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GEL-TYPE GREASE

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This invention relates to an improved lubricant. In one of its more specific aspects, it relates to a mixed soap base grease having good initial penetration and smoothness. In another of its more specific aspects, it relates to a lithium-potassium grease. In another of its more specific aspects, it relates to a method for preparing a mixed base grease.

In the grease-making art, two types of grease are commonly found. The two types of grease are fiber and gel greases. Each of these types of grease has its own particular application and characteristics. In making the gel-type grease, it has heretofore been quite difficult to obtain a grease which has good initial penetration while at the same time being smooth and homogeneous. It is desirable that a gel-type grease have temperature reversibility, i. e., the temperature properties of the grease not be dependent upon the prior temperature history of the grease nor the number of temperature cycles to which the grease has been subjected. When this requirement of temperature reversibility has been added to the other requirements for a grease, the problem of production of such a grease becomes even more acute. The additional problem of work stability of a grease, when combined with those set forth above, also adds untold difficulties to the production of a desirable gel-type grease.

Grease making, according to the prior art, usually consists of heating, in a large kettle, a mixture of saponifiable oil and an aqueous solution of a metal hydroxide in the presence of a mineral oil. During this heating, which requires from 8 to 24 hours, a soap is formed, most of the water is boiled off, and the soap becomes dispersed in the mineral oil to form grease which is usually diluted and gradually cooled by the slow incorporation of more oil. During the heating and blending process, the kettle content must be continually stirred to maintain intimate contact of the saponifiable oil and the aqueous alkali, to prevent scorching and secure a uniform product. During a considerable portion of this time the mixture is a stiff paste-like mass, the stirring of which consumes much power and requires the attention of a skilled grease maker. This method requires large floor space and expensive specialized equipment. The operation also lacks flexibility as small batches cannot conveniently nor cheaply be turned out in large kettles nor production changed from one type of grease to another in a short period of time. Use of large kettle equipment usually means that a large inventory of finished products is carried on hand.

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There have been numerous continuous processes for making grease described in the patent literature, but most of the workers have been interested in using the continuous process for saponifying the fatty oil and therefore use a temperature of about 200° F. to about 300° F. for a period of from 8 to 24 hours. Although the literature pertaining to greases contains many references relating to their preparation and which references usually contain directions for cooling and milling (working) many of the directions which have been recorded refer to greases which on cooling, or on cooling and slight working, give a homogeneous grease-like structure. The statements concerning cooling are often contradictory and inexact, depending on the type of soap present, and are usually qualitative statements unaccompanied by any supporting evidence of the extent or the effect of cooling. The prior art is voluminous with directions describing substances to add to soap and oil mixtures to stabilize the oherwise incompatible mixtures. The substances most often used as stabilizers are water, glycerin, unsaponified fat, unsaponifiable portions of fat, free fatty acid, high molecular weight alcohol, wool fat, pitch, high molecular weight amides, glycols, and the like. The art is very careful to teach that certain combinations of soap and oils alone are very difficult if not impossible to secure and to maintain in stable combination, and that stabilizing substances must be present to prevent syneresis, which in extreme cases forms an oil-soaked granular mass of soap in liquid oil. The fact that this breakdown of the structure occurs for many combinations of soap when heated in oil and cooled has been repeatedly demonstrated.

Broadly speaking, this invention comprises the production of a mixed metal soap grease composition and a method for the production of such a grease which grease has good initial penetration properties, together with smoothness and homogeneity, temperature reversibility, and good work stability. This grease is produced by the incorporation into a lubricating oil of an amount of a lithium soap, together with a smaller quantity of a potassium soap, and providing in such a mixture a small quantity of free fatty acid within a given carbon content range.

An object of this invention is to provide a rapid and unique process for producing greases of improved qualities. Another object of this invention is to provide a method for producing a lithium-potassium grease having desirable initial penetration properties. Another object of the invention is to provide a lithium-potassium grease

having improved work stability. Another object of the invention is to provide a grease which has good initial penetration properties and which is smooth and homogeneous without working. Another object of the invention is to provide a grease which has good initial penetration qualities and which is smooth and homogeneous with good work stability and temperature reversibility. Other and further objects and advantages of this invention will be apparent to those skilled in the art upon study of the accompanying disclosure.

It has previously been found that greases having good work stability can be produced by incorporating a metal soap in a lubricating oil at a temperature sufficient to cause the mixture to become homogeneous but below the decomposition temperature of the soap, and rapidly cooling the resulting mixture. The resulting product is a hard gel which crumbles and breaks but which, after considerable working, results in a grease of good work stability. In commercial operation it is desirable to eliminate the requirement for the working step because of the requirement for expensive machinery and equipment necessary to work the stiff gel mass to homogeneity.

I have found that by incorporating in a lubricating oil an amount of lithium soap having between 12 and 22 carbon atoms, together with a lesser amount of potassium soap having between 16 and 22 carbon atoms per molecule, and also with an amount of free fatty acid having between 12 and 22 carbon atoms per molecule, a grease which has desirable initial penetration values and which is smooth and homogeneous is produced without subsequent working. This grease has been found to have desirable work stability characteristics and temperature reversibility. The improvement of this grease over other greases utilizing free fatty acid has been found to be due to a cooperative effect of lithium soap with potassium soap in such a grease.

It is preferred that a lubricating oil having a viscosity index of at least 60 be used for the production of the grease of this invention. It is desirable that the lubricating oil be a hydrocarbon lubricating oil and preferably a mineral lubricating oil. The lubricating oil should have a viscosity at 100° F. of between 125 and 1500 SUS.

The amount of free fatty acid which is utilized in the production of the grease of this invention ordinarily ranges between 0.1 and 1.5 weight per cent and preferably less than 1.0 weight per cent of the total grease. This amount of free fatty acid is sometimes present when an unneutralized commercial soap is used in the formation of the grease. The exact amount of free fatty acid present in the reaction mixture may be adjusted to bring the acid content of the grease within the range of 0.1 and 1.5 weight per cent.

The total amount of lithium-potassium soap mixture which is incorporated into the lubricating oil is within the range of between 10 and 28 per cent by weight, based upon the final grease product. Between 10 weight per cent and 40 weight per cent of the total soap incorporated in the grease is a potassium soap having between 16 and 22 carbon atoms per molecule. The oil is present in the mixture in an amount generally within the range of 71 per cent to 90 per cent by weight of the final grease product.

The grease of this invention is produced by adding lithium soap having between 12 and 22 carbon atoms per molecule, together with potassium soap having between 16 and 22 carbon atoms per molecule, to a lubricating oil in such amounts that the quantity of soap is within the ranges set forth above. The metal soap is ordinarily a commercial soap which may be treated so as to provide a small amount of free fatty acid therein within the range set forth above. The mixture of soap and oil, together with the free fatty acid, is heated to a temperature of between 365° F. and the decomposition temperature of the soap, preferably between 400° F. and 475° F. The soap is incorporated in the oil during such heating and the resulting solution is then cooled to form the desired product. No agitation is required during the cooling although sufficient agitation may be utilized to facilitate the cooling of the solution.

Although the discussion heretofore has been limited to the formation of the grease of this invention with preformed soaps, the product is also produced by forming the soaps in situ. Free fatty acids rather than glycerides are used in this method so as to assure the absence of glycerine in the soap product. If neutral soaps are used, free fatty acid is added to the soap-oil mixture in the desired amount.

It is preferred that the lithium soap which is used be one with a fatty acid group containing at least 16 carbon atoms per molecule. A grease which is produced by incorporating the preferred lithium soaps, together with the potassium soaps and free acid in oil is homogeneous upon cooling and has for all practical purposes complete temperature reversibility. Greases made from lithium laurate or myristate do not always have complete temperature reversibility for there are in some cases two phases present when the greases are cooled from a relatively high temperature. Even in these cases, however, an improved grease is produced for the two phases resulting upon cooling of the soap-oil mixture become homogeneous with very little working. Thus the greases of this invention which are not substantially completely reversible with temperature, become homogeneous very rapidly under normal operating conditions and this lack of complete reversibility is insufficient to be objectionable. In the case of a grease such as one made by incorporating neutral lithium stearate in an oil without the modifying potassium soap and free fatty acid as disclosed in this invention, it is necessary to utilize extensive working in order to obtain a homogeneous grease because of the fact that the soap cake which is formed is quite hard. Better understanding of this invention will be obtained upon reference to the following specific examples.

SPECIFIC EXAMPLES

Several greases were prepared by incorporating soaps into a 1:1 blend of a solvent refined SAE 20 grade mineral lubricating oil and a solvent refined SAE 250 grade mineral lubricating oil. The properties of this blend were a viscosity (SUS) at 100° F. of 1050, viscosity (SUS) at 210° F. of 88, and a viscosity index of 95. The greases were prepared by heating the mixtures of oil and soaps and, where present, the free fatty acid to a temperature within the range of approximately 430° F. to 465° F. to produce a liquid solution. The solutions were then cooled without agitation. The greases were then worked in an ASTM grease worker using a worker plate containing 0.06 inch holes as described in Army-Navy grease specifications AN-G-25. Micropenetration results were obtained for the greases at intervals during the working.

A first grease was prepared by incorporating 16 weight per cent of a neutral lithium stearate soap in the oil described above. Results of the penetration tests which were obtained during working of the grease are set forth below in Table I.

A second grease was produced by incorporating 16 weight per cent of a lithium stearate soap and 1.11 weight per cent of free fatty acid having at least 16 carbon atoms per molecule in the above described oil at a temperature within the above designated temperature range. Upon cooling and working it was determined that this soap had desirable initial penetration characteristics but work stability of the grease was not satisfactory. Results of the penetration tests obtained during working of this second grease are set forth in Table I below.

A third grease was prepared by incorporating 16 weight per cent of lithium stearate soap, together with 6 weight per cent of potassium stearate soap, and 1.01 weight per cent of a free fatty acid containing at least 16 carbon atoms per molecule, in the lubricating oil described above and under the conditions set forth above. The resulting grease was found to possess desirable initial penetration qualities and upon working was found to have much better work stability than that of the second grease. Results of the penetration tests obtained during working of this grease are set forth below in Table I.

A fourth grease was prepared by incorporating 16 weight per cent of lithium stearate soap, together with 0.51 weight per cent of free fatty acid, in the lubricating oil described above and under those conditions set forth above. This grease was found to possess desirable initial penetration qualities but, like grease 2, did not possess satisfactory work stability characteristics. Results of penetration tests obtained during working of this grease are set forth below in Table I.

A fifth grease was obtained by incorporating 12 weight per cent of lithium stearate soap, 4 weight per cent of potassium stearate soap, and 0.28 weight per cent of a free fatty acid in the lubricating oil described above. The resulting product was found to possess desirable penetration qualities and upon working was found to possess very desirable work stability characteristics. Results of penetration tests obtained during working of this grease are set forth below in Table I.

TABLE I

Characteristics of greases having various compositions

	Penetration After Working for Indicated No. of Strokes, AN-G Worker Plate				
	1	60	200	2,000	5,000
Grease 1	.....	118	96	93	95
Grease 2	43	72	78	117	141
Grease 3	49	62	63	66	60
Grease 4	30	58	65	102	128
Grease 5	50	59	62	78	89

Study of the results of penetration tests obtained with grease 1 will diclose the fact that that grease does not possess desirable initial penetration characteristics. Such a grease does, however, possess very good work stability after the resulting gel has been worked to homogeneity. A study of the results obtained during penetration tests while working grease 2 and comparison thereof with the results obtained during tests

of grease 1 will disclose the fact that where grease 1 did not result in desirable initial penetration qualities, grease 2 did possess those desirable qualities. On the other hand, however, whereas grease 1 possessed excellent work stability, once the gel had been worked to a homogeneous form, grease 2 lost consistency with continued working of the grease.

A comparison of the results obtained upon testing grease 2 and grease 3 will disclose the effect of 6 weight per cent of potassium stearate upon the work stability of grease 3. Grease 2 and grease 3 had very comparable initial penetration qualities but upon working 5,000 strokes in an ASTM grease worker using an AN–G–25 worker plate, grease 3 showed very good work stability qualities while the work stability qualities of grease 2 were not satisfactory.

In order to determine whether or not the total soap content of the grease resulted in the improved quality of good initial penetration together with good work stability characteristics thereof or whether that improved quality was due to the presence of the potassium soap in admixture with the lithium soap, greases 4 and 5 were prepared. Each grease contained 16 weight per cent of soap incorporated in the oil, grease 4 containing 0.51 weight per cent free fatty acid and grease 5 containing 0.38 weight per cent of free fatty acid. Four per cent of the soap of grease 5 was potassium stearate which was incorporated in the oil with 12 weight per cent of lithium stearate to form the total of 16 weight per cent soap in the grease. Grease 4, like grease 2, possessed desirable initial penetration characteristics but also like grease 2 did not possess suitable work stability characteristics. Grease 5 had very comparable initial penetration characteristics with greases 4 and 2 but results of the penetration tests obtained during working of grease 5 disclosed the fact that the work stability of grease 5 is much better after 5,000 strokes of an ASTM grease worker than is the work stability of grease 4. From this comparison it is at once apparent that the potassium soap provides the unique characteristic of combined good initial penetration properties and improved work stability to the mixed soap grease of this invention.

When soaps are formed in situ, one method for adjusting the amount of free fatty acid in the soap is to add potassium hydroxide to the soap in an amount less than the stoichiometric amount required for complete neutralization. In this manner the acid content can be adjusted within the specific limits set forth above.

Various modifications of this invention will be apparent to those skilled in the art upon study of the accompanying disclosure. It is believed that such modifications are within the spirit and scope of this disclosure.

I claim:

1. An improved grease comprising between 71% and 90% by weight of a lubricating oil; between 10% and 28% by weight of a mixture of a lithium soap having between 12 and 22 carbon atoms per molecule and a potassium soap having between 16 and 22 carbon atoms per molecule, a major portion of the soap mixture being lithium soap and a minor portion of at least 4% by weight being said potassium soap; and between 0.1% and 1.5% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

2. An improved grease comprising between 71% and 90% by weight of a hydrocarbon lubricating oil having a viscosity index of at least

60, and a viscosity within the range of 125–1500 SUS at 100° F.; between 10% and 28% by weight of a mixture of a lithium soap having between 12 and 22 carbon atoms per molecule and a potassium soap having between 16 and 22 carbon atoms per molecule, a major portion of the soap mixture being lithium soap and a minor portion of at least 4% by weight being said potassium soap; and between 0.1% and 1.5% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

3. An improved grease consisting essentially of a hydrocarbon lubricating oil; between 10% and 28% by weight of a mixture of a lithium soap having between 12 and 22 carbon atoms per molecule and a potassium soap having between 16 and 22 carbon atoms per molecule, a major portion of the soap mixture being lithium soap and a minor portion of at least 4% by weight being said potassium soap; and between 0.1% and 1.0% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

4. An improved grease consisting essentially of a hydrocarbon lubricating oil; between 10% and 28% by weight of a mixture of a lithium soap having between 12 and 22 carbon atoms per molecule and a potassium soap having between 16 and 22 carbon atoms per molecule, a major portion of the soap mixture being lithium soap and a minor portion of at least 4% by weight being said potassium soap; and between 0.1% and 1.5% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

5. An improved grease consisting essentially of a hydrocarbon lubricating oil; between 10% and 28% by weight of a mixture of a lithium soap having between 12 and 22 carbon atoms per molecule and a potassium soap having between 16 and 22 carbon atoms per molecule, between 10% and 40% by weight of the total soap being potassium soap; and between 0.1% and 1.0% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

6. An improved grease consisting essentially of a hydrocarbon lubricating oil having a viscosity index of at least 60, and a viscosity within the range of 125–1500 SUS at 100° F.; between 10% and 28% by weight of a mixture of lithium stearate and potassium stearate, between 10% and 40% by weight of the total soap being potassium stearate; and between 0.1% and 1.5% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

7. An improved grease consisting essentially of a hydrocarbon lubricating oil having a viscosity SUS of about 1050 at 100° F. and a viscosity index of about 95; 16% by weight of lithium stearate; 6% by weight of potassium stearate; and about 1.0% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

8. An improved grease consisting essentially of a hydrocarbon lubricating oil having a viscosity SUS of about 1050 at 100° F. and a viscosity index of about 95; 12% by weight of lithium stearate; 4% by weight of potassium stearate; and about 0.4% by weight of free fatty acid having between 12 and 22 carbon atoms per molecule.

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