Journal of Applied Sciences

ISSN 1812-5654

Journal of Applied Sciences 8 (4): 687-691, 2008
ISSN 1812-5654

Fume Silica Base Grease

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Abstract: In the present investigation, the effect of base oil viscosity and fume silica concentration on properties of formulated greases has been investigated. Mixture of greases is formulated using poly-methylsiloxane (with viscosity 500, 1000 and 2000 cS at 25°C) as base oil and thickened with 5-13% wt. fume silica. The results of testing have been done on formulated grease (penetration test, oil separation test, drop point test, corrosive substance, stability of the consistency at high temperature test) show that the increase in the fume silica concentration at same base oil viscosity increases the consistency of formulated grease, while the oil separation rapidly decreases by increasing the fume silica concentration at same oil viscosity. After 10% wt. addition of fume silica, oil separation becomes approximately unchanged. The result of the study of the effect of the base oil viscosity on the oil separation and consistency of formulated grease using 10% addition of fume silica shows that oil separation decreases and consistency increase with viscosity. No drop point of formulated greases was discovered (340°C$^+$) and this means that the structure of formulated grease remains stable.

Key words: Fume silica, greases, penetration, consistency, oil viscosity, oil separation

INTRODUCTION

The basic definition of Lubricating grease as mentioned by Jolicoeur (1992) is a semisolid product resulting from the fine dispersion of a thickening agent in lubricating oil. 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 (Lubrizol Corporation, 2000).

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 grease and are mostly composed of lubricating oil, which would normally have been employed, together with a gelling agent, which lends stiffness to the mixture.

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 (Grandou and Masson, 1996).

During the past time, several investigators did their work on improving and studying the properties of the greases used in lubrication in the industry (Adhvaryu *et al*., 2005) discussed the preparation of lithium soap-based soy greases using different fatty acids and the determination of crystallite structure of soap using Transmission Electron Microscopy (TEM). Lithium soaps with palmitic, stearic, oleic and linoleic acids were synthesized and mixed with Soybean Oil (SBO) and additives to obtain different grease compositions. His TEM measurements have revealed that the soap crystallite structure impact grease consistency. Soap fiber length and their cross-linking mechanism in the matrix control grease consistency (National Lubricating Grease Institute (NLGI) hardness, ASTM D-217 method). He showed that Lithium stearate-based soy grease has a relatively more compact fiber structure than Li palmitate. Linoleic acid with two sites of C-C in saturation in the chain has a much thinner and more compact fiber network than oleic acid (C18). The presence of additive in grease produces soap with looser network and larger fiber structure than similar grease containing no additive.

Franco *et al*. (2005) evaluated the manufacture of lubricating greases through the mixing rheometry technique, by studying the effect of some processing

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variables such as rotation speed, intensity and duration of the homogenization treatment and thermal profile applied, in terms of the power-draw characterization and rheological behavior of the final product. With this aim, their lithium lubricating greases were prepared by inducing the saponification reaction between 12-hydroxystearic acid and hydrated lithium hydroxide within a naphtenic lubricating oil medium in an open vessel. The saponification reaction occurred until neutralization by stirring with a controlled-rotational speed mixing rheometer using an anchor impeller. Different rotational speeds were selected. Finally, a highly intensive homogenization treatment was applied using a rotor-stator turbine in order to reduce crystal sizes. Different homogenization treatments and cooling profiles were applied on the incipient greases.

The American air force desired a lubricating grease to operate for several hundred hours at 204-232°C and yet be suitable for use at temperatures as close as possible to -53°C. Lubricating greases were therefore made, using silicon oil with a flash point of 316°C and pour point of -46°C. When operated in bearing at 232°C, a product containing 10% of silica as a thickener failed in 13 h and this is the applications of silica thickened greases have been used in aircraft.

Number of investigators has made claims for the use of silica and other materials for thickening lubricating fluids. Fume silica 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.

Generally, fume silica is a simple mixture of approximately 8% of fume silica gel with non-polar oil will yield grease of good consistency but with a matte surface.

Greases based on organosiloxane fluids (such as poly methylsiloxan) thickened with fume silica are stabilized on storage and against the effects of heat. Fume silica particles (thickening agent) hold the fluid by adsorption (Murray, 2000; Avci, 2003). Electron micrograp hs of silica bodied lubricating fluids show aggregates of more or less rounded particles of the thickening agent. The adsorption of fluid by myriad of such particles may be responsible for holding the fluid when fine silica is used for thickening lubricating oil.

Fume silica greases are useful in special high temperature application (Billelt, 1979). The upper limits are for the thickeners are determined by phase transformations of thickener, which cause modification in the grease structure. However, the presence of mineral oil limits their upper temperature use to approximately 200°C.

Fume silica provides gellants that form greases having dropping points about 260°C. Due to lack of phase changes of such gellants, they form lubricants with lower torque than those containing soaps when subjected to low temperature. Fume silica-base greases have very good performance characteristics, good mechanical stability and good water resistance and depend on the liquid phase good resistance to both high temperature and oxidation.

In the present investigation, it is desired to study of the effect of mixing time and process temperature on formulated grease consistency and the effect of fume silica concentration and base oil viscosity on grease yield and properties are performance.

MATERIALS AND METHODS

The experimental work was hold in a simple rig consists of stainless steel beaker of one liter with the agitator and electrical heater with temperature control.

The fluids employed for the grease formulation are essentially a linear siloxane polymers which soluble in ordinary organic solvents and with a formula R3SiO $(R_2SiO)_nSiR_3$. Table 1 shows the properties of poly-methylsiloxanes.

The thickening agent employed in the grease formulation is fume silica having surface area of 80 $m^2\ g^{-1}$, with average particles size of 8 A°.

Grease formulation: The samples of greases have been formulated from fluids with viscosities of 500, 1000 and 2000 cS at 25°C and different percentages of fume silica (5-13% wt.). In the first step of formulated grease, the poly-methyl siloxan heated to the proper temperature and then the fume silica added incrementally as rapidly as it could be incorporated into oil. During the process of fume silica addition the mixing was continued at and the desired temperature were maintained at 90°C. 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, to ensure good dispersion of the mixed components.

Test method: Tests used in evaluation of greases are of value for the purpose of determining the uniformity of the product and also for the suitability of the properties for

Table 1: Properties of poly-methyl siloxan

Viscosity at 25°C (cS)	Pour point (°C)	Flash point (°C)	Specific gravity at 25/4°C
500	-50	316	0.972
1000	Less than-50	316	0.973
2000	Less than-50	316	0.973

general or specific applications. These tests are penetration, corrosive substance (ASTM, 1984), oil separation, drop point (ASTM, 1984) and stability of the consistency at high temperature.

Stability of the consistency at high temperature is tested by applying a 2 mm film of the past onto a piece of metal to be tested and setting the piece of metal thus coated perpendicularly and heating it for 60 min at 200°C. If the paste does not appear to run off this piece of metal, the stability of the consistency is satisfactory.

RESULTS AND DISCUSSION

Effect of mixing time and process temperature on formulated grease consistency: The mixing time and process temperature are the significant processing variables, therefore their effect on grease formulation properties were studied.

Figure 1 show the effect of mixing time on the consistency at 10% addition of fume silica. The penetration of formulated grease with 10% fume silica had a minimum value at 30 min. The increasing the mixing time increases gradually the penetration and the penetration reaches the maximum value at 1.5 h. This may be due to the wetting of the agglomerate of fume silica particles by fluid with increasing the time and at about 90 min of mixing the wetting is completed.

Figure 2 show that the temperature increasing increases the penetration. After 90°C the increasing of penetration is very small. This observation is in good agreement with the results of (Milberger, 1957) which consider that the only significant processing variables when they employed utilized scraping blade and contra-rotating paddle action to prepare greases from mineral oil and silica gel, were mixing time and temperature.

Figure 2 and 3 allows to select that the best mixing time and process temperature for grease formulation in this investigation 1.5 h and 90°C, respectively.

Effect of fume silica concentration on consistency and oil separation: 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 (Boner, 1954).

Figure 3 clarifies the effect of fume silica percentage on penetration for base oil with 1000 cS at 25°C. Generally, the adding of fume silica significantly decreases the penetration (all penetration measurements are in an inverse scale of consistency, that is, the softer the consistency, the higher the penetration number). Further decreasing in penetration was observed by increasing fume silica percentage. This may be 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. This agrees with Kistler. The increase of silica concentration will tend to increase the stiffness of mixture (grease).

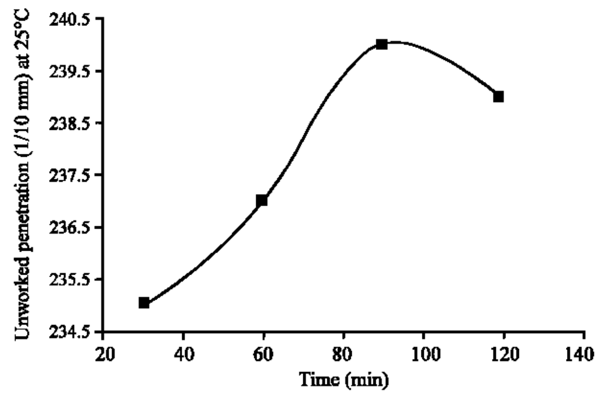

Fig. 1: Effect of mixing time on the consistency at 10% addition of fume silica

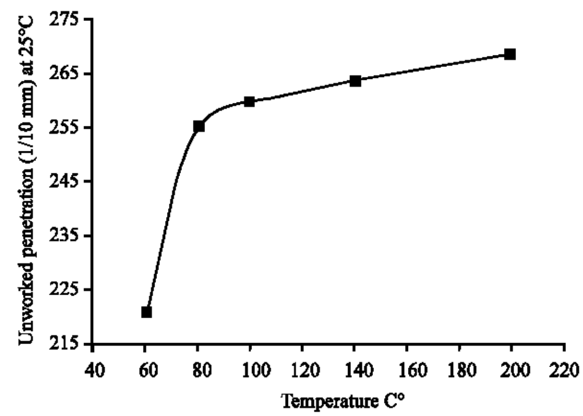

Fig. 2: Effect of temperature on the penetration at 10% addition of fume silica

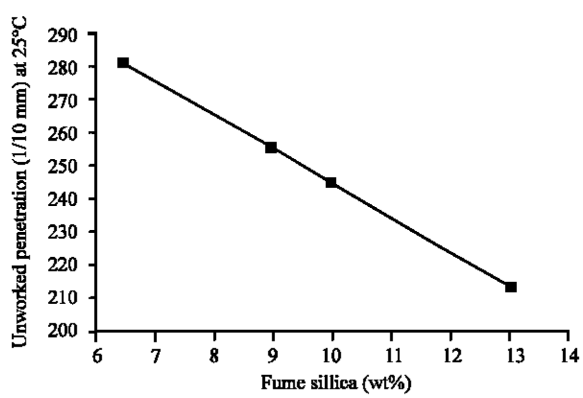

Fig. 3: Effect of fume silica percentage on penetration for base oil with 1000 cS at 25°C

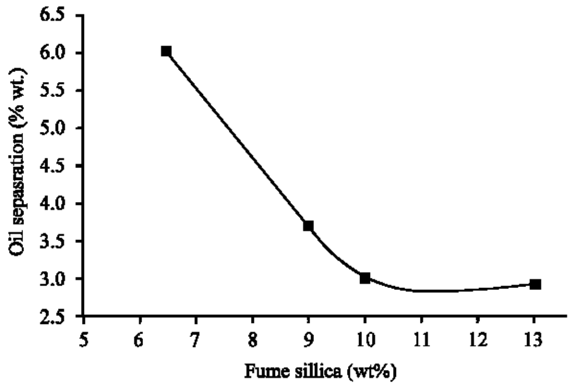

Fig. 4: Effect of the percent of fume silica on oil separation using poly-methyl siloxan with viscosity 1000 cS at 25°C

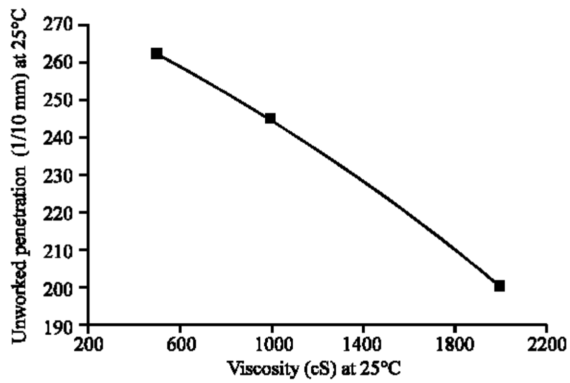

Fig. 5: Effect of fluid viscosities on the penetration

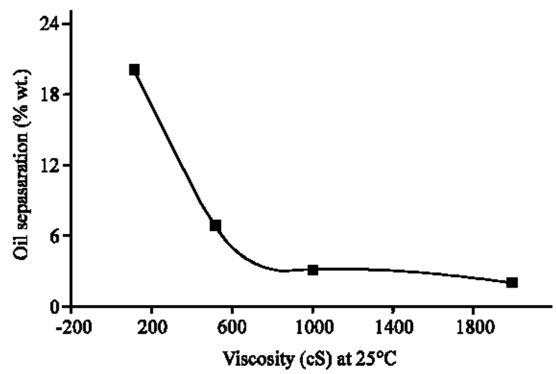

Fig. 6: Effect of viscosity on oil separation

Figure 4 shows the effect of the percent of fume silica on oil separation using poly-methyl siloxan with viscosity 1000 cS at 25°C. It can be noticed that by increasing the percentage addition of fume silica from 5 to 10% wt. decreases rapidly the oil separation. After 10% wt. of fume silica addition, 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 and this also, indicated by Boner who 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 also, he said that the fume silica particles have greater attraction for each other than for oil.

The drop point of formulated greases with concentration of fume silica less than 5% wt. is 260°C. At higher concentration the formulated grease shows no drop point (340°C$^+$) and there is no effect of increasing the fume silica concentration on drop point. The consistency stability for formulated grease is satisfactory at concentration higher than 5% wt. of fume silica.

The corrosive substance test is intended for the detection of corrosion caused by grease. The results of corrosive substance test show that the formulated greases have no negative effect on the metals surface. So the formulated grease passes the corrosive substance test.

Effect of the viscosity of base oil on consistency and oil separation: Figure 5 show the effect of fluid viscosities on the penetration. Within the viscosity range studied, the highest viscosity oil (2000 cS at 25°C) requires the minimum quantity of fume silica concentration for a given grease penetration and that due to the higher deviation from Newtonian flow for fluids with higher viscosity (above 1000 cS). The apparent viscosity of the fluid with higher viscosity under shear decreases as shear rate increases.

On the other hand, oil separation was found to vary with the viscosity of the oil employed. It is evident from Fig. 6, that a poly-methyl siloxan with viscosity of 1000 cS at 25°C or higher must be used to obtain the minimum bleed specified.

CONCLUSIONS

Significant processing variables were mixing time and temperature. It was found that the best mixing time and process temperature for grease formulation in this investigation 1.5 h and 90°C, respectively.

- The increasing in fume silica percent in formulated grease significantly decreases the penetration of formulated grease. Oil separation decreases with fume silica addition in the range of 5-10% wt. After 10% wt. of fume silica, oil separation becomes approximately unchanged.

- From greases have been formulated from fluids with viscosities of 500, 1000 and 2000 cS at 25°C and different percentages of fume silica (5-13% wt.), it was found that the highest viscosity oil requires the least fume silica concentration for a given penetration.
- Oil separation of formulated grease was found to decrease with the increases of fluid viscosity. That a poly-methyl siloxan with viscosity of 1000 cS at 25°C or higher must be used to obtain the minimum bleed specified.
- The formulated greases using 5% wt. fume silica addition and above has no drop point and its structure remains stable up to 340°C+. Also, the results of corrosive substance test show that the formulated greases have no negative effect on the metals surface.

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