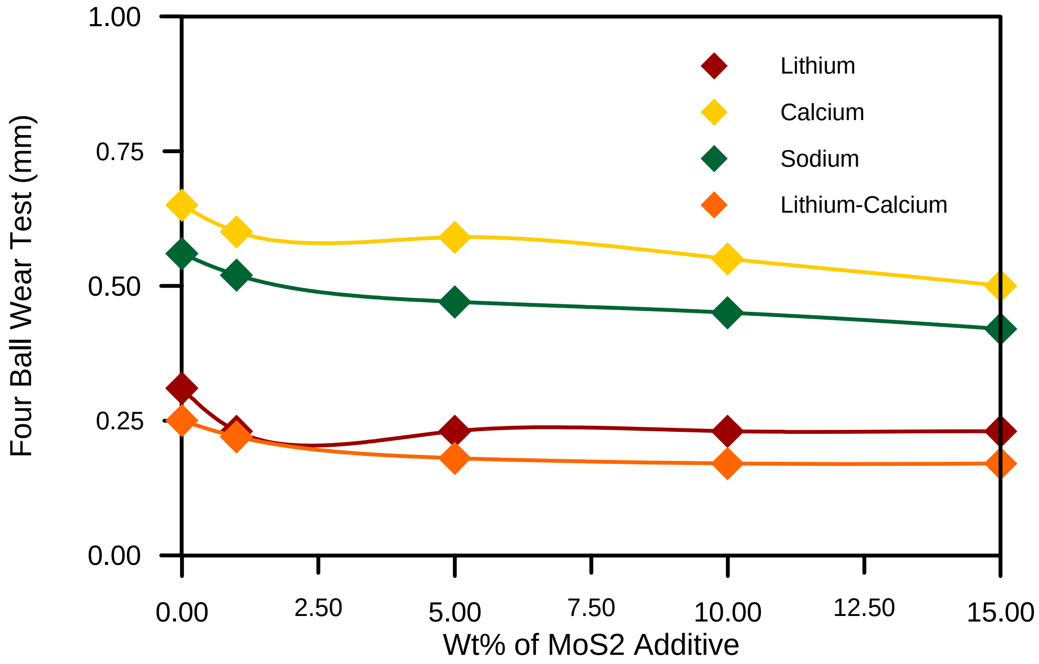


Figure 4-22 Effect of molybdenum disulfide percent additive on wear test.

It is found that wear has decreased when all types of greases were used. What makes molybdenum disulfide more economical than graphite is that it shows more efficient in wear test.

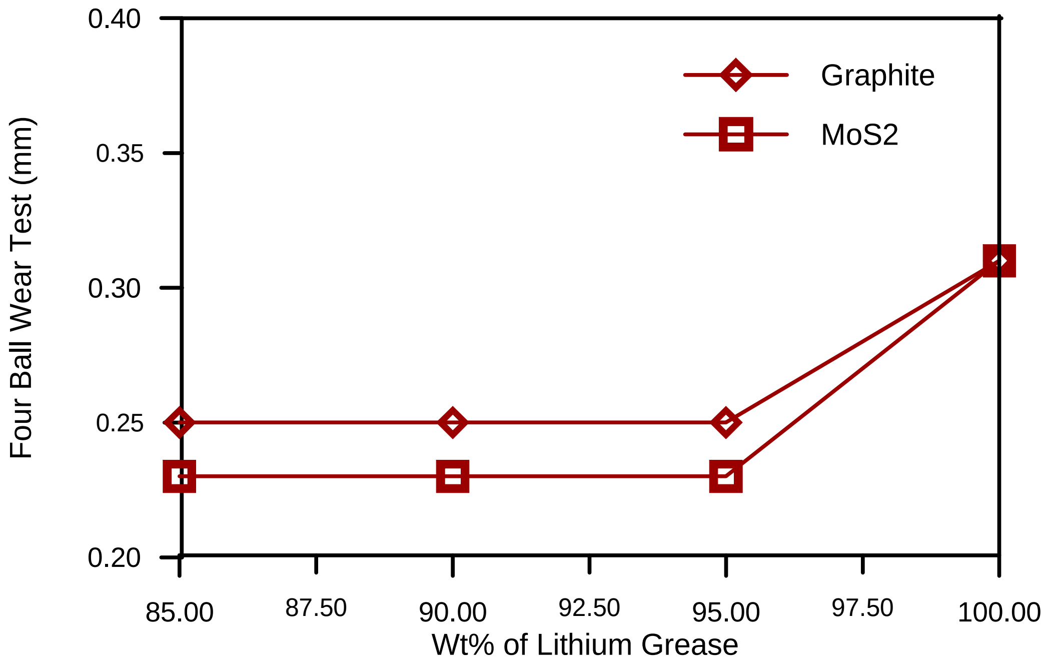


Figure 4-23 Effect of graphite and molybdenum disulfide on wear test in lithium grease.

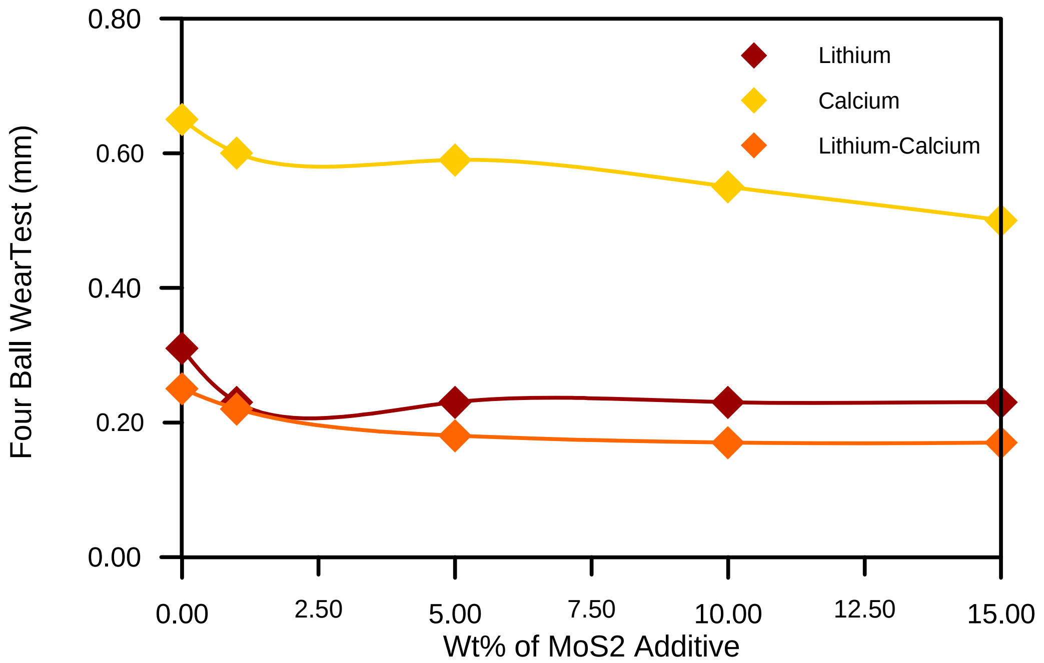


Figure 4-24 Comparison of molybdenum disulfide percent additive effect on four-ball wear test in lithium, calcium and lithium-calcium grease.

Figure 4-25 demonstrates the effect of molybdenum disulfide on drop point test. It is found that no change in drop point is taking place for all types of grease. This conclusion is in agreement with references [45,72]

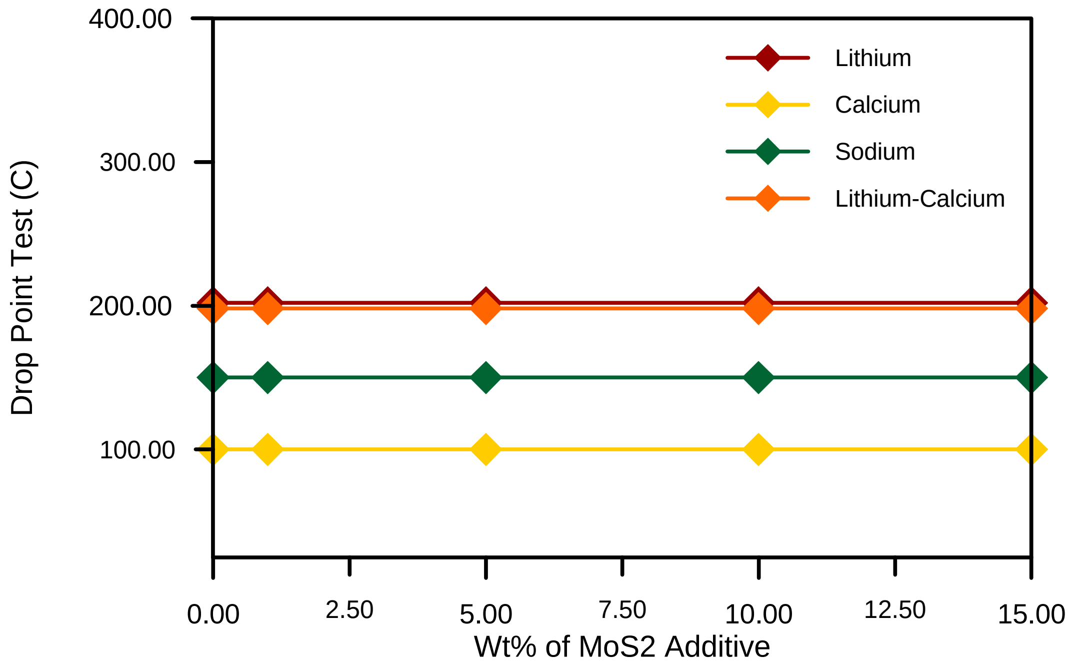


Figure 4-25 Effect of molybdenum disulfide on drop point test.

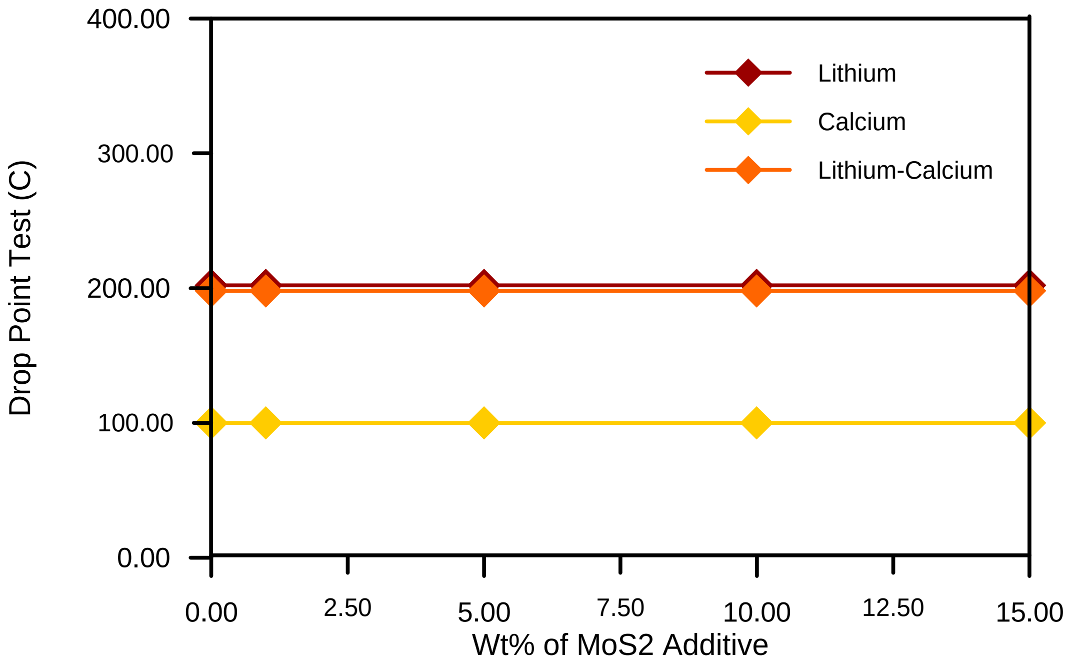


Figure 4-26 Comparison of molybdenum disulfide effect on drop point in lithium, calcium and lithium-calcium grease.

Figure 4-27 shows the effect of molybdenum disulfide on worked penetration. The results show the worked penetration was increased in general.

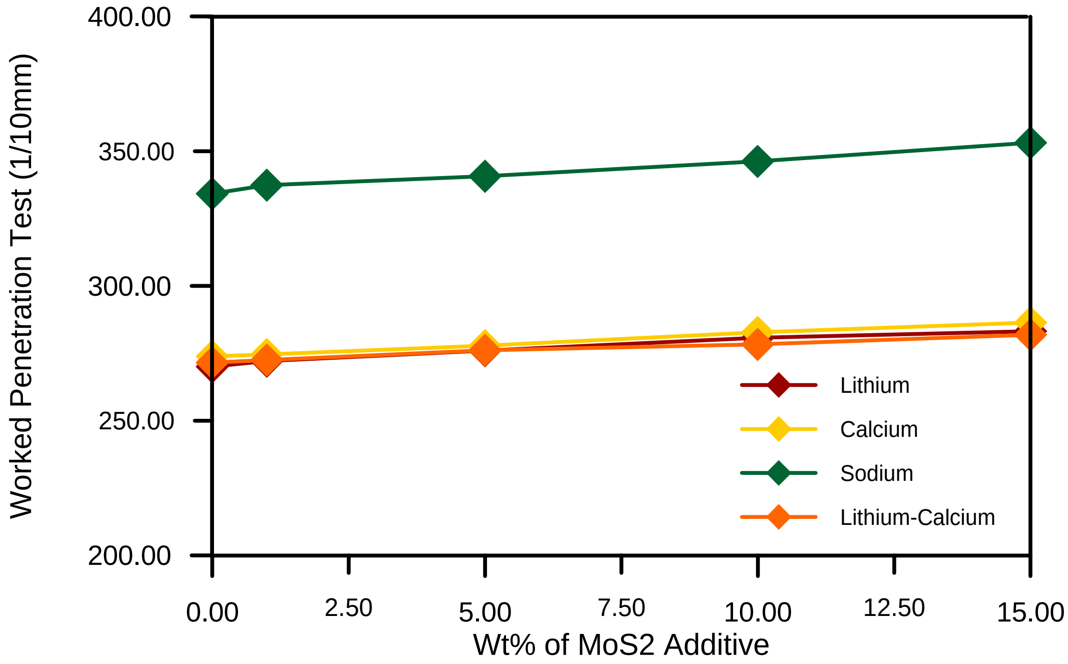


Figure 4-27 Effect of molybdenum disulfide on worked penetration test.

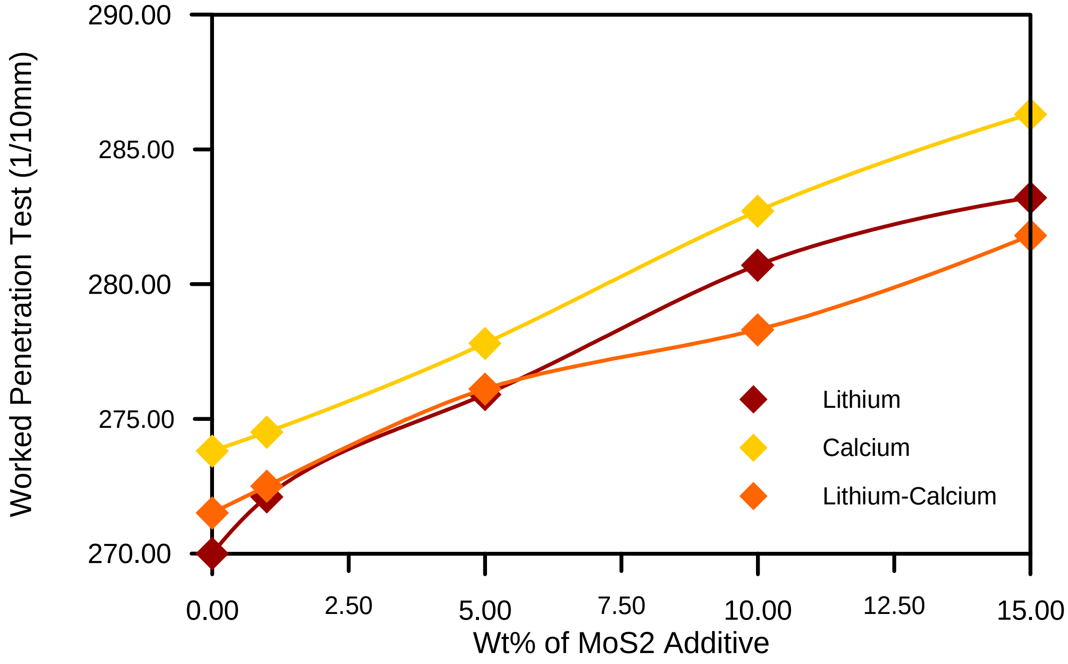


Figure 4-28 Comparison of molybdenum disulfide effect on worked penetration in lithium, calcium and lithium-calcium grease.

4.5 Effect of Carbon Black Additive:

Carbon black can be classified as a physical type of additive which is selected as alternative for molybdenum disulfide and graphite on the basis of availability and low cost as factors contributing in the extensive use of this additive.

Figures 4-29, 4-31, 4-33 and 4-35 show the effect of carbon black addition on four-ball welding, wear, drop point and worked penetration tests.

It is noticed that the best addition percentage ranges between (2.5-10%).

From the addition of carbon black in the selected range of percentages (2.5-10%) the following conclusions (phenomenon) were noted:

1. Maximizing the heavy load carrying, i.e. welding.
2. Minimizing the wear effect.
3. Increasing the dropping point.
4. A little effect upon consistency which showed that it is safe to be used.
5. It hardly changes the color.
6. No softening effect due to such addition.

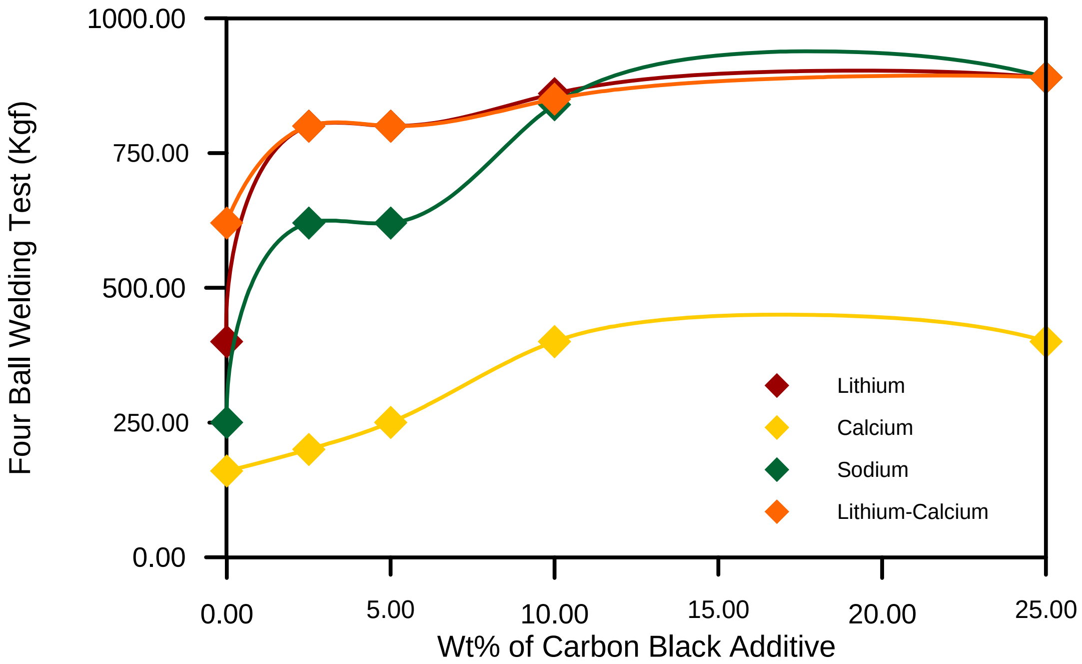


Figure 4-29 Effect of carbon black additive on four-ball welding test.

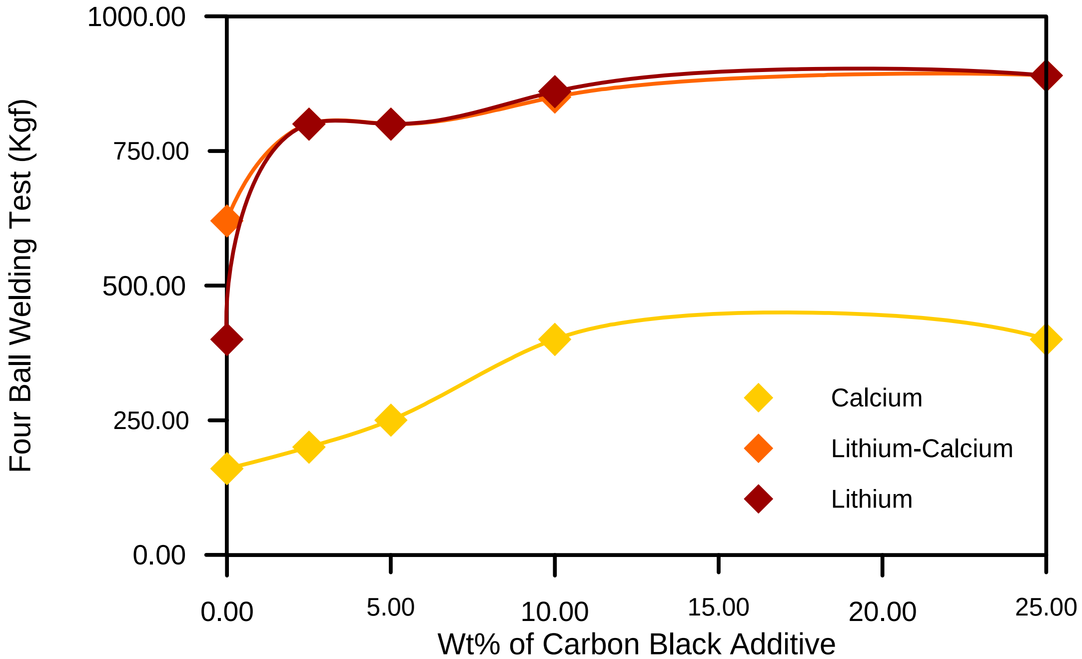


Figure 4-30 Comparison of carbon black additive effect on four-ball welding in lithium, calcium and lithium-calcium grease.

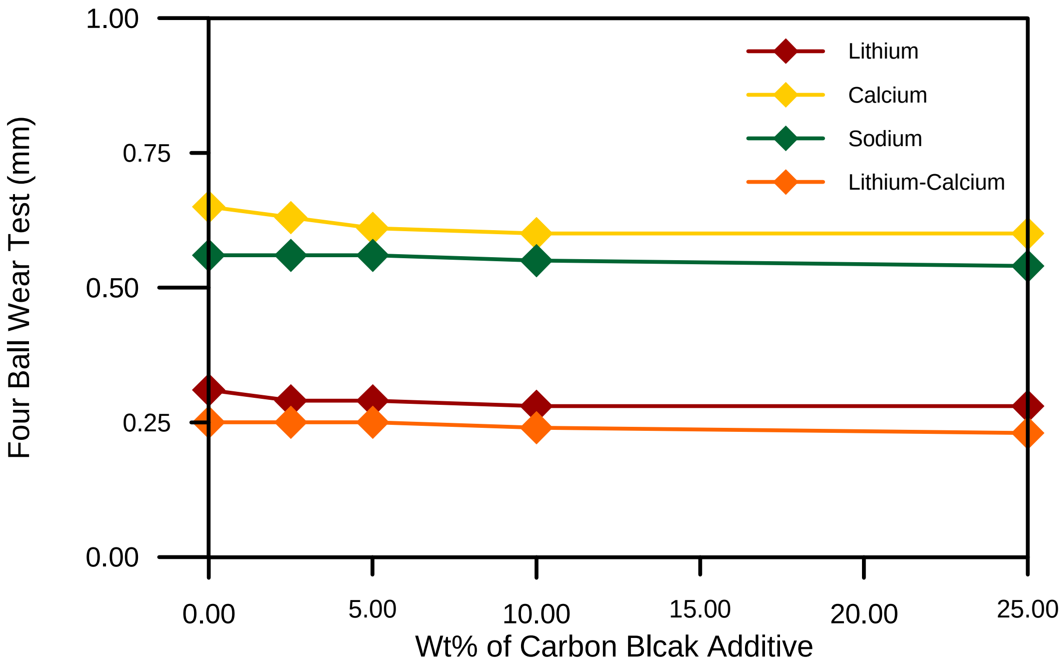


Figure 4-31 Effect of carbon black additive on four-ball wear test.

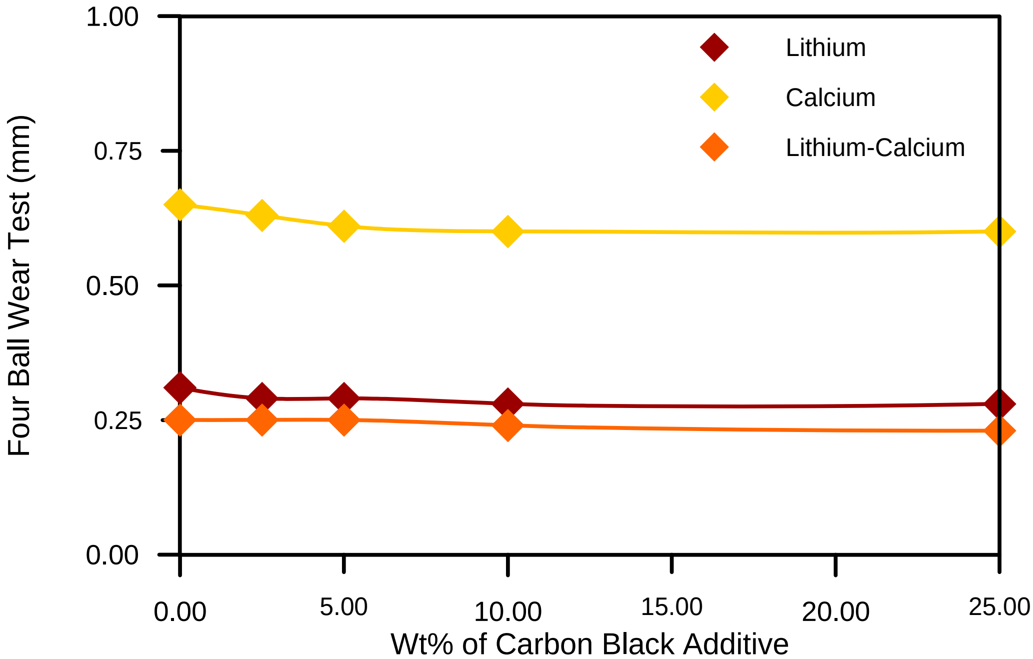


Figure 4-32 Comparison of carbon black additive effect on wear test in lithium, calcium and lithium-calcium grease.

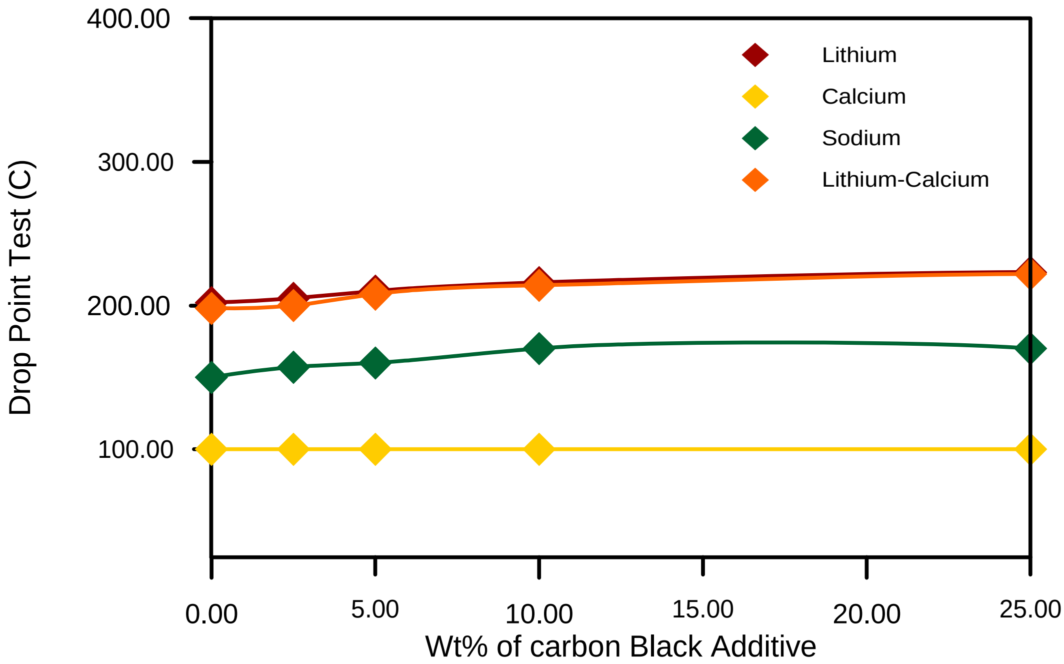


Figure 4-33 Effect of carbon black additive on drop point test.

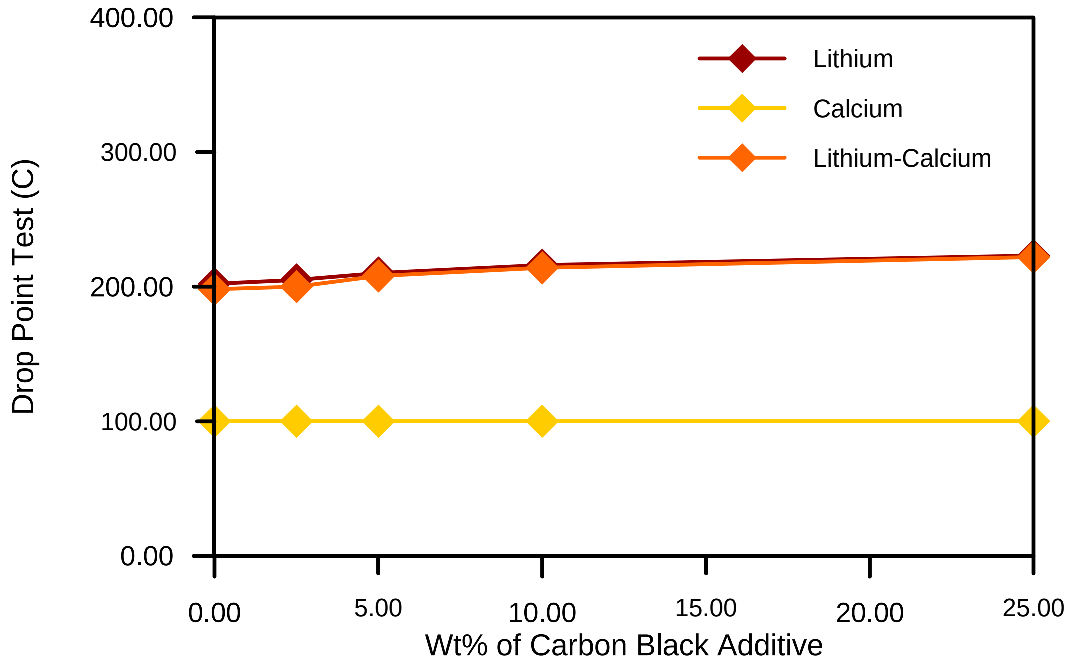


Figure 4-34 Comparison of carbon black additive effect on drop point in lithium, calcium and lithium-calcium grease.

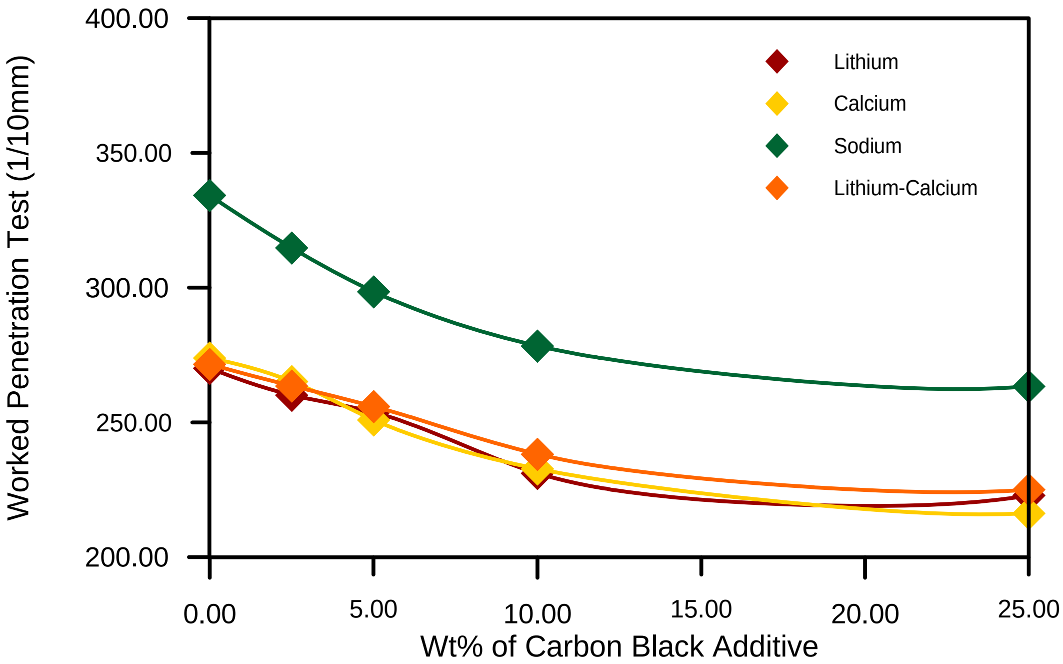


Figure 4-35 Effect of carbon black additive on worked penetration test.

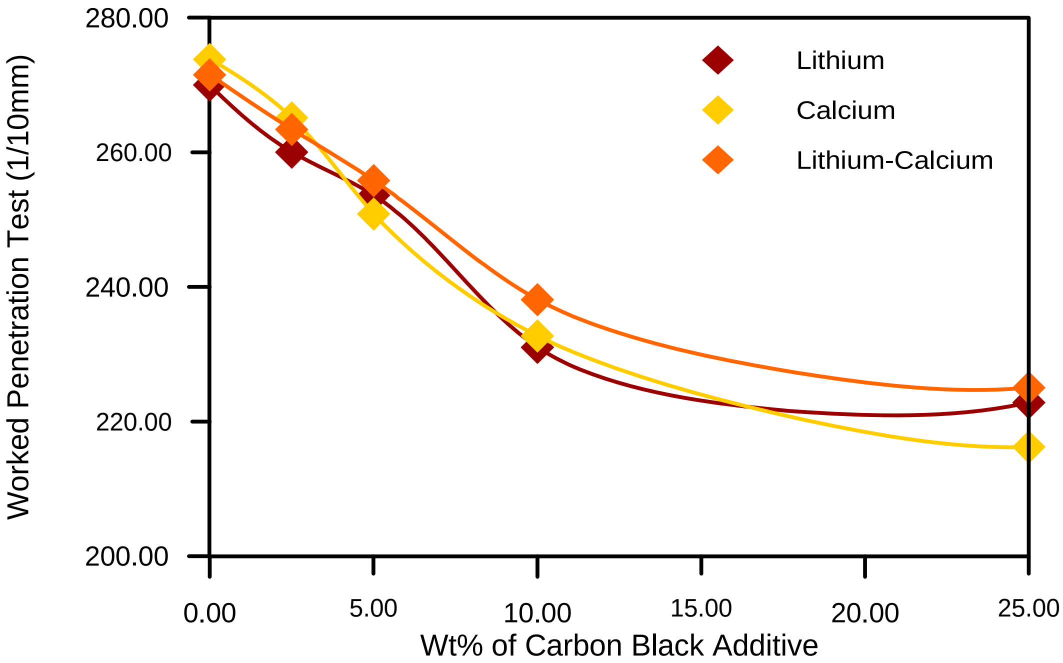


Figure 4-36 Comparison of carbon black additive effect on worked penetration in lithium, calcium and lithium-calcium grease.

4.6 Effect of Corrosion Inhibiter Additive:

One of the most important additives is the corrosion inhibitor. It can be classified as a chemical additive type. It is extensively used in Al-Dora Refinery and thus studied in this work. Its effect is measured by observing a cupper strip using a methodology set by ASTM. There are three cases to recognize the corrosion on this strip according to the blackening of the strip. To make sure that grease does not affect corrosion in machinery and equipment, corrosion inhibiter is added to prevent corrosion in a grease medium. By adding 3% of corrosion inhibitor to all types of grease the corrosion become (1a, namely slight tarnish), a bright strip is occurred, i.e., no corrosion is taking place as shown in Fig. 4-37. The effect of this corrosion inhibitor starts after adding 1% to lithium-calcium grease where the strip becomes bright, while calcium grease is basically, without additive, shows a bright strip. Since calcium base lubricating grease is water repellant it has been supposed that they do not require the addition of a corrosion inhibitor [34]. In the case of lithium grease, the effect started after adding 2%. In the case of sodium grease, the effect appears at 3%.

To insure that this additive does not have any negative effect on grease, the drop point and worked penetration is measured for each addition as shown in Figures 4-39, 4-41. Lithium, sodium, and lithium-calcium greases proved that the drop point dose not change at any percent of addition, while calcium showed a slight transition because its fiber length is short [34].

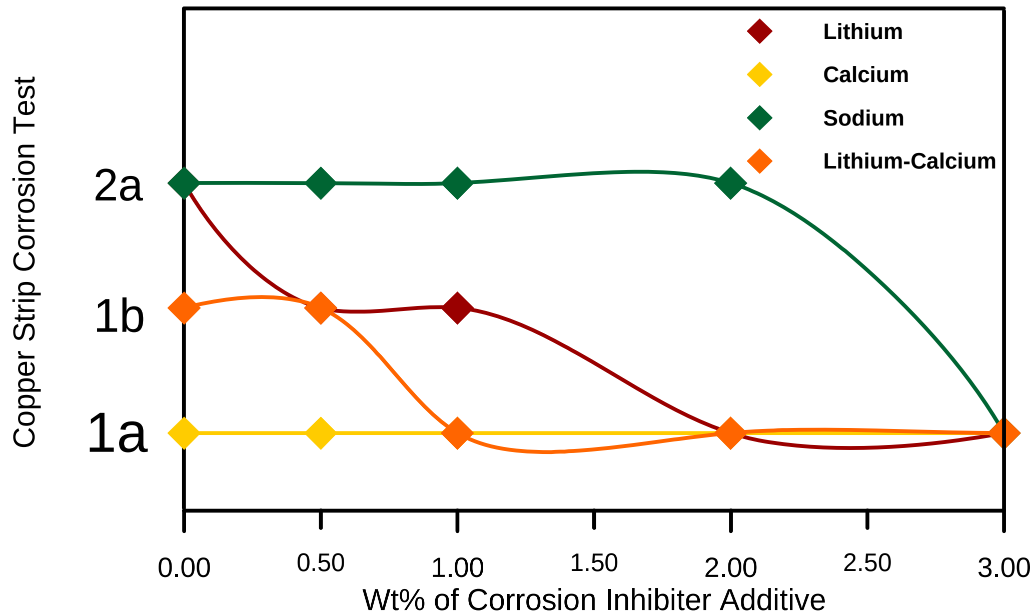


Figure 4-37 Effect of corrosion inhibiter additive on copper strip corrosion test.

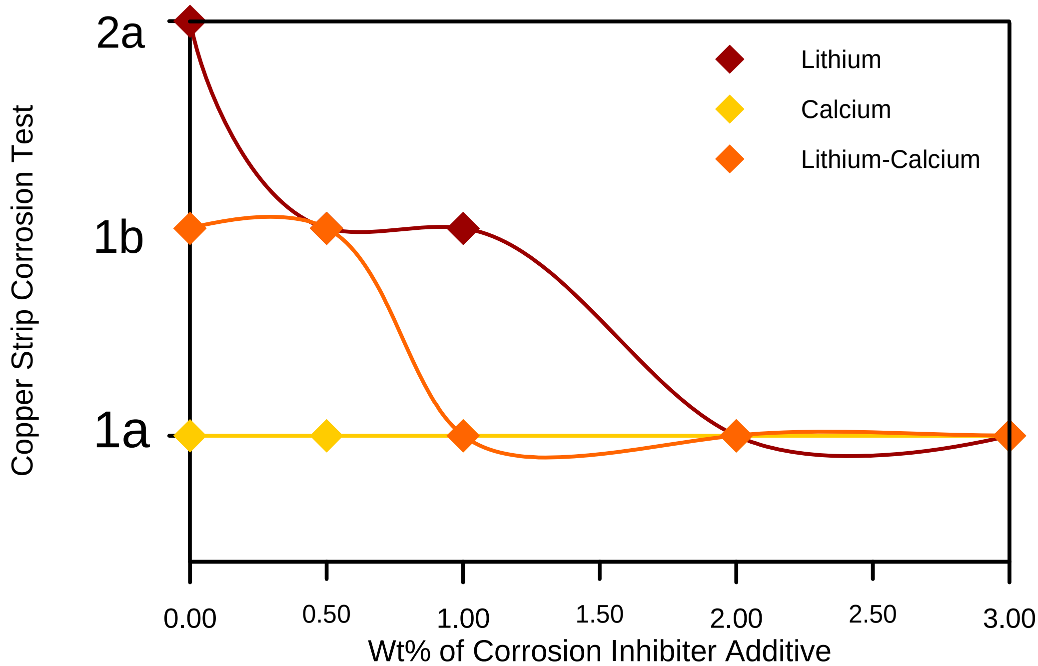


Figure 4-38 Comparison of corrosion inhibiter additive effect on copper strip corrosion test in lithium, calcium and lithium-calcium grease.

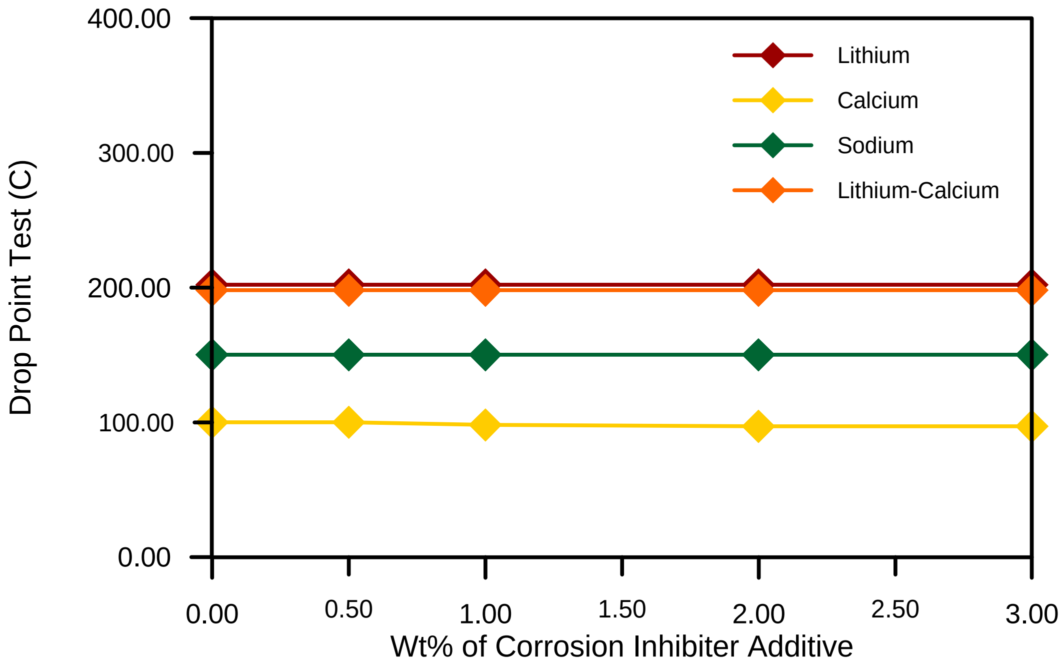


Figure 4-39 Effect of corrosion inhibiter additive on drop point test.

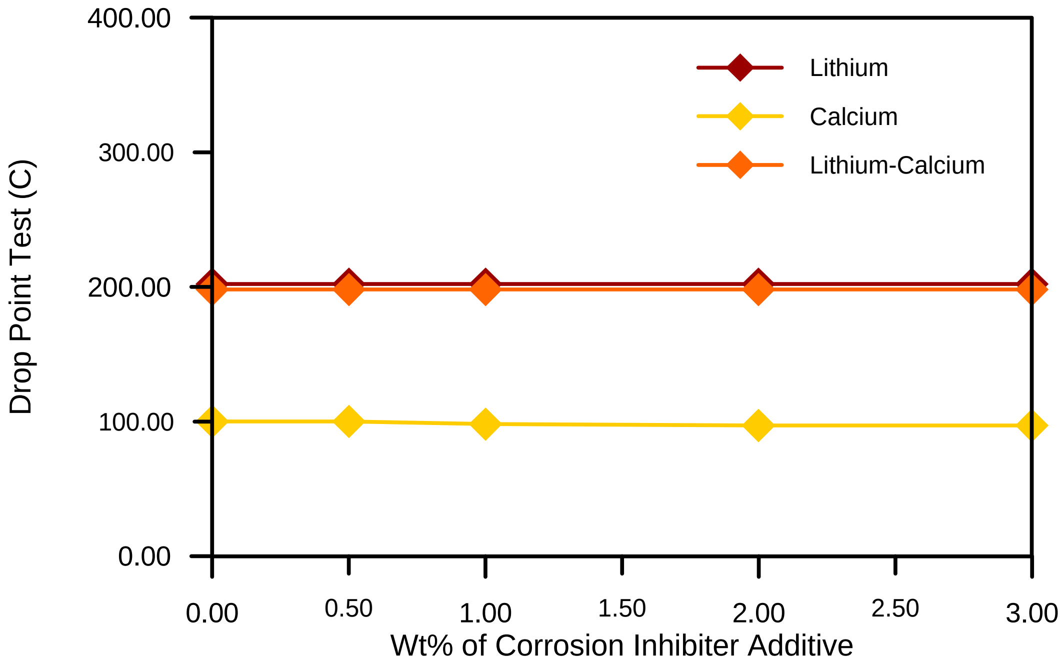


Figure 4-40 Comparison of corrosion inhibiter additive effect on drop point test in lithium, calcium and lithium-calcium grease.

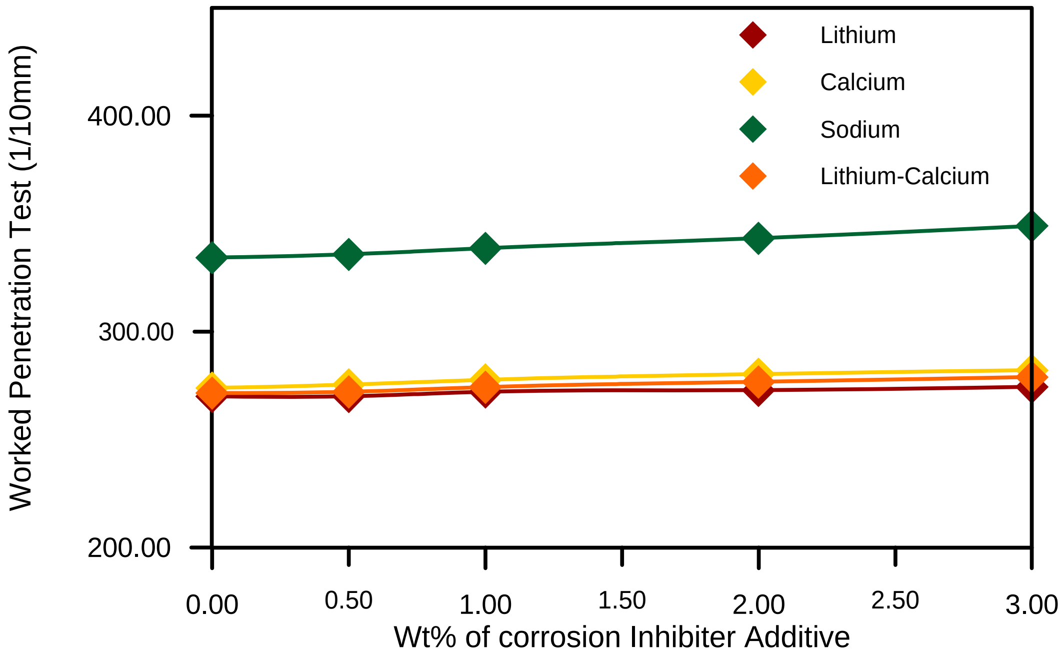


Figure 4-41 Effect of corrosion inhibiter additive on worked penetration test.

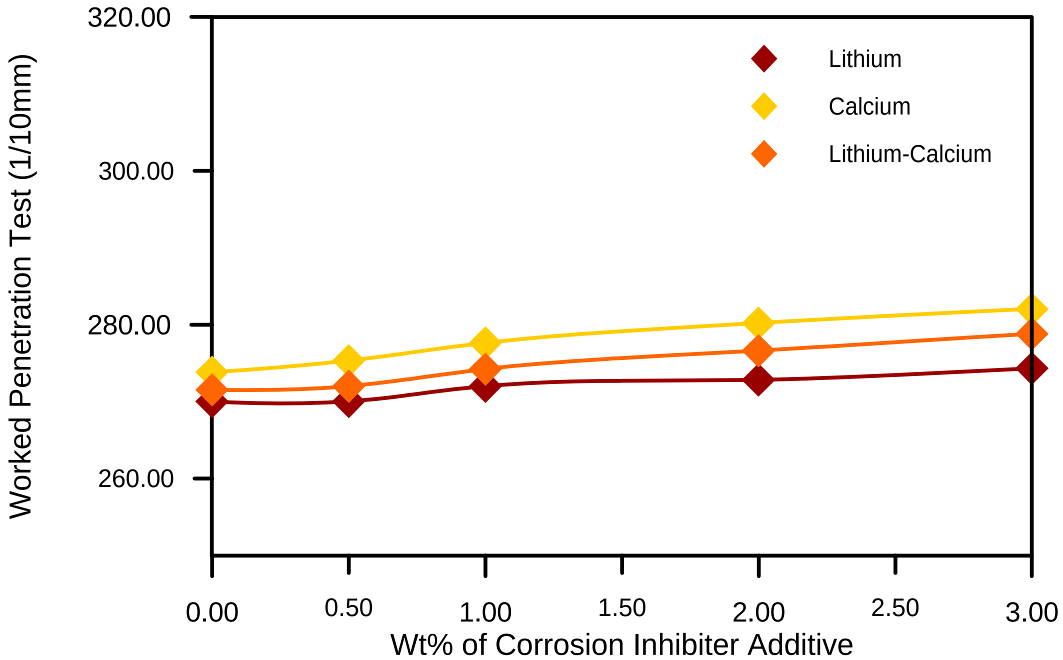


Figure 4-42 Comparison of corrosion inhibiter additive effect on worked penetration test in lithium, calcium and lithium-calcium grease.

4.7 Effect of Base Stock oils (40, 60, 150) Additive:

In many cases, the grease is subject to outside oil, or oil may be added, to improve its properties and increase its storage time or any other property. To evaluate the effect of this outside oil after manufacturing, three samples of stock oil were tested based on the viscosity of each type to find its effect on grease properties. It is noticed that as the viscosity increases the effect of oil on changing grease properties will decrease.

A stock oil 40 was gradually added, on a weight percentage basis, to the greases used in this work and the four-ball welding test was carried out as shown in Figures 4-43, 4-44.

Generally, it was noticed that when adding 20% of stock oil 40, the load carrying property start to decrease. It was also noticed that at the same weight percentage the balls wear increased as shown in Fig. 4-45. The drop point also, at that weight percentage was notably decreased and the worked penetration was increased, as shown in Figures 4-47, 4-49, which indicate that the grease start to lose its properties.

When a weight percent of the stock oil 60 was added, the four types of grease started to lose property at 30% as shown in Figures 4-51, 4-53, 4-55, 4-57.

For stock oil 150, when the addition reached 30% no significant change in properties was noticed, as shown in figures 4-59, 4-61, 4-63, 4-65.

Figures 4-68, 4-69 show, as an example, the values of drop point corresponding to various % of lithium grease for stock oils 40, 60 and 150.

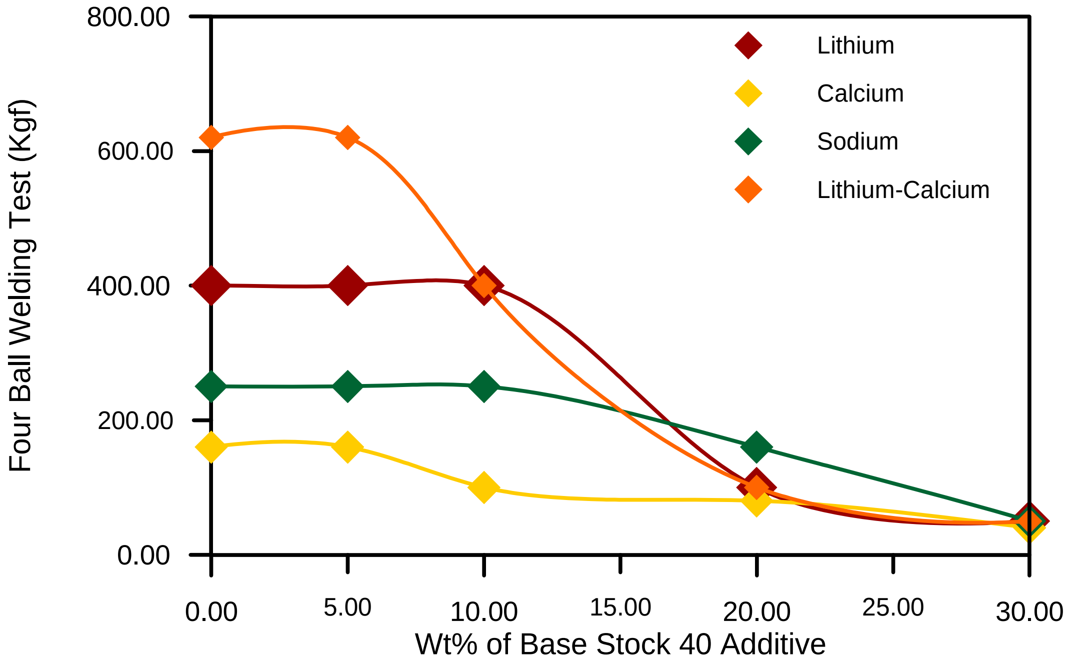


Figure 4-43 Effect of base stock 40 additive on four-ball welding test.

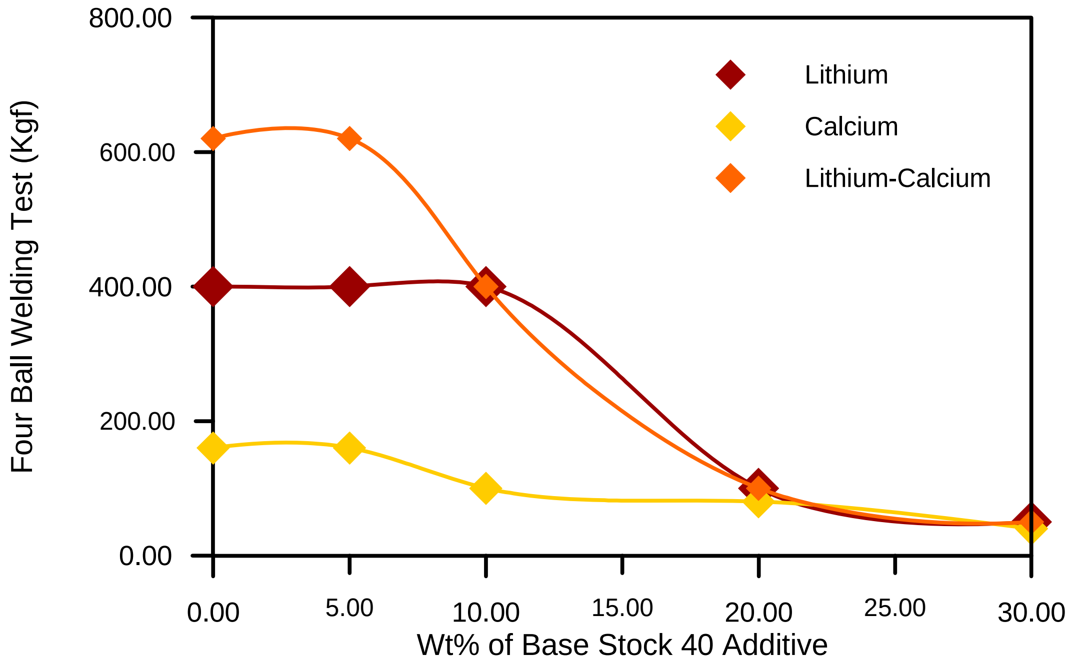


Figure 4-44 Comparison of base stock 40 additive effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

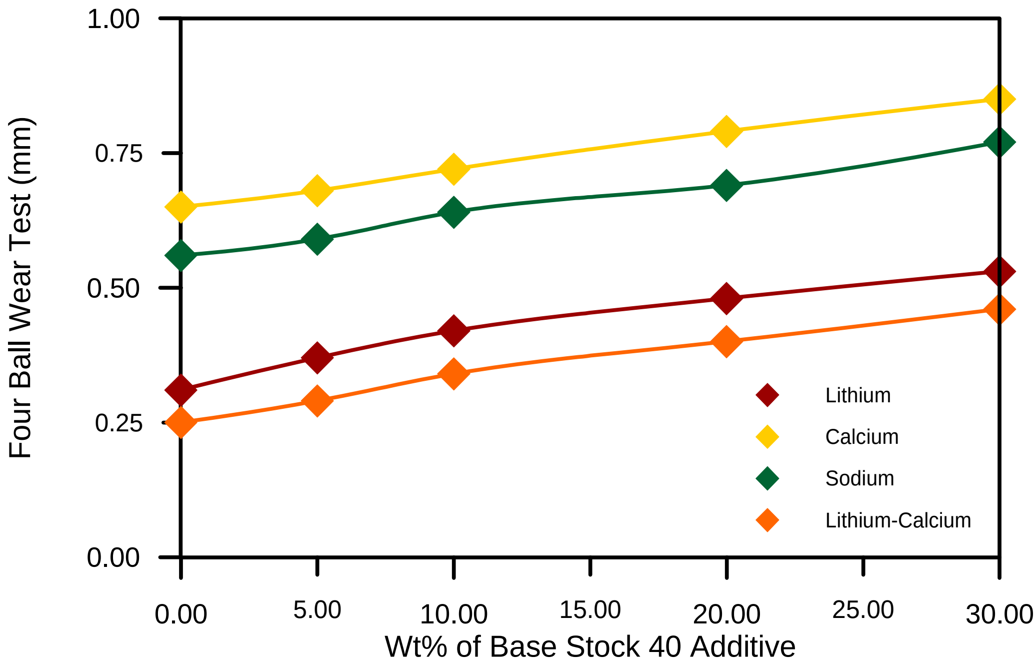


Figure 4-45 Effect of base stock 40 additive on four-ball wear test.

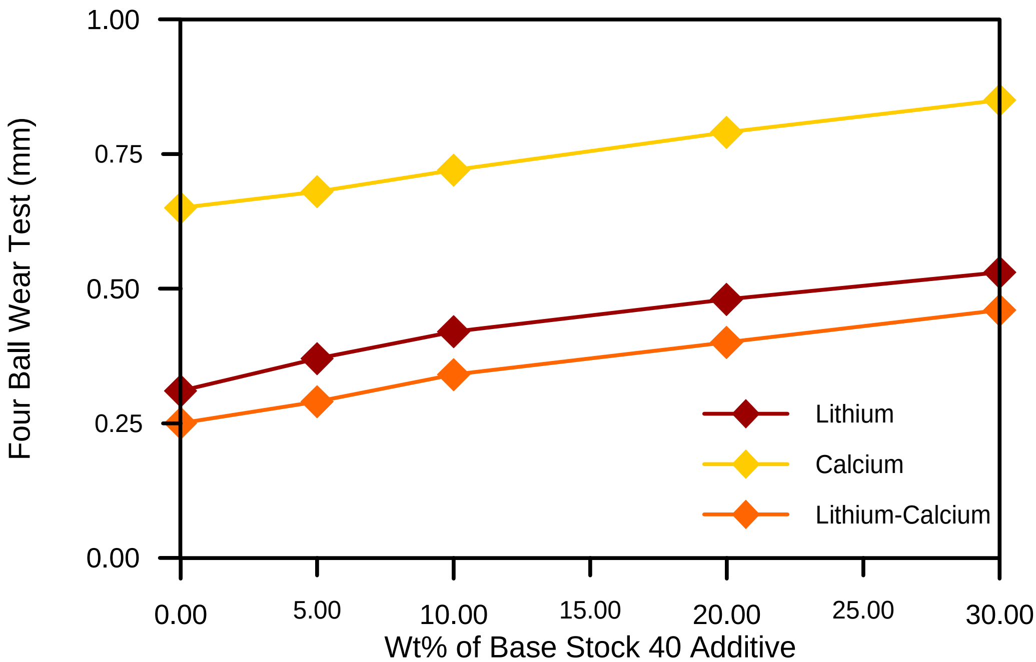


Figure 4-46 Comparison of base stock 40 additive effect on four-ball wear test in lithium, calcium and lithium-calcium grease.

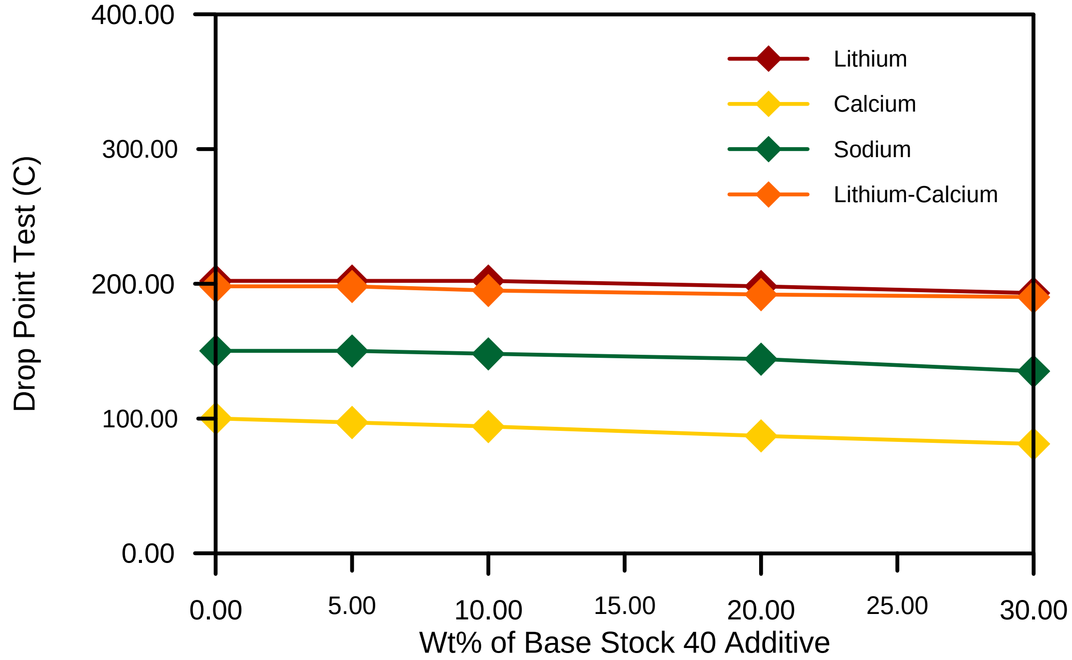


Figure 4-47 Effect of base stock 40 additive on drop point test.

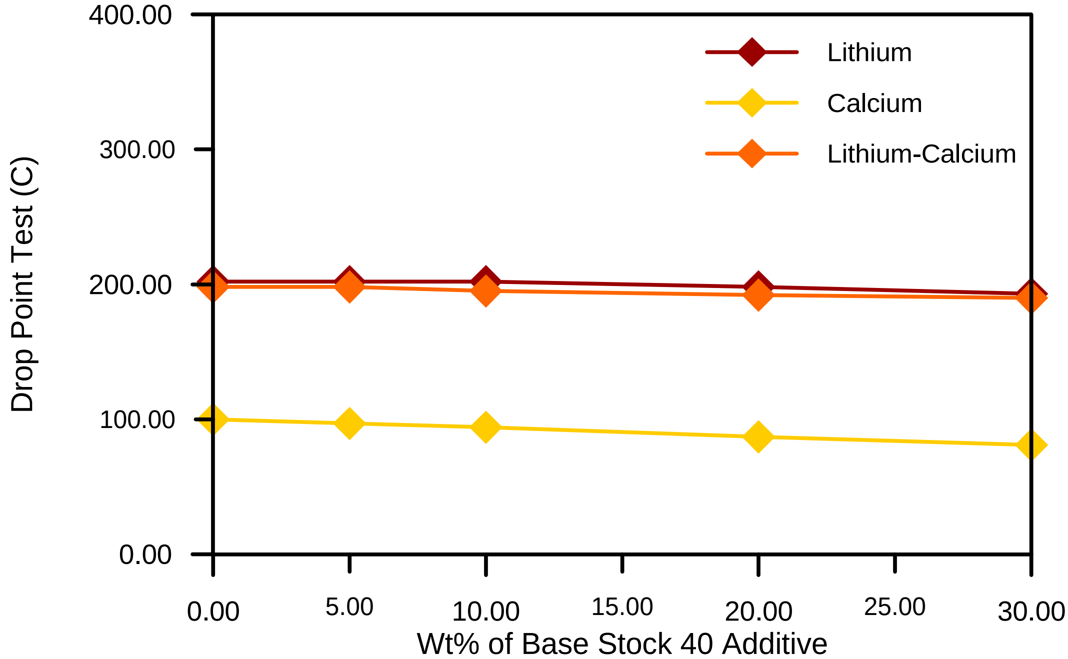


Figure 4-48 Comparison of base stock 40 additive effect on drop point test in lithium, calcium and lithium-calcium grease.

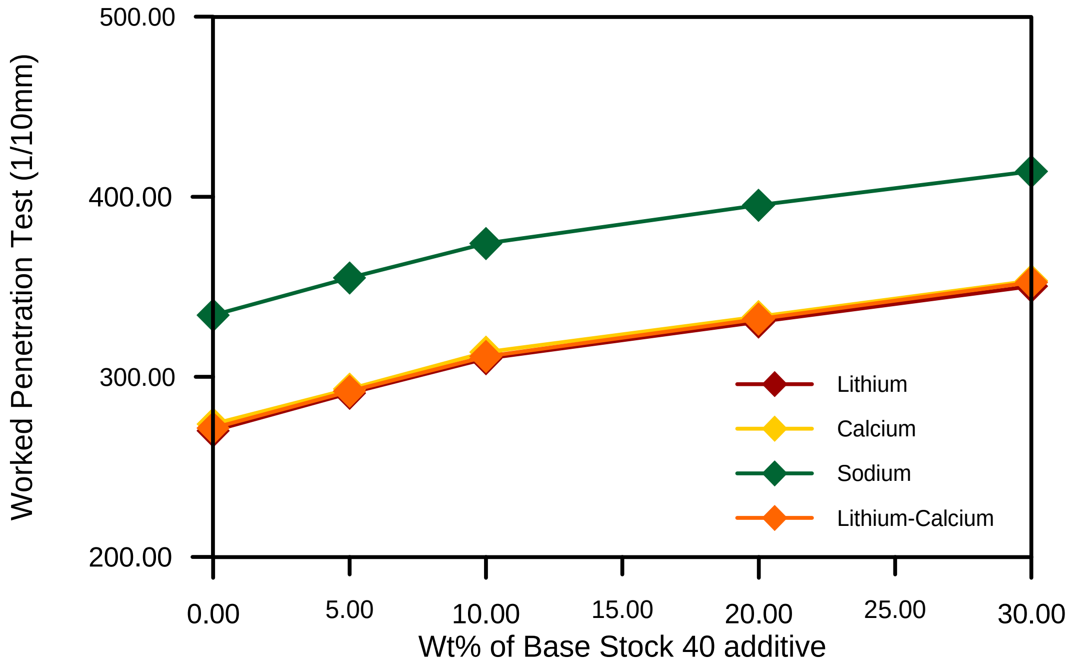


Figure 4-49 Effect of base stock 40 additive on worked penetration test.

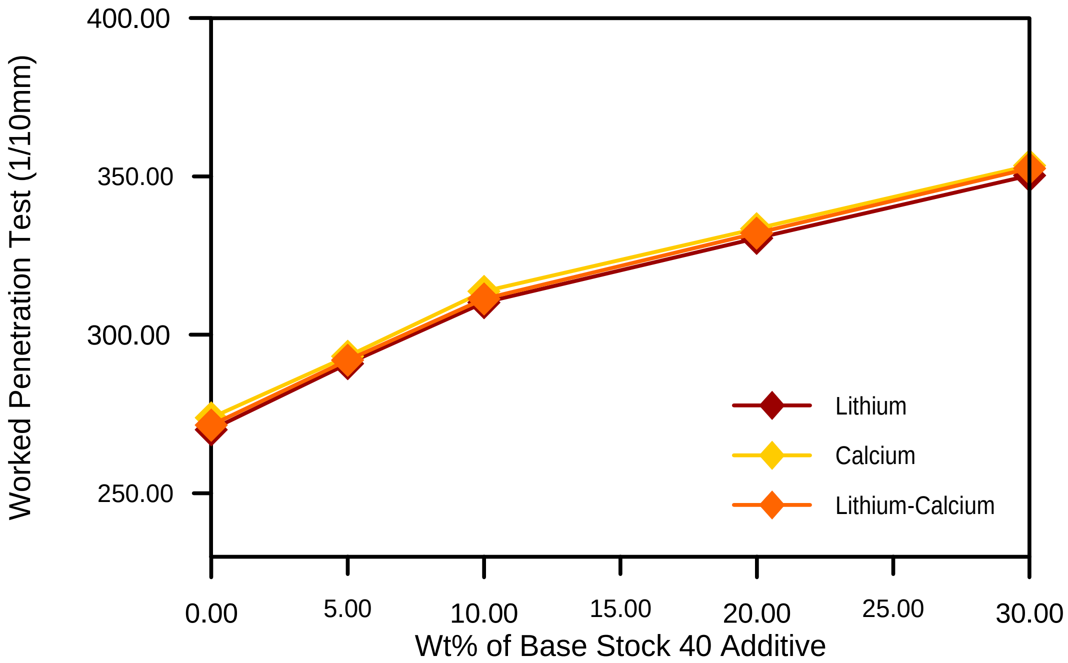


Figure 4-50 Comparison of base stock 40 additive effect on worked penetration test in lithium, calcium and lithium-calcium grease.

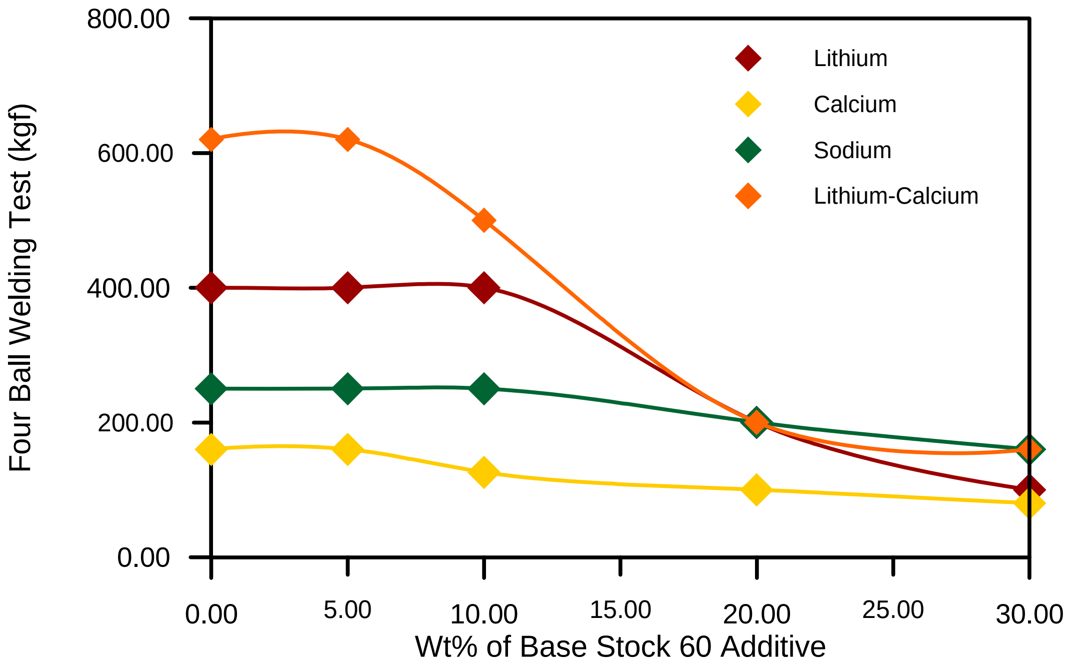


Figure 4-51 Effect of base stock 60 additive on four-ball welding test.

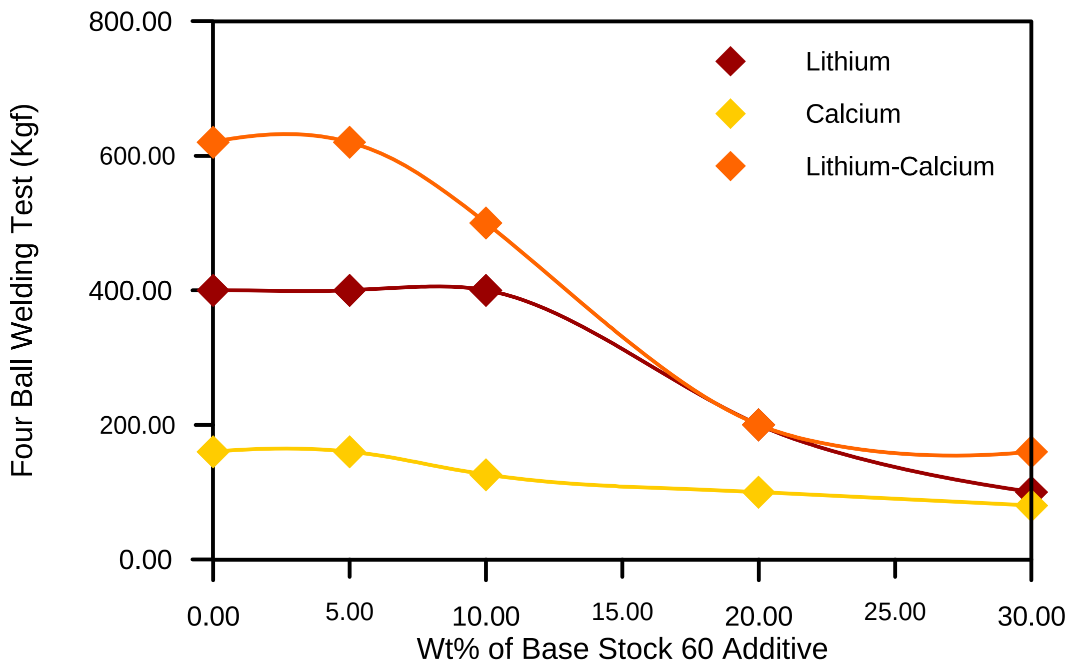


Figure 4-52 Comparison of base stock 60 additive effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

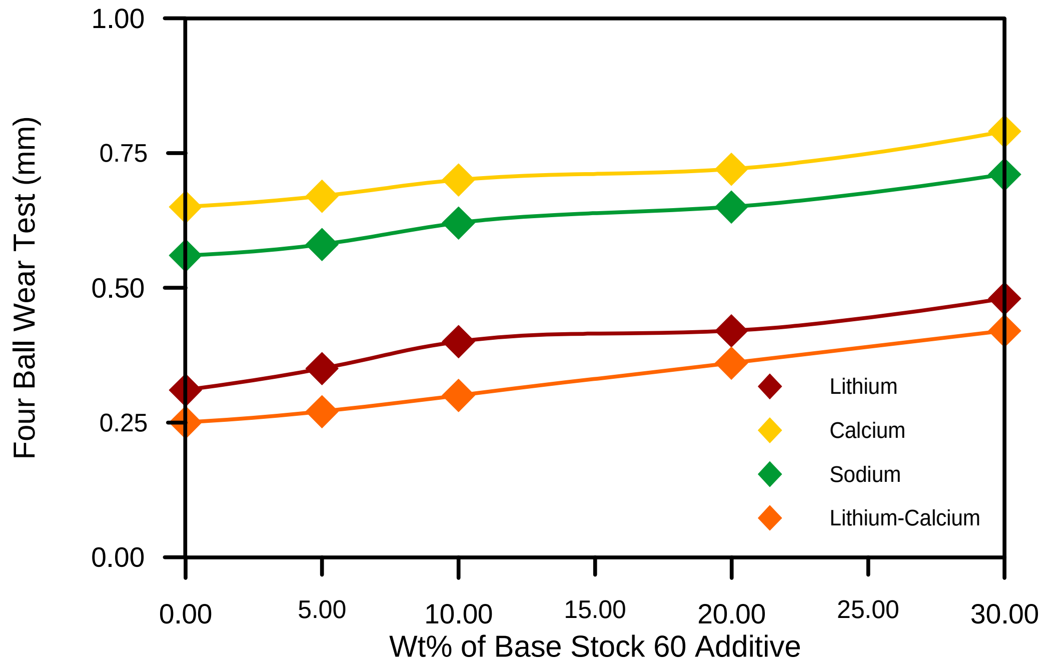


Figure 4-53 Effect of base stock 60 additive on four-ball wear test.

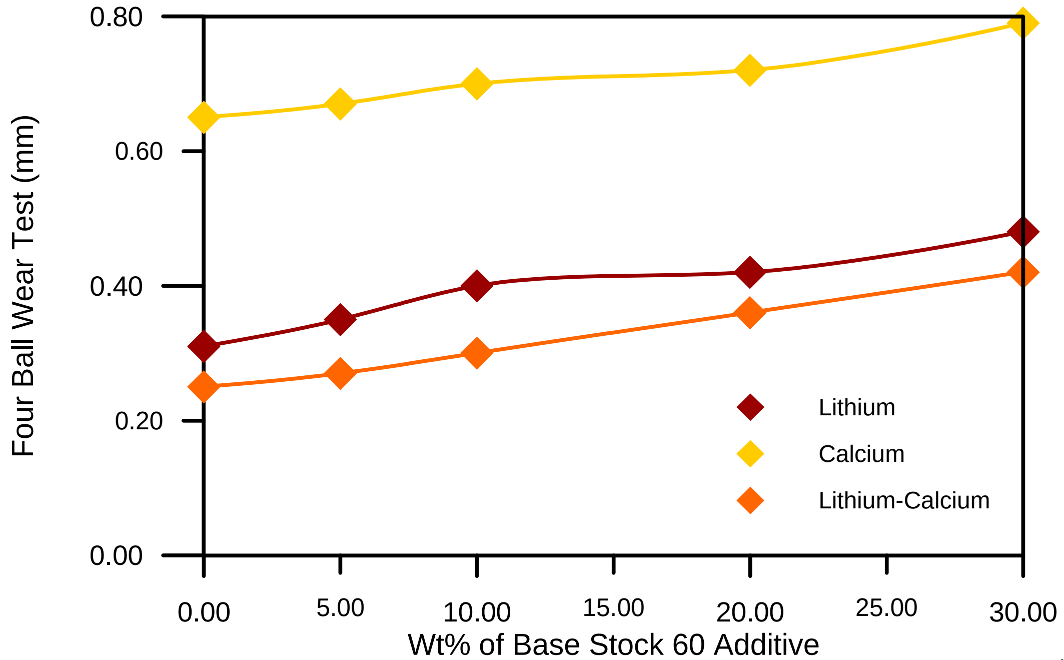


Figure 4-54 Comparison of base stock 60 additive effect on four-ball wear test in lithium, calcium and lithium-calcium grease.

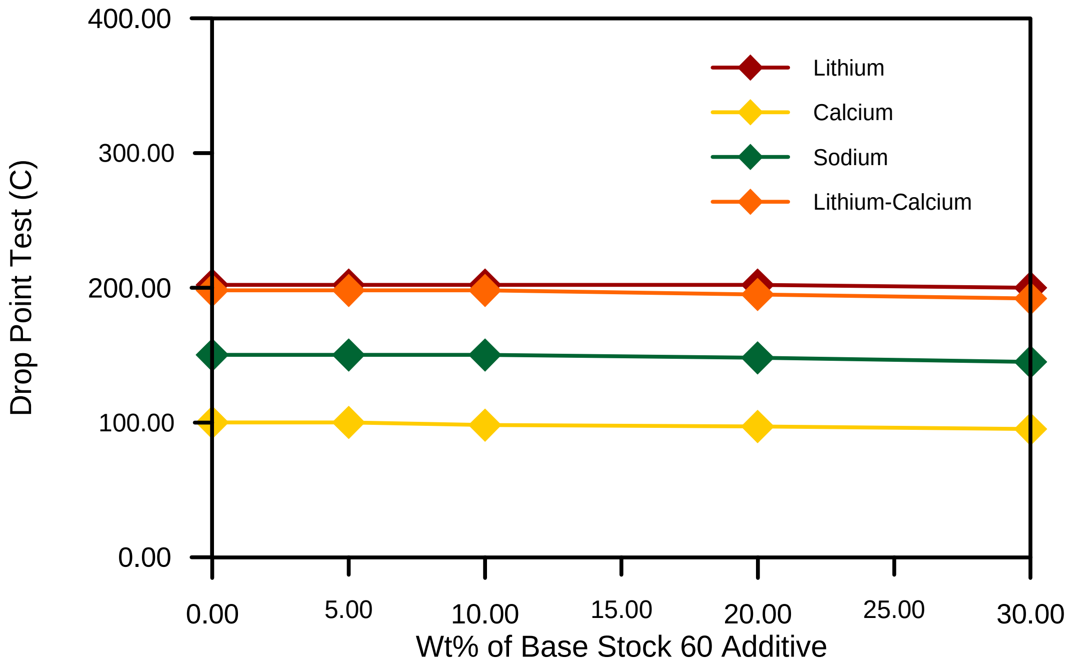


Figure 4-55 Effect of base stock 60 additive on drop point test.

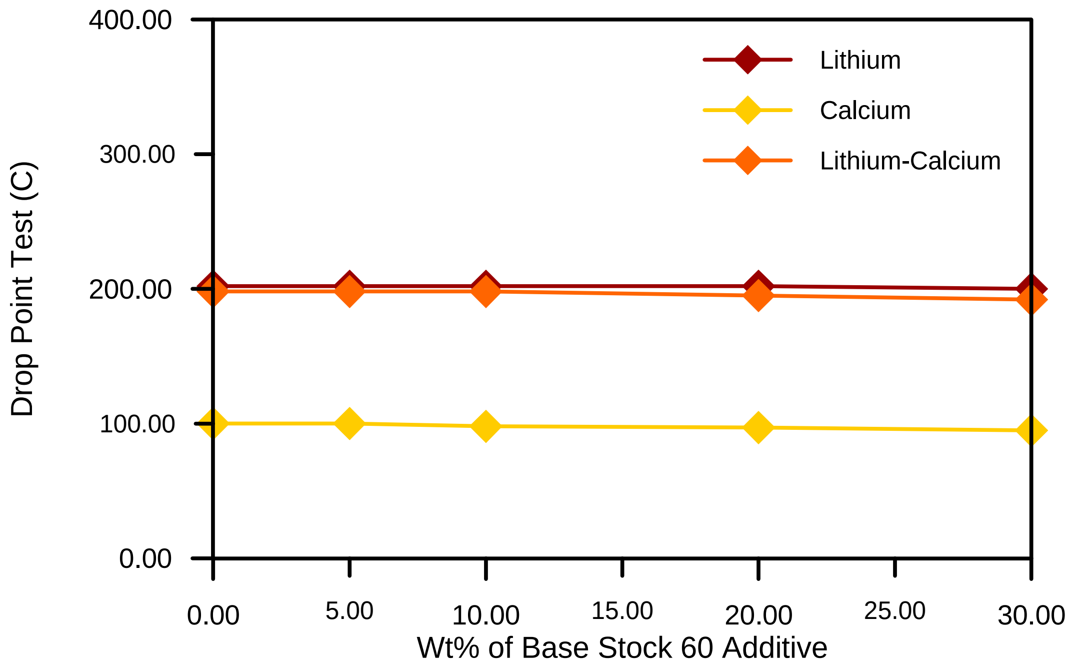


Figure 4-56 Comparison of base stock 60 additive effect on drop point test in lithium, calcium and lithium-calcium grease.

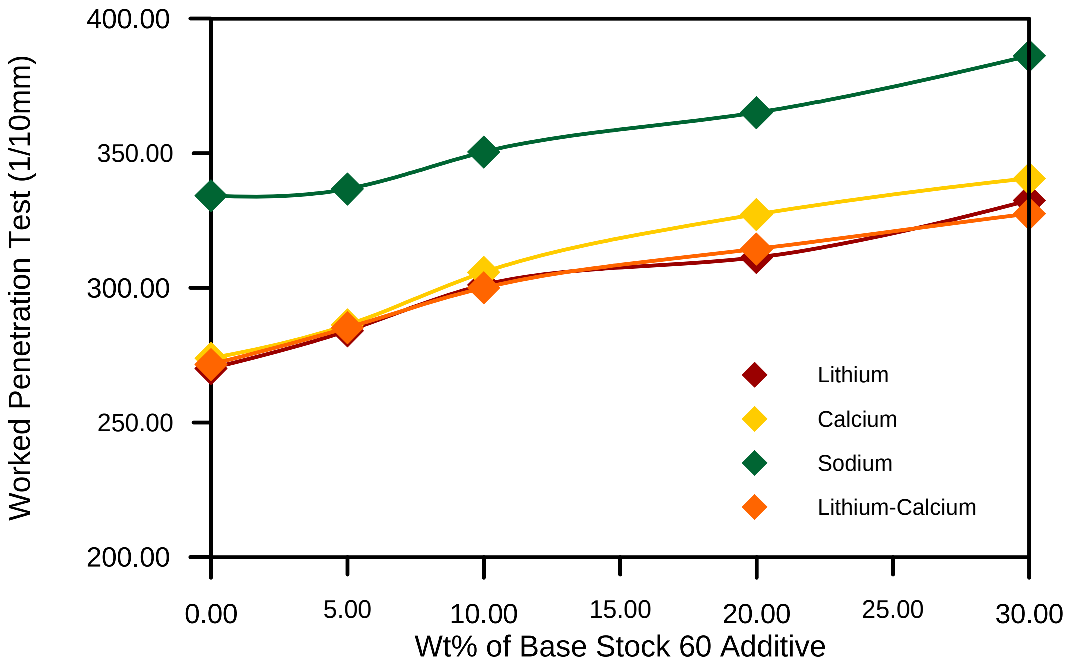


Figure 4-57 Effect of base stock 60 additive on worked penetration test.

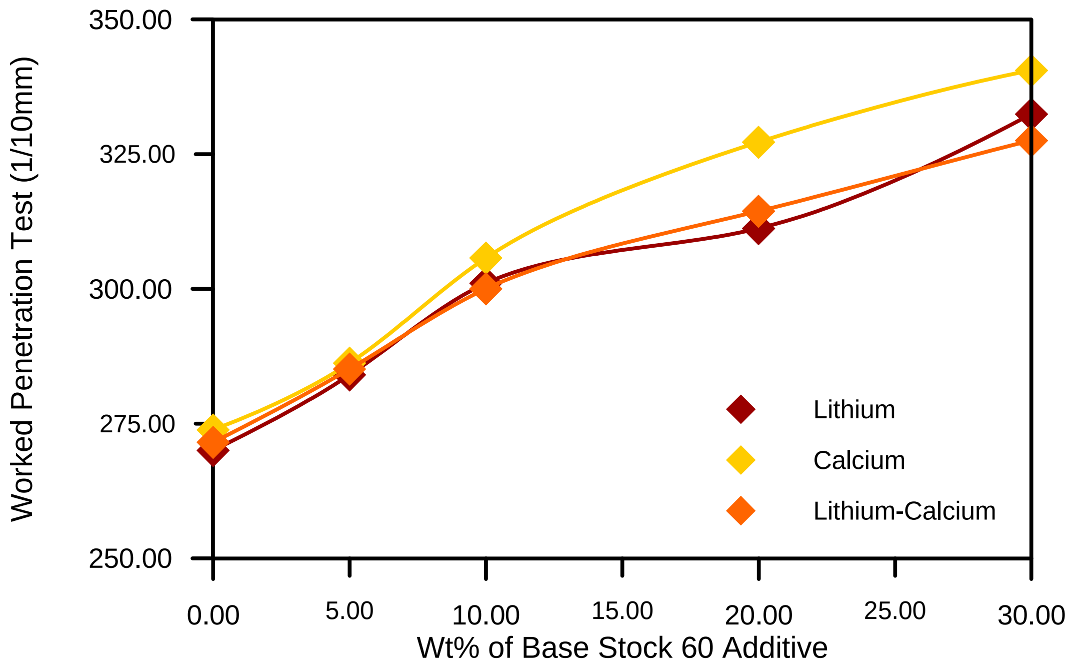


Figure 4-58 Comparison of base stock 60 additive effect on worked penetration test in lithium, calcium and lithium-calcium grease.

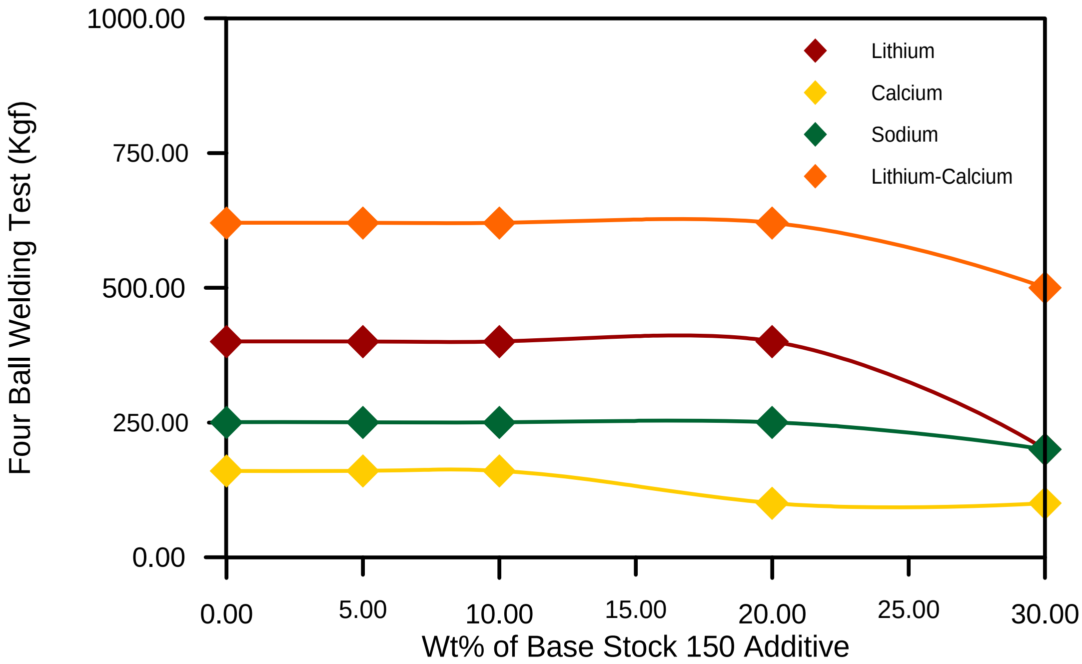


Figure 4-59 Effect of base stock 150 additive on four-ball welding test.

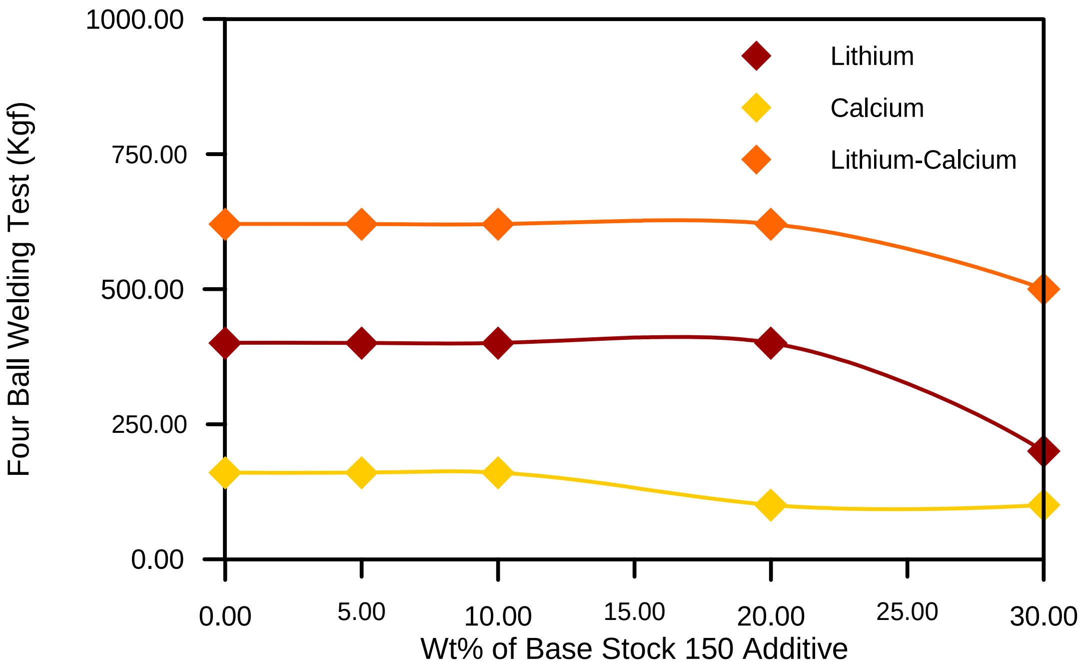


Figure 4-60 Comparison of base stock 150 additive effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

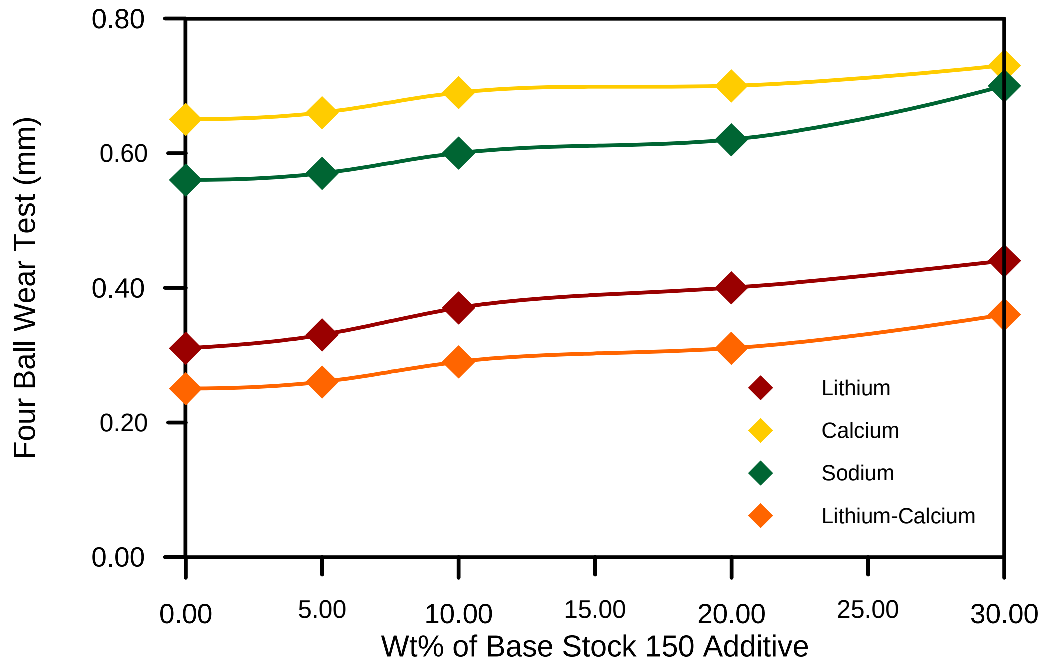


Figure 4-61 Effect of base stock 150 additive on four-ball wear test.

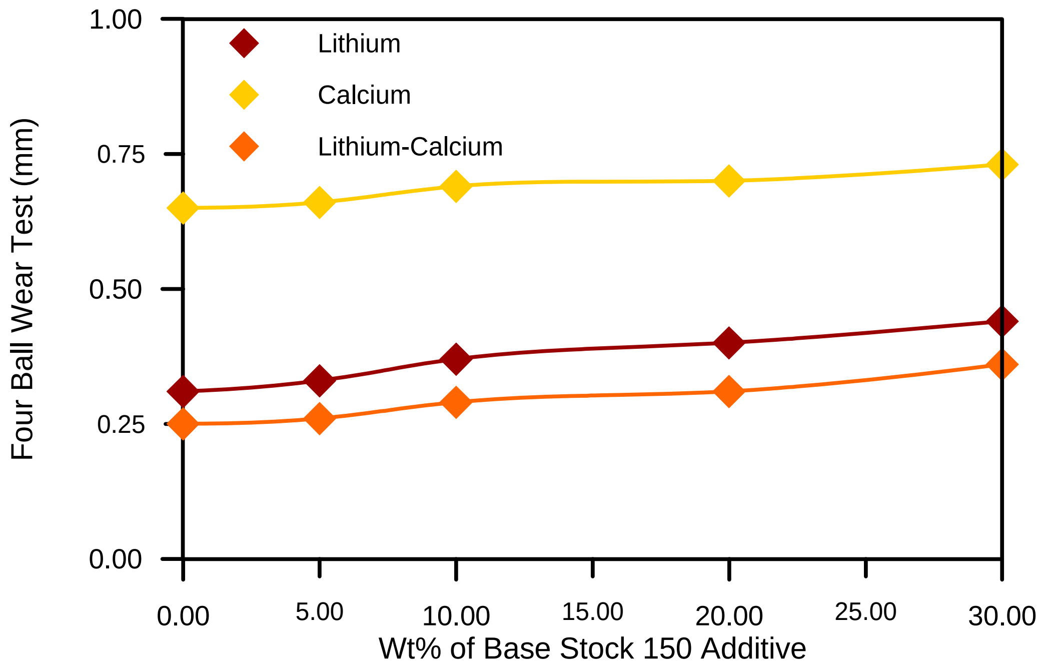


Figure 4-62 Comparison of base stock 150 additive effect on four-ball welding test in lithium, calcium and lithium-calcium grease.

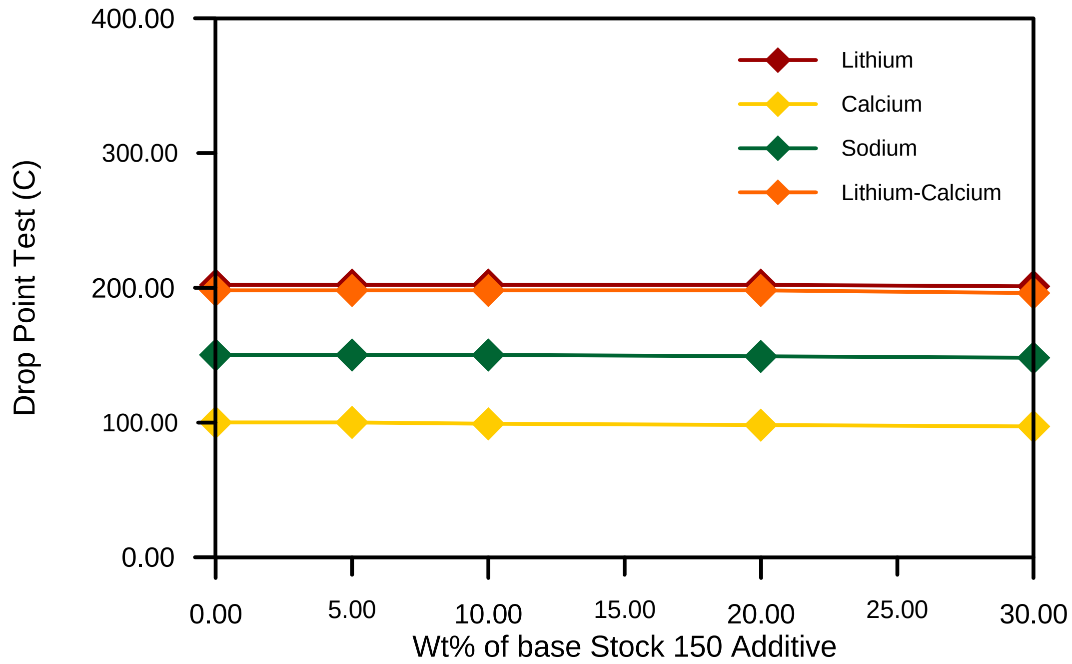


Figure 4-63 Effect of base stock 150 additive on drop point test.

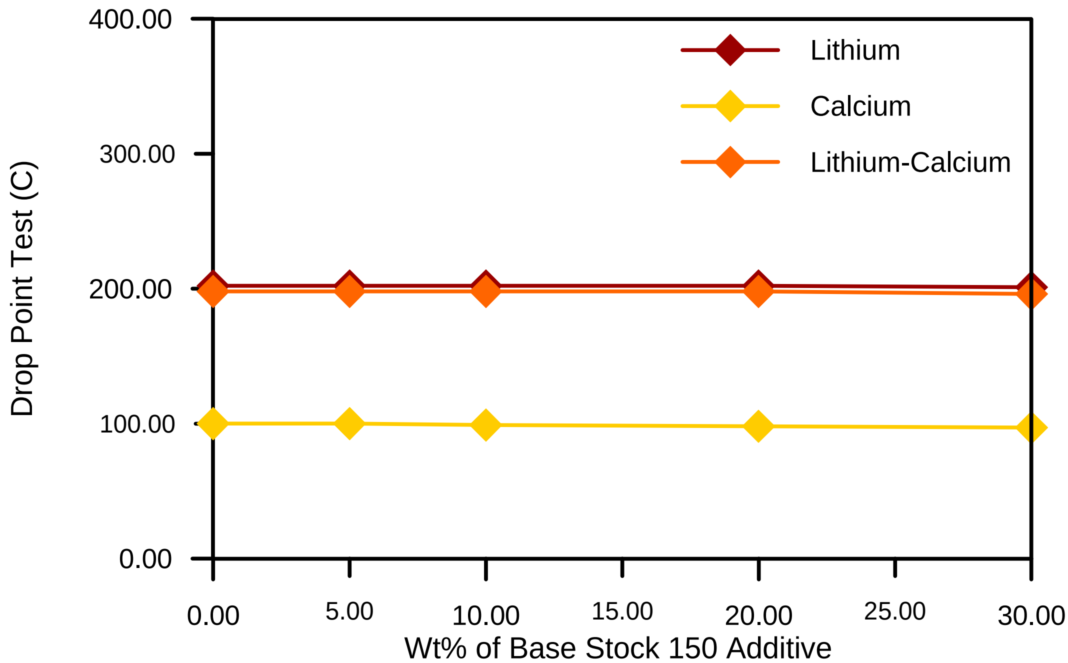


Figure 4-64 Comparison of base stock 150 additive effect on drop point test in lithium, calcium and lithium-calcium grease.

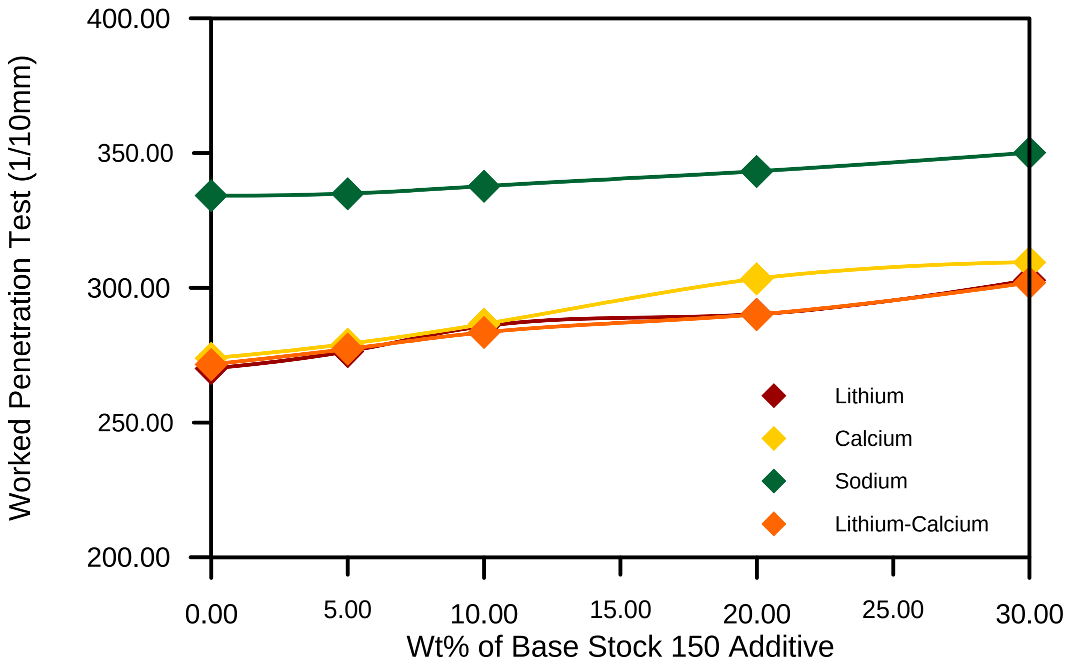


Figure 4-65 Effect of base stock 150 additive on worked penetration test.

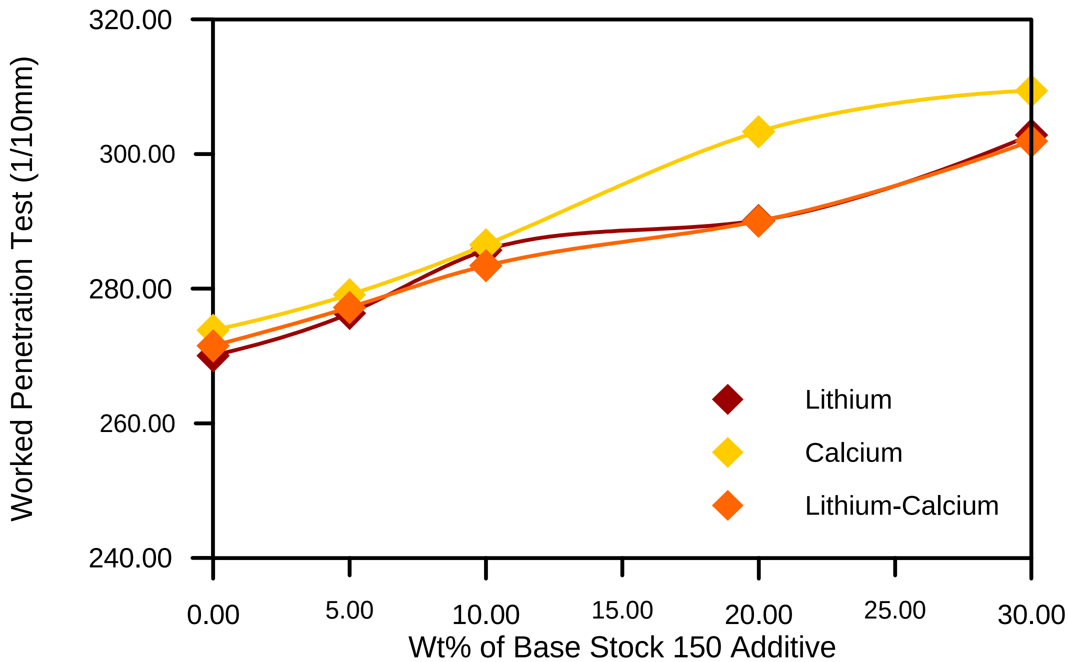


Figure 4-66 Comparison of base stock 150 additive effect on worked penetration test in lithium, calcium and lithium-calcium grease.

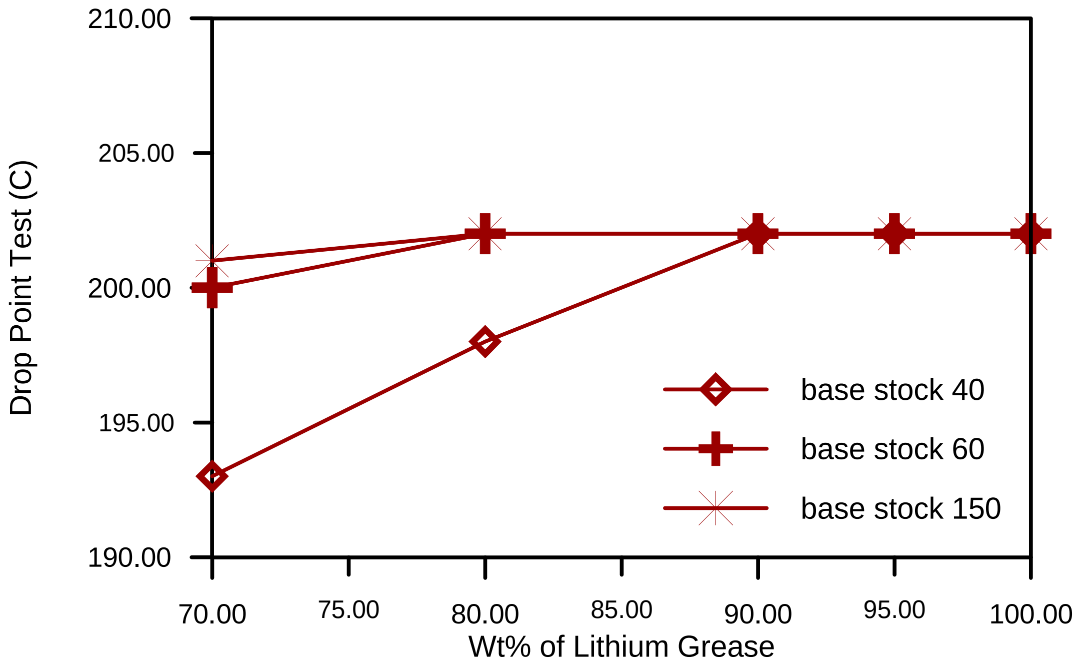


Figure 4-67 Comparison between base stock oils (40, 60, 150) effect on drop point test in lithium grease.

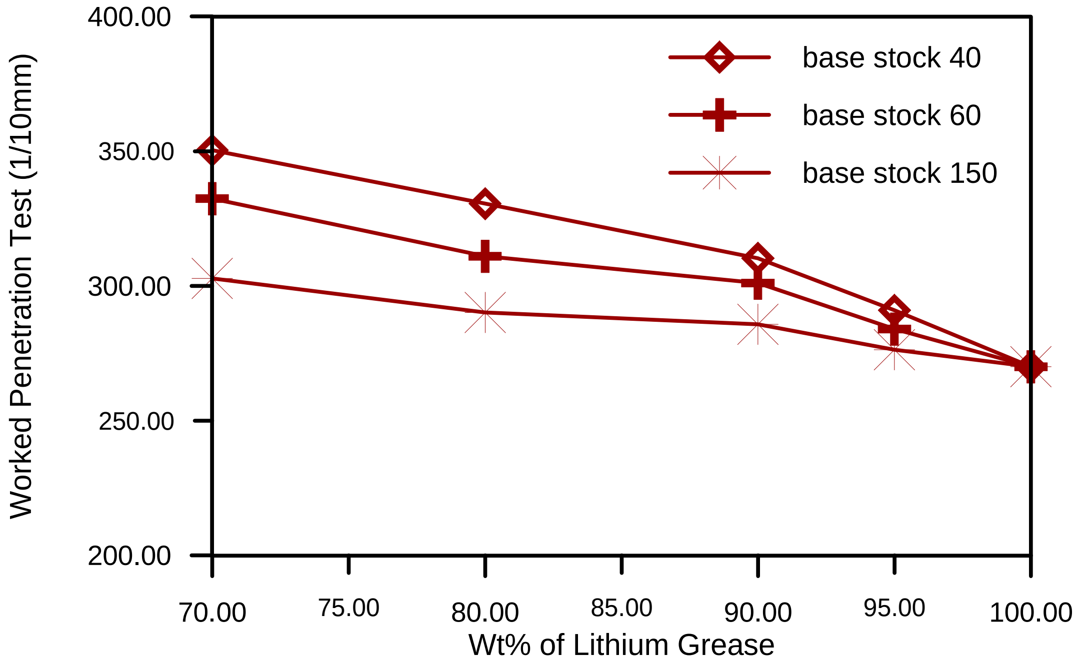


Figure 4-68 Comparison between base stock oils (40, 60, 150) effect on worked penetration test in lithium grease.

Chapter Five

Conclusions and Recommendations

5.1 Conclusions:

1. Improvement of grease to withstand high pressure and having less wear is much affected by the type and percentage of additive used. The best percentage of addition of extreme pressure additive, graphite, molybdenum disulfide and carbon black are 1.5, 3, 1, and 2.5 % respectively in all types of greases used in this study.
2. When adding corrosion inhibiter to calcium grease, the test strip did not show any sign of corrosion. This is an indicator that calcium grease is water repellant and does not need the addition of corrosion inhibiter. The effect of adding corrosion inhibiter to lithium-calcium grease started to be noticed at 1%, 2% at lithium grease and 3% at sodium grease.
3. Adding base stock oils (40, 60, 150) to grease decreases some of the properties, i.e., load carrying, wear resistance, drop point and worked penetration, at 20% of stock oil 40, at 30% of stock oil 60 and no significant change of stock oil 150. This indicates that as the viscosity increases, the change in properties will be less.
4. Lithium grease has many good properties such as load carrying, high temperature performance and shear stability and thus it is considered valuable grease and very commonly used in most parts of the world.
5. When mixing lithium grease with calcium grease and comparing it with lithium grease, it is found that it has slightly better properties than lithium grease. This does not justify leaving the use of lithium grease. That is true for economical reasons due to the need for mixing equipment and electrical power and consequently a higher cost is incurred.

5.2 Recommendations:

1. Studying the other factors which affect the characteristics of grease such as amount and type of thickener, physical characteristics and oil viscosity and low or high temperature performance.
2. Studying the preparation of inorganic additive e.g., bentonite because it is commonly available in Iraq. In Al-Daura Refinery lithium grease is produced from lithium hydroxide and 12-hydroxy stearic acid (both of them are imported) to form a soap. The grease cost can be minimized if bentonite is used instead of the imported materials.
3. Recycling of animal oils (chicken oil and other animal oils waste) can be accumulated and used for production purposes to produce grease by mixing them with mineral oil or other vegetable oils.

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Appendix

Table A-1 Effect of extreme pressure additive on four-ball welding test at various types of greases.

Extreme pressure Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
1	800	200	400	800
1.5	800^+	800	800	800^+
4	800^+	800	800^+	800^+
8	800^+	800	800^+	800^+

Table A-2 Effect of extreme pressure additive on four-ball wear test at various types of greases.

Extreme pressure Wt%	Four-ball wear test (mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	0.31	0.65	0.56	0.25
1	0.24	0.63	0.50	0.22
1.5	0.24	0.60	0.48	0.20
4	0.22	0.58	0.48	0.20
8	0.22	0.54	0.44	0.18

Table A-3 Effect of extreme pressure additive on drop point test at various types of grease.

Extreme pressure Wt%	Drop point (°C)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
1	202	98	150	198
1.5	202	97	150	198
4	202	97	150	198
8	202	95	150	198

Table A-4 Effect of extreme pressure additive on worked penetration test at various types of greases.

Extreme pressure Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
1	270.8	274.5	335.1	272.0
1.5	271.2	275.8	340.3	272.6
4	278.4	282.1	348.2	276.4
8	284.6	293.4	355.3	279.5

Table A-5 Effect of graphite on four-ball welding test at various types of greases.

Graphite Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
3	800^+	400	800	800^+
5	800^+	400	800	800^+
10	800	400	400	800
15	800	400	400	800

Table A-6 Effect of graphite on four-ball wear test at various types of greases.

Graphite Wt%	Four-ball wear (mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	0.31	0.65	0.56	0.25
3	0.26	0.63	0.54	0.24
5	0.25	0.63	0.54	0.24
10	0.25	0.60	0.50	0.23
15	0.25	0.59	0.48	0.22

Table A-7 Effect of graphite on drop point test at various types of greases.

Graphite Wt%	Drop point (^oC)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
3	202	100	150	198
5	202	100	150	198
10	202	100	150	198
15	202	100	150	198

Table A-8 Effect of graphite on worked penetration test at various types of greases.

Graphite Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
3	268.2	268.9	326.7	270.3
5	262.0	260.3	312.1	265.5
10	256.7	255.2	300.0	260.2
15	250.0	243.1	282.3	258.0

Table A-9 Effect of molybdenum disulfide (MoS_2) on four-ball welding test at various types of greases.

MoS_2 Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
1	800^+	315	400	800^+
5	800^+	400	400	800^+
10	800	400	620	800
15	800	400	800	800

Table A-10 Effect of molybdenum disulfide (MoS_2) on four-ball wear test at various types of greases.

MoS_2 Wt%	Four-ball wear(mm)			
	Lithium	**Calcium**	**Sodium**	**lithium-Calcium**
0	0.31	0.65	0.56	0.25
1	0.23	0.60	0.52	0.22
5	0.23	0.59	0.47	0.18
10	0.23	0.55	0.45	0.17
15	0.23	0.50	0.42	0.17

Table A-11 Effect of molybdenum disulfide (MoS_2) on drop point test at various types of greases.

MoS_2 Wt%	Drop point (°C)			
	Lithium	**Calcium**	**Sodium**	**lithium-Calcium**
0	202	100	150	198
1	202	100	150	198
5	202	100	150	198
10	202	100	150	198
15	202	100	150	198

Table A-12 Effect of molybdenum disulfide (MoS_2) on worked penetration test at various types of greases.

MoS_2 Wt%	Worked penetration (1/10mm)			
	Lithium	**Calcium**	**Sodium**	**lithium-Calcium**
0	270.0	273.8	334.2	271.5
1	272.1	274.5	337.4	272.5
5	275.9	277.8	340.7	276.1
10	280.7	282.7	346.2	278.3
15	283.2	286.3	353.1	281.8

Table A-13 Effect of carbon black on four-ball welding test at various types of grease.

Carbon black Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
2.5	800	200	620	800
5	800	250	620	800
10	800^+	400	800^+	800^+
25	800^+	400	800^+	800^+

Table A-14 Effect of carbon black on four-ball wear test at various types of greases.

Carbon black Wt%	Four-ball wear(mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	0.31	0.65	0.56	0.25
2.5	0.29	0.63	0.56	0.25
5	0.29	0.61	0.56	0.25
10	0.28	0.60	0.55	0.24
25	0.28	0.60	0.54	0.23

Table A-15 Effect of carbon black on drop point test at various types of greases.

Carbon black Wt%	Drop point (°C)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
2.5	205	100	157	200
5	210	100	160	208
10	216	100	170	214
25	223	100	170	222

Table A-16 Effect of carbon black on worked penetration test at various types of greases.

Carbon black Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
2.5	260	265.1	314.7	263.4
5	253.6	250.8	298.4	255.8
10	231.0	232.7	278.3	238.1
25	222.8	216.2	263.3	225.0

Table A-17 Effect of corrosion inhibiter on copper strip corrosion test at various types of greases.

Corrosion inhibiter Wt%	Copper strip corrosion			
	Lithium	Calcium	Sodium	lithium-Calcium
0	2a	1a	2a	1b
0.5	1b	1a	2a	1b
1	1b	1a	2a	1a
2	1a	1a	2a	1a
3	1a	1a	1a	1a

Table A-18 Effect of corrosion inhibiter on drop point test at various types of greases.

Corrosion inhibiter Wt%	Drop point (°C)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
0.5	202	100	150	198
1	202	98	150	198
2	202	97	150	198
3	202	97	150	198

Table A-19 Effect of corrosion inhibiter on worked penetration test at various types of greases.

Corrosion inhibiter Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
0.5	270.0	275.3	335.7	272.0
1	272.0	277.6	338.5	274.2
2	272.0	280.2	343.2	276.6
3	274.3	282.0	348.9	278.8

Table A-20 Effect of base stock 40 on four-ball welding test at various types of greases.

Base stock 40 Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
5	400	160	250	620
10	400	100	250	400
20	100	80	160	100
30	50	40	50	50

Table A-21 Effect of base stock 40 on four-ball wear test at various types of greases.

Base stock 40 Wt%	Four-ball wear (mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	0.31	0.56	0.56	0.25
5	0.37	0.68	0.50	0.29
10	0.42	0.72	0.64	0.34
20	0.48	0.79	0.69	0.40
30	0.53	0.85	0.77	0.46

Table A-22 Effect of base stock 40 on drop point test at various types of greases.

Base stock 40 Wt%	Drop point (°C)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
5	202	97	150	198
10	202	94	148	195
20	198	87	144	192
30	193	81	135	190

Table A-23 Effect of base stock 40 on worked penetration test at various types of greases.

Base stock 40 Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
5	290.9	293.2	354.9	292.0
10	310.2	313.7	374.1	311.3
20	330.5	333.5	395.3	332.1
30	350.3	353.4	414.1	352.5

Table A-24 Effect of base stock 60 on four-ball welding test at various types of greases.

Base stock 60 Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
5	400	160	250	620
10	400	126	250	500
20	200	100	200	200
30	100	80	160	160

Table A-25 Effect of base stock 60 on four-ball wear test at various types of greases.

Base stock 60 Wt%	Four-ball wear (mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	0.31	0.65	0.56	0.25
5	0.35	0.67	0.58	0.27
10	0.40	0.70	0.62	0.30
20	0.42	0.72	0.65	0.36
30	0.48	0.79	0.71	0.42

Table A-26 Effect of base stock 60 on drop point test at various types of greases.

Base stock 60 Wt%	Drop point (°C)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
5	202	100	150	198
10	202	98	150	198
20	202	97	148	195
30	202	95	145	192

Table A-27 Effect of base stock 60 on worked penetration test at various types of greases.

Base stock 60 Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
5	284.0	286.2	336.7	285.1
10	301.0	305.7	350.4	300.0
20	311.2	327.2	365.0	314.4
30	332.4	340.5	386.1	327.5

Table A-28 Effect of base stock 150 on four-ball welding test at various types of greases.

Base stock 150 Wt%	Four-ball welding (kg_f)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	400	160	250	620
5	400	160	250	620
10	400	160	250	620
20	400	100	250	620
30	200	100	200	500

Table A-29 Effect of base stock 150 on four-ball wear test at various types of greases.

Base stock 150 Wt%	Four-ball wear (mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	0.31	0.65	0.56	0.25
5	0.33	0.66	0.57	0.26
10	0.37	0.69	0.60	0.29
20	0.40	0.70	0.62	0.31
30	0.44	0.37	0.70	0.36

Table A-30 Effect of base stock 150 on drop point test at various types of greases.

Base stock 150 Wt%	Drop point (°C)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	202	100	150	198
5	202	100	150	198
10	202	99	150	198
20	202	98	149	198
30	201	97	148	196

Table A-31 Effect of base stock 150 on worked penetration test at various types of grease.

Base stock 150 Wt%	Worked penetration (1/10mm)			
	Lithium	Calcium	Sodium	lithium-Calcium
0	270.0	273.8	334.2	271.5
5	276.3	279.1	334.9	277.2
10	285.7	386.5	337.7	283.4
20	290.1	303.3	343.2	290.0
30	302.8	309.4	350.1	301.9

الخلاصة

يهدف البحث الى دراسة بعض الاضافات التي تؤثر على خصائص الشحوم الصابونية (ليثيوم، كالسيوم، صوديوم، ليثيوم – كالسيوم) بأضافة اولا: مضافات تشمل الكرافيت، ثاني كبريتيد المولبيدينوم، اسود الكاربون، مانع التاكل، ومضاف الضغط العالي. ثانيا: زيوت الاساس التي تشمل 40، 60، 150 الى الشحوم.

تم اضافة نسب مختلفة من تلك المضافات الى الشحوم للحصول على افضل النسب التي تحسن خواص الشحوم من حيث قابليتها على تحمل الضغط العالي، مقاومة السوفان، مقاومة الصدأ، درجة السقوط، والنفاذية.

تم التوصل الى ان افضل النسب الوزنية التي اعطت خواصا جيدة لانواع الشحوم المستخدمة كافة هي 1,5% مضاف الضغط العالي، 3% كرافيت، 1% ثاني كبريتيد المولبيدينوم، 2,5% اسود الكاربون.

أما مانع التاكل فقد كانت افضل نسبة وزنية للأضافة هي 1% لشحم الليثيوم – كالسيوم، 2% لشحم الليثيوم، 3% لشحم الصوديوم. ولم تظهر الحاجة لأضافته الى شحم الكالسيوم.

وبالنسبة لأضافة زيوت الأساس (40,60، 150) الى الشحوم لوحظ انخفاض خواص الشحوم عند أضافتها بنسب 20% لزيت الأساس 40، 30% لزيت الأساس 60، وعدم ظهور تغير ملحوظ بالنسبة لزيت الأساس 150.

شكر وتقدير

اود ان اعبر عن خالص شكري وتقديري وامتناني العميق للمشرف الاستاذ الفاضل الدكتور مهند عبد الرزاق لما قدمه لي من توجيهات قيمة ونصائح سديدة طوال فترة اعداد البحث.

كما اود ان اشكر جميع اساتذة وموظفي قسم الهندسة الكيميائية في جامعة النهرين لابدائهم المساعدة اللازمة اثناء هذا العمل .

واتقدم بالعرفان والتقدير الى السادة السؤولين في مصفى الدورة على ما قدموه من تسهيلات عملية ساعدتني في اجراء الفحوصات اللازمة لاكمال البحث.

ولاأنسى ان اشكر كل من ساعدني في اكمال هذا العمل واخص بالذكر الاستاذ الفاضل المهندس الاستشاري عصام أحمد عطية.

واخيرا شكري الجزيل الى ابي وامي وافراد عائلتي لوقوفهم معي طوال مدة اعداد هذا البحث.

زينب حيدر عبود

تأثير بعض الإضافات على خصائص الشحوم

رسالة
مقدمة إلى كلية الهندسة في جامعة النهرين
وهي جزء من متطلبات نيل درجة ماجستير علوم في الهندسة الكيمياوية

من قبل
زينب حيدر عبود الموسوي
بكالوريوس علوم في الهندسة الكيمياوية 2006

ذو الحجة 1430 هـ
كانون الأول 2009 م