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METHOD OF PREPARING LITHIUM BASE GREASES

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This invention relates to improvements in the methods of preparing lithium base greases and particularly the lithium base greases adapted for the lubrication of aircraft control bearings at temperatures approximating —100° F.

In general, lithium base greases, prepared from saturated fats or fatty acids, possess a high dropping point and are water-resistant. These properties are particularly desirable in that they permit lubrication over a wide temperature range and minimize the solubilizing or leaching effect of water and moisture condensation on the grease. However, there are certain difficulties associated with the production of these greases, together with certain undesirable performance characteristics, which prevent any large scale acceptance thereof as a commercial lubricating grease.

In the preparation of the conventional lithium base grease, a slurry of a lithium soap, such as the stearate, which is either performed or prepared in situ, and a mineral oil, are heated to temperatures of around 360–500° F. until a homogeneous solution is obtained. This solution of lithium soap in oil is then drawn from the kettle and allowed to cool statically until a gel structure is set up. The cooled gel is then subjected to a milling operation where it is homogenized to a homogeneous grease composition. It becomes quite apparent that the efficiency and economies of such a method are quite poor, due to the high temperature operation, special equipment required, and the lack of control over the consistency and yield of the final product.

The disadvantages of these high temperature methods of preparation are particularly apparent in the preparation of so-called low temperature lithium base greases which are designed for the lubrication of ball and roller bearings, such as found in aircraft controls, at ultra low temperatures of the order of —60 to —100° F. In order to meet the requirements of low starting and operating torques at these low temperatures it is necessary to use as the mineral oil component an oil possessing a viscosity in the range of 40–70 S. U. S. at 100° F. These low viscosity oils also possess a low flash point which is within the temperature range required by the methods of manufacture used in the preparation of the greases. Since the preparations usually are carried out in fire-heated kettles in order to achieve these temperatures, it is easily recognized that a fire hazard exists. This merely increases the difficulties associated with the preparation of the lithium base greases and requires additional

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safety equipment to insure adequate ventilation during the preparation of the grease and prevent flashing of the volatilized mineral oil.

In accordance with the present invention it has been found that lithium base greases may be prepared by a process which involves temperatures attainable in the usual steam-heated kettle, does not require gelling and homogenizing equipment, and provides an improved control over the consistency and yield of the finished grease. This process is particularly applicable to the preparation of the low temperature lithium base greases in that the temperature required in the process are well below the temperatures at which these oils vaporize to any appreciable extent and thereby further obviates the requirement of the special safety equipment and the precautionary measures normally associated with the preparation of these greases.

It has been discovered that by using a soap-forming hydroxy fatty acid as the acidic component of the lithium soap, it is possible to prepare lithium base greases in accordance with an improved and simplified low temperature method of manufacture. The essential features of the process of the invention include saponification of a soap-forming hydroxy fatty acid with a basic lithium compound, dehydrating the reaction mass at temperatures of at least 270° F., and adding an oleaginous vehicle to obtain the desired consistency. This method of manufacturing lithium base greases is peculiar to the hydroxy fatty acids and is not applicable to the other fatty acids normally used in the preparation of grease compositions.

The hydroxy fatty acids contemplated by this invention are those containing twelve or more carbon atoms and one or more hydroxyl radicals separated from the carboxyl group by at least one carbon atom. In the following detailed description of the invention 12-hydroxy stearic acid is used as the representative soap-forming hydroxy fatty acid. It is to be understood, however, that the invention is not limited to this particular acid and that other soap-forming hydroxy fatty acids, or mixtures thereof, may be used in the preparation of lithium base greases by the low temperature method of manufacture. As an example, the hydroxy fatty acids produced by the catalytic oxidation of hydrocarbon oils and waxes which have been extracted and fractionated to the desired molecular range may also be used in the practice of the invention.

The type of lubrication for which the grease is intended has a direct bearing upon the selection

of the oleaginous vehicle or oil component to be used in the preparation of the grease. The oleaginous vehicles contemplated herein include the mineral lubricating oils, the synthetic lubricating oils prepared by cracking and polymerizing products of the Fischer-Tropsch process and the like, or the synthetic oleaginous compounds within the lubricating oil viscosity range. These synthetic oleaginous compounds are those organic compounds which possess lubricating characteristics and may be substituted in whole or in part for the conventional lubricating oils. Examples of these compounds are the aliphatic dicarboxylic acid esters, such as the alkyl esters of sebacic acid, the high molecular weight aliphatic ethers, such as normal hexyl ether, and the aromatic acid esters, such as the alkyl esters of benzoic or phthalic acids. Since the invention is particularly applicable to the preparation of low temperature lithium base greases, the preferred oleaginous vehicle is a mineral lubricating oil within the viscosity of 40–70 S. U. S. at 100° F. possessing a low pour point and a viscosity index of 30 or more. In general, the procedure involved in the preparation of these lithium base greases may be outlined as follows:

A steam-jacketed open kettle is charged with a mixture of lithium hydroxide and water and heated with stirring at 160–180° F. until all the lithium hydroxide is dissolved. The required amount of 12-hydroxy stearic acid and an equal amount of oil are then added and stirring is continued for 3–5 hours at 170–200° F. or until the hydroxy stearic acid is completely saponified. The saponification product is then heated to 270–300° F. and held at such temperatures until the soap mass becomes translucent, which usually requires from 2–5 hours, depending upon the temperature. The heat is then reduced and oil addition is begun. When the temperature reaches approximately 180–200° F. an oxidation inhibitor, if required in the final composition, is then added and thereafter oil addition is continued until the product possesses the desired consistency. The product is then drawn from the kettle as the finished lithium base grease.

A dehydration temperature of at least 270° F. is particularly desirable in the preparation of the greases of the invention, but it is preferable that dehydration temperatures of around 300° F. be used. At temperatures of around 270° F. it is necessary to heat for several hours in order to cause the soap mass to become translucent and lose its cloudy-white appearance, whereas at temperatures of around 300° F. a shorter dehydration time may be used. At temperatures below 270° F. the yields of the final grease fall off materially and an unsatisfactory product results.

In accordance with the invention it is also possible to prepare a lithium base grease by the low temperature method of manufacture, using a preformed lithium soap of a hydroxy fatty acid. In this modification the lithium soap, such as lithium hydroxy stearate, is charged to the kettle with a small amount of mineral oil. The temperature is gradually increased to around 300° F. with continuous stirring and periodic additions of the mineral oil. The temperature is then held at around 300° F. and the soap mass stirred until the mass is kneaded to a smooth plastic condition. Thereafter, the temperature may be gradually lowered and mineral oil added to obtain the desired consistency.

The following detailed example of the preparation of a low temperature grease illustrates the process of the invention:

A steam-jacketed open kettle was charged with 2.14 pounds of lithium hydroxide and 15 pounds of water. The temperature was gradually raised and the kettle contents stirred at 27 R. P. M. to dissolve the lithium hydroxide. At approximately 170° F. all the lithium hydroxide was dissolved, and at this point 22.5 pounds of a mildly-refined mineral oil and 15.0 pounds of 12-hydroxy stearic acid were added. The mineral oil used in this preparation possessed the following characteristics:

Gravity, °API	26.9
Flash, COC, °F	300
Fire, COC, °F	330
Vis. at 100° F., kinematic, cs	8.90
Vis. at 100° F. S. U. S. (converted)	55.1
Vis. index	44
Pour, °F	−55

The temperature was maintained at approximately 170–190° F. for a period of five hours. After this time saponification was substantially complete and the temperature then raised to 270° F. to effect dehydration. During the dehydration period stirring difficulties were encountered and additional mineral oil was added periodically. After approximately four hours at temperatures ranging from 270–285° F., the soap mass became translucent and the dehydration was complete. The temperature was then reduced and the mineral oil was gradually added. After 42.5 pounds of oil had been added the temperature was approximately 190° F. and at this point 0.33 pound of phenyl alpha naphthylamine, dissolved in oil, were added. Oil addition was then continued. After 50 pounds of oil were incorporated a one pound sample was removed for control penetration and free fatty acid determined (unworked =308 at 78° F., worked =350 at 78° F. and free fatty acid =0.10%). The temperature was again increased to 170° F. and 0.46 pound of granulated 12-hydroxy stearic acid added. Three pounds of mineral oil containing 0.5% phenyl alpha naphthylamine were then added as the temperature was decreased, and another one pound sample was removed for control penetration. At this point the unworked penetration was 293 at 78° F., and the worked penetration was 348 at 80° F. The grease was then drawn from the kettle at about 145° F.

The following analysis was obtained on the resulting grease composition:

Lithium soap, percent	23.2
Free alkali (LiOH), percent [1]	0.06
Free fatty acid (oleic), percent [1]	0.36
Free natural fat, percent	0.36
Dropping point, °F	376

[1] "The Institute Spokesman," National Lubricating Grease Institute, January, 1944. "Test Methods for Determining Free Acid and Free Alkali in Greases."

The low temperature method of manufacturing a lithium base grease from a hydroxy fatty acid glyceride, such as hydrogenated castor oil, is disclosed and claimed in our co-pending application, Serial No. 548,940, filed August 10, 1944.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof and, therefore, only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. A method of preparing a low temperature

gel-type lithium base grease containing a lithium soap of a soap-forming hydroxy fatty acid as the essential soap constituent thereof, said method enabling the use of temperatures attainable in a steam-jacketed kettle without fire hazard, which comprises saponifying with a basic lithium compound at a temperature within the range of about 170°–200° F. of a soap-forming hydroxy fatty acid in the presence of a small proportion of the oleaginous vehicle included in the grease, then raising the temperature of the saponified product to about 270–300° F. to dehydrate the product and render the same translucent, thereafter reducing the heat and adding the balance and large proportion of the oleaginous vehicle as the product cools and the desired consistency is attained, and finally drawing the resultant gel-type grease as a final product.

2. The method according to claim 1, wherein

the hydroxy fatty acid is 12-hydroxy stearic acid, and the oleaginous vehicle is a mineral lubricating oil having a viscosity within the range of 40–70 S. U. S. at 100° F.

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