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COMPLEX SOAP GREASES

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The present invention relates to complex soap greases, and particularly to lubricating greases using a new type of complex soap as the thickening agent. The invention also relates to a process of preparing such greases, and more particularly to a process by which voluminous precipitates of a complex structure may be converted into lubricating grease compositions by exchanging a lubricating oil for the normal water content of the precipitate.

It has been suggested in the prior art, that certain inorganic gels, and the like, might be used as the thickening agent in lubricating greases by exchanging a mineral base lubricating oil for the normal water content of the inorganic gel. The present invention is an improvement over such prior art and involves the use of specific types of precipitates formed from combinations of salts and soaps which for convenience may be referred to as "complex" polyvalent metal salts of fatty acids and dibasic acids.

According to the present invention, a lubricating grease of good texture and superior stability may be prepared by forming a combination of salt and soap of a polyvalent metal or a complex salt which is first prepared as a voluminous aqueous precipitate, thereafter exchanging the solvent (water) of the precipitate with an oil miscible solvent which preferably is readily volatile, and finally replacing the volatile solvent with lubricating oil, preferably mineral base lubricating oil. The composition prepared as above is fairly stable, even in the presence of moisture, but it may be rendered more stable by waterproofing it with a small amount of a suitable waterproofing material, for example by combining with the complex salt a small amount such as 0.1 to 2% by weight, based on the total composition, of an appropriate resin, e. g. an alkyd resin.

In broad terms, the invention contemplates a lubricating grease composition, consisting of a suitable lubricating oil which may be either a synthetic oil or a petroleum oil, or a mixture thereof, said oil being thickened to grease consistency with about 5 to 20% by weight, based on the total composition, of the complex salt obtained by combining, for example, about one molar part of an aluminum soap of C_{12} to C_{24} fatty acid with about 3 to 15 molar parts of C_2 to C_6 dibasic carboxylic acid. In lieu of aluminum, other polyvalent metals may be substituted such as calcium or barium. Specifically, a preferred form of the composition of the present invention is a grease prepared from mineral lubricating oil of appropriate viscosity, e. g. 35 to

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1000 S. S. U. at 100° F., thickened with 5 to 20% by weight, based on the total composition, of the complex salt made by combining the aluminum salts of stearic acid and succinic acid. In molar proportions, the succinic acid should predominate, 4 to 10 mols of the dibasic acid for each mol of fatty acid being preferred. In general, any one or more of the C_{12} to C_{24} fatty acids may be used, preferably the saturated fatty acids. The dibasic acid may be any of the dibasic acids of 2 to 6 carbon atoms, but the saturated dicarboxylic acids such as malonic, succinic, glutaric and adipic acid are preferred.

In the preparation of such grease compositions, the complex soaps described above are formed by saponifying a previously prepared mixture of polybasic and mono-basic acids of the type described above with an alkali such as sodium hydroxide or potassium hydroxide. The molar ratios of polybasic acid to mono-basic should correspond to the ratios of the salts desired in the complex, i. e. from 3 to 15 molar parts of polybasic acid for each molar part of the monobasic acid.

After the complex or mixed salts of suitable alkali metals are thus prepared, these are doubly decomposed by using an aqueous solution of a suitable salt, e. g., sulfate, of the desired polyvalent metal, such as aluminum, calcium or barium. Aluminum is preferred. The resulting complex salt is immediately precipitated as an aqueous gel and may be separated from other ingredients by decanting. The precipitate is then washed free of soluble matter and it may be dried, if desired. It is preferably solvent exchanged without drying, using a hydrocarbon miscible solvent such as acetone. This, in turn, is solvent exchanged with the selected lubricating oil. A volatile solvent is preferred so that it can be readily removed by evaporation when the lubricating oil is introduced into the gel.

The use of the oil miscible solvent is not always necessary since, if the correct ratios of acids are used, the dried soaps may be dispersed directly in hydrocarbon oils by heating in the conventional manner in which lubricating greases are prepared. For this purpose, the molar proportions of polybasic acid should be between 4 and 10 for each mol of the fatty acid. In other proportions, direct dispersion may be difficult. The resulting grease, produced by direct dispersion of the dried soap, has a high melting point and very good water resistance. The water resistance may be improved by adding a waterproofing resin such as 0.1 to 2% by weight, based

on the total composition, of a suitable alkyd resin, as disclosed in a copending application filed December 18, 1948, Serial No. 66,130.

Where the solvent exchange method is used, the soap and salt mixture or complex in which the water has been replaced by acetone, ether, or the like is finely dispersed in lubricating oil by any suitable method. Preferably, from 5 to 20 parts by weight of the soap, on a dried basis, not counting the weight of acetone or other solvent, are combined with 78 to 95 parts by weight of oil. The mixture or dispersion is then heated to a temperature above the boiling point of the solvent which is then flashed off and the grease is finished by subjecting it to severe mechanical working under high shear conditions, preferably in a colloid mill. Small quantities of an alkaline material such as lithium or hydroxide can be added to the grease if it is desired to neutralize any excess acidity. As suggested above, the use of a small amount of a waterproofing agent such as a modified alkyd resin is usually beneficial not only as a waterproofing material but also to prevent a tendency of the oil to bleed from the complex soap.

Greases prepared by either method, direct dispersion or solvent exchange, show desirable characteristics. Thus, certain samples prepared from 10% by weight of the aluminum salt mixture or complex soap of stearic acid and succinic acid with 90% of a mineral oil of 300 S. S. U. viscosity at 100° F. and 95 viscosity index not only have a high melting point but also pass the water resistance test, as described in N. R. C. Report No. C34-47S after 50 strokes in a standard grease worker. According to this test, a 5 gram sample of grease is placed in a 50 cc. grease worker fitted with a plunger having a single orifice of $\frac{1}{16}$ " diameter. 40 cc. of water are added and the grease and water are worked and agitated together for 50 strokes at 70° C. temperature. If no separation of grease components occurs the grease is considered to be water resistant. While mineral oils are preferred, the synthetic oils, either ester type or polyglycol or glycol ether type may be used alone or in combination with each other and/or with mineral oil.

The melting point of the final grease product, and the ease with which the complex soaps may be dispersed in the oil, may be controlled by varying the molar ratio of mono-basic to polybasic acid radical. In general, oil solubility is improved by increasing the proportions of the fatty acid soap but the thickening power and the melting point of the product tend to increase with larger proportions of salts of the polybasic acids.

The invention will be further explained by the following examples:

Examples

(1) Ten molar parts of succinic acid and one molar part of stearic acid were saponified with aqueous potassium hydroxide. The aluminum salt was next formed by the addition of aluminum sulfate solution. The washed precipitate was then solvent exchanged with acetone and the exchanged precipitate was added to a 300 S. S. U. viscosity 95 viscosity index mineral oil in the quantity of 10 parts by weight of dried salt complex to 90 parts of oil. The acetone was then flashed off and the resulting mixture was sheared and homogenized by passing it through a colloid mill.

This grease passed the water resistance test and had no melting point that could be determined.

(2) An aluminum complex salt similarly prepared by using a molar ratio of 4:1 succinic acid to stearic acid, was washed free of solubles with water and dried. Ten percent by weight of this dry complex was dispersed with heat into a mineral oil of 300 S. S. U. viscosity and 95 viscosity index, the oil also containing 0.4 percent by weight of lithium carbonate. This composition produced a grease having a melting point of about 150° C. This grease also passed the water resistance test.

(3) An aluminum complex salt was prepared as above, using a molar ratio of 10:1 succinic acid to behenic acid (C₂₂). The solvent exchanged complex salt so produced was incorporated into 300 S. S. U. viscosity 95 viscosity index oil producing a grease composition having a melting point of 55° C. for 8% concentration and 70° C. for a 10% concentration of salt. This grease also passed the water resistance test described above.

(4) An aluminum complex salt was prepared as above, using a molar ratio of 10:1 adipic acid to stearic acid. The grease prepared from this complex salt after solvent exchanging the complex and adding it to the 300 S. S. U. oil described above had a melting point of 70° C. for 8% concentration of salt.

What is claimed is:

1. A lubricating grease composition consisting essentially of a mineral base lubricating oil thickened to a grease consistency with 5 to 20% by weight, based on the total composition, of a complex polyvalent metal salt of one molar part of C₁₂ to C₂₄ fatty acid and 3 to 15 molar parts of C₂ to C₆ dibasic carboxylic acid, said salt having its normal water content replaced by said oil to form a stable gel structure.

2. Composition according to claim 1 in which the complex salt is an aluminum salt.

3. The process of preparing a grease which comprises combining one molar part of C₁₂ to C₂₄ fatty acid with 3 to 15 molar parts of C₂ to C₆ dibasic carboxylic acid, saponifying said acids with an alkali to form a complex alkali metal salt, treating said complex alkali metal salt with a polyvalent metal inorganic salt in solution to substitute said polyvalent metal into said complex salt and form a precipitate thereof, solvent exchanging said precipitate with a volatile oil-miscible organic solvent, mixing said solvent exchanged precipitate with a lubricating oil, and flashing off said solvent to form a gel.

4. Process according to claim 3 wherein said oil is mineral base lubricating oil.

5. Process according to claim 3 wherein said solvent is acetone and is flashed off after incorporating the oil.

6. Process according to claim 3 wherein said polyvalent metal salt is obtained by first forming the complex salt with an alkali and thereafter conducting an ionic exchange to substitute said polyvalent metal.

7. The process which comprises combining one molar part of stearic acid with 4 to 10 molar parts of succinic acid, saponifying said combined acids with aqueous potassium hydroxide to form a potassium complex salt, treating said complex salt with an aqueous solution of aluminum sulfate to form a complex aluminum salt precipitate, treating said precipitate with acetone to solvent exchange said acetone into said precipitate, adding 78 to 95 parts of mineral base lubricating oil of 100 to 1,000 S. S. U. viscosity at 100° F. to said solvent exchanged precipitate, and

flashing off volatile constituents to convert said precipitate and oil to a grease.

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