FORMULATION OF MULTI-PURPOSE GREASE FOR USE UNDER SEVERE CONDITIONS

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ABSTRACT

Mixtures of greases are formulated using polymethyl-silioxan as base oil with viscosity 1000 cS at 25°C and are thickened with the 2-20 % wt. fume silica. Iron octoate in the range 0.1 to 0.5 % wt. and molybdenum disulfide in the range 2 to 10 % wt were included to achieve the necessary oxidation, and load carrying properties, respectively.

Keywords: Non-soap grease, thickening agent, fume silica, polymethyl-silioxan.

INTRODUCTION

The use of liquid lubricant generally requires sealing of bearing against loss of lubricants. This sealing problem can often be simplified if lubricants are employed which resist the deforming effect of gravity. Such "solidified" lubricants are usually called "greases", and are mostly composed of lubricating oil, which would normally have been employed, together with a gelling agent, which lends stiffness to the mixture [1].

Grease, as a rule, is a viscous plastic product, which adhere strongly to the packing material [2]. Grease consists of two basic structural components: a thickening agent and liquid, or base fluid, in which that thickening agent is dispersed. Many types and combination of thickener and base fluids, along with supplemental structure modifiers and performance additives, give final grease formulation their specialty [3].

The essential property which grease must possess is the ability to form a film between surfaces so that surface-to-surface contact is prevented. This film may be quite thick, where grease is being used as an anti-friction bearing [4].

Many thickening agents are being added to both petroleum oils and synthetic oils to make grease, which are usually soap-thickened mineral oils to which the different soaps employed as thickeners give a variety of texture. Some of more common thickening agents are non-soap-thickening agent as bentonite [4].

In modern practice, it has become increasingly important that a grease composition is able to provide adequate lubrication at high temperature. Further, because many of the newer high temperature grease applications, e.g. high level of the lubricant activity for extended time periods, it also essential, that the thickener imports a high degree of mechanical stability on the grease formulation at such high temperatures[5].

Perhaps one of the most contributions of grease industry has been to the aircraft industry, particularly the military aircraft equipment [6].

The temperature over which aircraft greases are expected to give satisfactory performance is probably the most severe requirement to be met and is one of important factors that set aircraft lubrication a port from ground equipment. Aircraft and equipment must be kept to a minimum weight; this tends toward reduction of torque available for overcoming frictional resistance of grease and toward high rise in some lubricated parts. It's interesting to note the temperature range in which such lubricants must function. Thus, the low temperature encountered with control pulley bearing and normal maximum operating temperature is 79.4 °C. On retracting and flap motors, the corresponding temperatures are –54 and 93.3 °C, while on wheel bearing –54 and 149°C are encountered [7].

There is an increasing demand for lubricants in many fields, such as in military applications, with high load carrying capacity and very high operating temperature, as well as very low temperature specified. These are requirements that give new significance to certain properties such as the thermal stability and relative indifference to temperature extremes. However, one of the important lubricants is silicon oil [8].

With the demand ever increasing for lubricants capable of resisting still higher operating temperatures, silicon fluids have proven ineffective to meet such condition. Iron octaote is present in new compositions silicon oils to impart against gelling of the silicon oils at high temperature [8].

The present work deals with formulation of multi-purpose grease for use wherever severe operating conditions are encountered. This work includes the study of the effects of thickened agent (fume silica), load- carrying additive (molybdenum disulfide) and anti-oxidant agent (iron octoate) on physical and chemical properties. In addition, iron octoate was prepared from octoic acid, ferrous sulfide and caustic soda using precipitation process.

EXPERIMENTAL WORK

Raw Materials

Poly-methyl siloxan fluid

The base oil is poly-methyl siloxan fluid, which is colorless and obtained from Ibn-Sena Company with the properties shown in table 1.

Table 1. Properties of poly-methyl siloxan.

Property	*Value*
Pour point ,°C	-50
Viscosity at 25°C, cS	1000
Flash point ,°C	316
Specific gravity at 25/4°C	0.973
Surface tension, dyne/cm	21.2

Fume silica

Fume silica is a wide amorophous powder having surface areas of 80 m2 per gram, with particles size of 8 A°.

Molybdenum di-sulfide (MoS_2)

Molybdenum di-sulfide, with 160.08 molecular weight and 7 μm average particles size, is still the most important solid-lubricant additives. MoS2 is supplied from BDH Chemicals Ltd.

Iron octoate

Iron octoate, with elementary analysis of 65%wt Fe, 5 %wt Na and 0.3%wt Ca, is used as anti-oxidan.

EXPERIMENTAL WORK

Grease formulation includes three steps. In the first step, the desired amount of fume silica from 2 to 20 %wt (this weight percent was decided due to the experimental needs) is slurred with 1/3 of the required amount of poly-methyl siloxan in the stainless steel container with stirring. The paddles are twisted so that half of paddle provides an upward thrust and half of the paddle a downward thrust. Mixing should continue until a smooth paste is obtained.

The second step, consist heating of the batch, with continued mixing at 80°C.

In the third step, the rest of poly-methyl siloxan used in the previous steps (2/3 of the required amount of poly-methyl siloxan) mixed with 0.1 to 0.5 %wt and 2 to 10 %wt of iron octoate and molybdenum di-sulfide, respectively. The cooled mixture is slowly blended with the heated paste for 60 minutes [9].

Test Methods

Some standard test methods have been employed on formulated grease. These tests conducted on grease have two main purposes in mind. The first is to insure uniformity in the product; that is, such tests are control or inspection tests. Next, and most important, such tests enable a prediction to be made of the performance of the lubricant when in service. Allied to both of these purposes are informational tests, which are necessities in any development work on lubricant. These tests are: Penetration test (ASTM D-127), Oil separation test (ASTM D-1742), Dropping point test (ASTM D-2265), Corrosive substance test (ASTM D-130), and Oxidation stability test (ASTM D-942).

Effect of Fume Silica Percent on Penetration, Oil Separation, and Drop Point of Formulated Grease

Fume silica with excellent grease–forming properties as a result of its elongated or chain like structure and high oil absorbing properties, was used as a thickening agent.

Figure 1 clarifies the effect of fume silica percent on penetration at different percent of iron octoate, while figure 2 shows the effect of fume silica percent on penetration at different percent of molybdenum disulfide.

The adding of fume silica significantly decreases the penetration. Further decreasing in penetration was observed by increasing fume silica percent as shown in figure 1. This is due to the large strength and small size of fume silica. The bonding of fume silica thickening agent in gel varied with the weight of the solids. Thus, the large strength of fume silica was thought to be due to the structure being knit together by means of primary valences bonds, and this agrees with Kistler [10].

The same tends of penetration decreasing with fume silica percent increasing is observed at different percent of molybdenum disulfide, as shown in Figure 2. The mixture will tend to behave as a semi-solid fluid by increasing solid concentration (fume silica, and molybdenum disulfide) and this increase the stiffness of mixture (grease) which can be seen clearly within high concentration of solid.

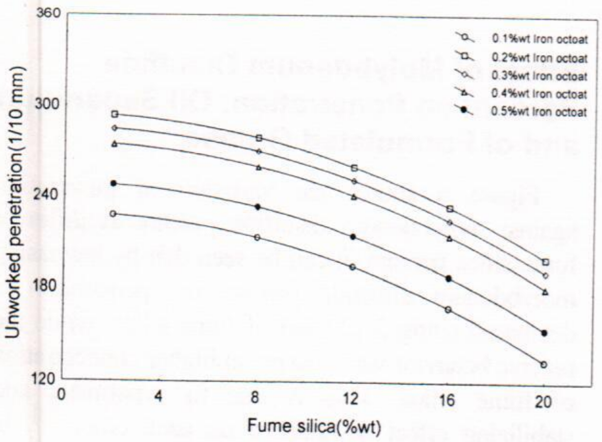

Figure 1.Effect of fume silica on penetration at constant concentration of molybdenum disulfide (3%wt.).

Figure 3 shows the effect of the percent of fume silica on oil separation percent at different percent of molybdenum disulfide. From this figure, it can be noticed that by increasing fume silica from 2 to 12 %wt the oil separation rapidly decreases. After 12 %wt of fume silica, oil separation becomes approximately unchanged. This is because of the too small percent of fume silica results in a deficient structure of grease and this increases the oil separation.

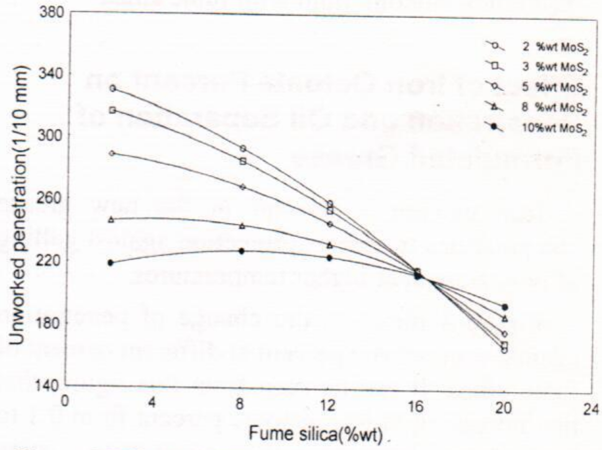

Figure 2.Effect of fume silica on penetration at constant concentration of iron octoate (0.2 %wt.).

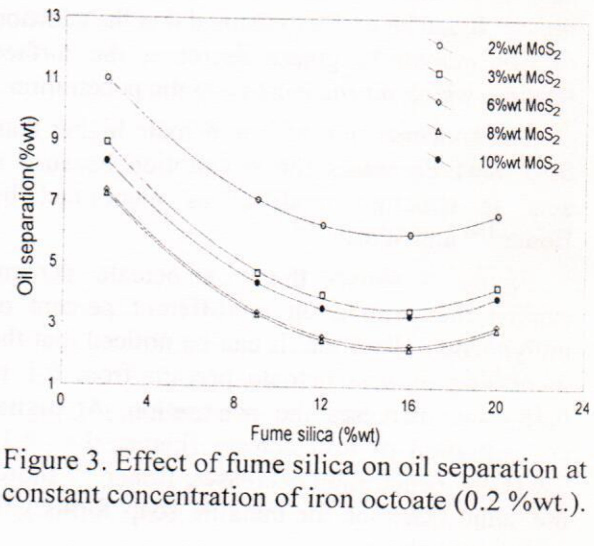

Figure 3. Effect of fume silica on oil separation at constant concentration of iron octoate (0.2 %wt.).

This also indicated by Mchennan [11]. He stated that leakage of oil from grease could be aggravated by too small percent of thickening agent and thus could be expected in the softer grades of grease. Furthermore, the fume silica particles have greater attraction for each other than for oil.

It can be easily noticed from figures 1 to 3 that the sufficient concentration of fume silica and molybdenum disulfide required for 4.7 % wt oil separation and penetration 277 were 9 % wt, and 3%wt., respectively.

Drop point is critical point at which the gel structure breaks down, and the whole grease becomes liquid. The experimental results indicates that the formulated grease shows no drop point (340°C+), and there is no effect of fume silica in the range from 5 to 20 %wt. This observation is a good agree with the results of Grant and Currie[6] for grease prepared by thickening silicone fluid with fume silica.

Effect of Iron Octoate Percent on Penetration and Oil Separation of Formulated Grease

Iron octoate is present in the new grease compositions to import protection against gelling of poly siloxan at higher temperatures.

Figure 4 observes the change of penetration against iron octoate percent at different percent of fume silica. It can be seen from this figure, that the increasing of iron octoate percent from 0.1 to 0.35 %wt increases the penetration using different percent of fume silica.

Generally, the iron octoate (metallic soap) acts a softener in the case of its addition to grease. It can be easily explained that the addition of iron octoate to grease decreases the surface tension, which means increasing the penetration.

The concentration of iron octoate higher than 0.35 %wt decreases the penetration because it acts as structure modifier as mentioned by Boner [12] and Worth [13].

Figure 5 shows the iron octoate percent against the penetration at different percent of molybdenum disulfide. It can be noticed that the increasing of iron octoate percent from 0.1 to 0.35 %wt increases the penetration. At higher concentration of iron octoate (higher than 0.35 %wt), the penetration decreases. Boner [12] shows the same behavior for metallic soap forms gels with liquid phase.

Figures 4 and 5 indicate that the desired penetration 277 was obtained using 0.2 %wt of iron octoate. This is in a good agreement with the results of Firtz [8] who proposed the 0.2 %wt iron octoate as optimum percent.

The experimental results showed that there is no effect of iron octoate concentration on oil separation in the range from 0.1 to 0.5%wt. Iron octoate is remaining in dispersed phase in base oil for prepared grease. It prevent the setting out of thickening agent as a mentioned by Miller and Morway. [14]

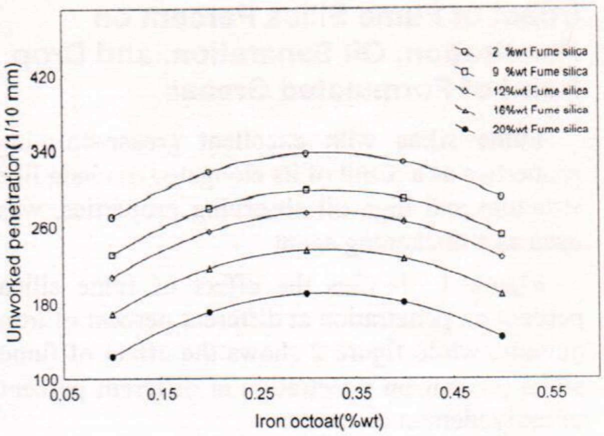

Figure 4. Effect of iron octoate on penetration at constant concentration of molybdenum disulfide (3 %wt.).

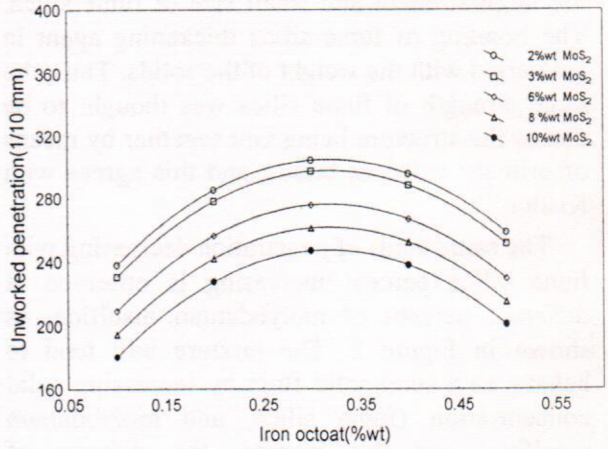

Figure 5. Effect of iron octoate on penetration at constant concentration of fume silica (9 %wt.).

Effect of Molybdenum Disulfide Percent on Penetration, Oil Separation and of Formulated Grease

Figure 6 shows the variation of penetration against molybdenum disulfide percent at different fume silica percent. It can be seen that by increasing molybdenum disulfide percent the penetration is decreased using 2-12 %wt of fume silica, while the reverse behavior was observe at higher concentration of fume silica. This is due to synergistic and stabilizing effect of additive on each other [15]. In general, the greatest benefits were obtained by adding molybdenum disulfide having the smallest possible particle size[16,17]. The penetration decreases by increasing molybdenum disulfide and that due to fine particle size of molybdenum disulfide [15]. Figure 7 observes the change of molybdenum disulfide against penetration at different percent of iron octoate. Figure 8 show that the increasing molybdenum disulfide percent decreases the oil separation and this is because the oil separation is largely governed by penetration [18].

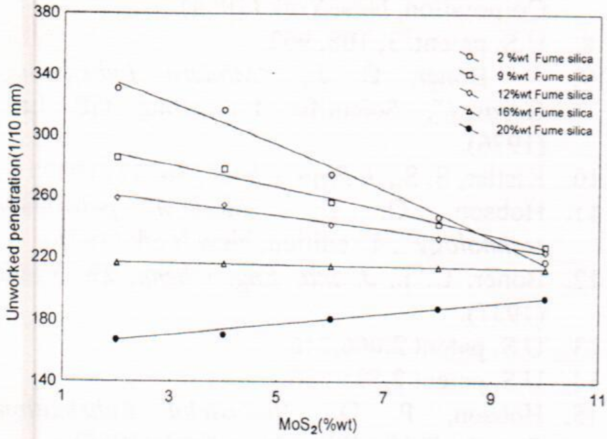

Figure 6. Effect of molybdenum disulfide on penetration at constant concentration of iron octoate (0.2 %wt.).

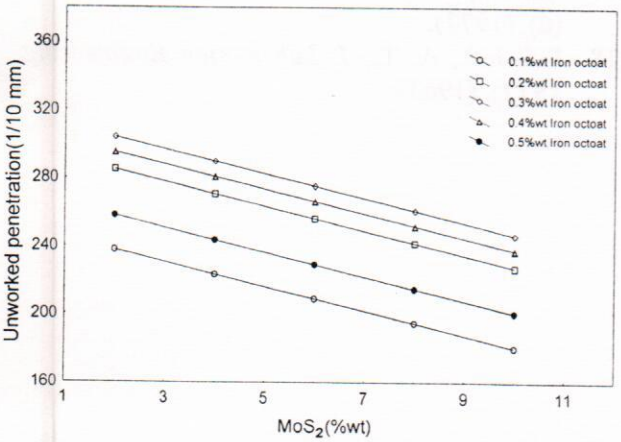

Figure 7. Effect of molybdenum disulfide on penetration at constant concentration of fume silica (9 %wt.).

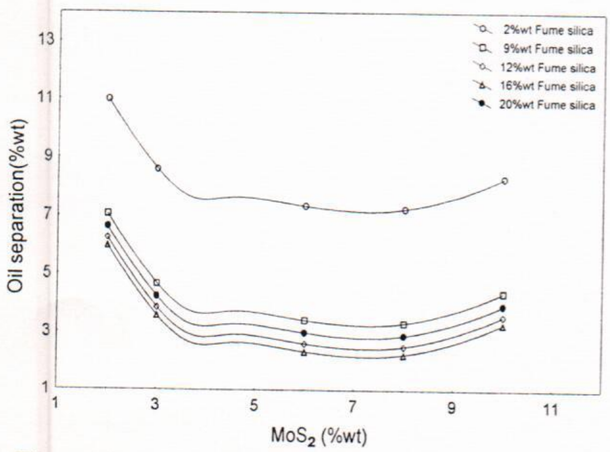

Figure 9. Effect of molybdenum disulfide on oil separation at constant concentration of iron octoate (0.2%wt.)

Figure 6 to 8 indicate that 3 %wt of molybdenum disulfide percent is required to obtain 4.7 %wt oil separation and 277 penetration.

Properties of the Formulated Grease

The multi-purpose formulated grease with desired properties can be obtained from mixed 87.8%wt poly methyl siloxan with 9%wt fume silica, 0.2%wt iron octoate and 3%wt molybdenum disulfide.

Table 2 shows the properties of the formulated grease and standard properties of AEROSHELL 22E. It can be seen that, from table 4 that some properties of formulated grease are showen a close agreement with standard properties of AEROSHELL grease 22E.

The drop point of formulated grease (340oC+) is higher than desired and that is better than standard properties. This means that the structure of formulated grease remains stable until the temperature rises so high that either base oil or thickener decomposes.

However the drop point are useful for the oxidation stability identification, where table 2 showed higher drop point compared with AEROSHELL grease 22E and this due to stabilization of the poly methyl siloxan with iron octoate.

Table 2. Comparison the properties of formulated grease with Aeroshell 22E grease.

Property	*Formulated grease*	*Aeroshell 22E grease*
Drop point (°C)	340⁺	260⁺
Worked penetration at 25°C (10^{-1}mm)	277	275
Unworked penetration at 25°C (10^{-1}mm)	274	271
Bomb oxidation pressure drop at 99°C for 100 h(psi)	4	5.5
Oil separation at 177°C and 3 h (%wt)	4.7	4.7
Copper corrosion 24 h at 100°C	Passes	Passes
Bearing protection 2 days at 52° C	Passes	Passes

CONCLUSIONS

1. For formulated grease the oil separation decreases with fume silica in the range of 2-20 % wt. and 2-10 % wt. for molybdenum disulfide. While unaffected by iron octoate concentration in the range 0.1-0.5 % wt.

2. The experimental study shows the final formula of grease contains 9 %wt fume silica, 0.2 %wt. iron octoate, 3 %wt. molybdenum disulfide and 87.8 % wt. polymethyl-siloxan.

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