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PRODUCTION OF COMPLEX BASIC ALUMINUM SOAP GREASES

Theodore H. Koundakjian and John L. Dreher, Berkeley, Calif., assignors to Chevron Research Company, a corporation of Delaware

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This invention relates to a process for the production of complex basic aluminum soap greases such as greases in which the soap is basic aluminum benzoate stearate.

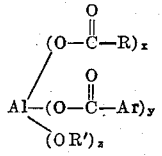
U.S. Patent 2,768,138 describes complex basic aluminum soap greases. In the examples of the patent the greases are prepared by first forming a complex aluminum soap and then dispersing the soap in hot oil to produce a grease. Complex aluminum soap greases prepared in this manner have not been notably successful because the basic complex aluminum soaps have very high melting points and it is difficult to obtain a good dispersion of the soap in the oil base without recourse to very high temperatures. If temperatures sufficiently high to melt aluminum soaps are employed, thermal degradation of the base oil and the soap becomes a problem and at lower temperatures incompleteness of the dispersion of the soap in the oil results in greases having considerably less than optimum properties.

Pursuant to the present invention, the basic complex aluminum soap is formed in the presence of the base oil by reacting a lower aluminum alcoholate with a mixture of a fatty acid, an aromatic carboxylic acid, and water, and excellent dispersions of the soap in the oil are obtained at moderate temperatures. In order, however, to obtain a grease by this method it is necessary that a substantial quantity of water be added to the reaction mixture after the reaction of the aluminum alcoholate with the acids is complete.

Pursuant to the invention approximately 1.5 to 2.5 molar parts of a mixture of higher fatty acids and aromatic carboxylic acids in which the mol ratio of higher fatty acid to aromatic carboxylic acid is in the range from about 0.25:1 to 4:1, approximately 1 molar part of a lower aluminum alcoholate, from 0 to 1.5 molar parts of water, and a large molar excess of an oil of lubricating viscosity, are heated to a temperature in the range about 150°–300° F. until reaction of the alcoholate with the acids is substantially complete, then a quantity of water in the range 0.05 to 10 molar parts and at least sufficient to bring the total water introduced to at least about 0.5 molar parts is added, and the resultant mixture is vigorously agitated at a temperature of from about 200° to 500° F.

In a preferred embodiment of the invention 2 molar parts of the mixture of fatty acids and aromatic carboxylic acids is reacted with one molar part of a lower aluminum alcoholate in the presence of a molar excess of an oil of lubricating viscosity without addition of any water. When the reaction between the acids and the aluminum alcoholate is complete as indicated by cessation of alcohol evolution from the mixture, from about 0.5 to 10 molar parts of water are added and the resultant mixture is vigorously agitated at a temperature of from 200° to 500° F. for at least one-half hour.

The basic complex aluminum soap may be described by the general formula:



in which R is an aliphatic group containing 8 to about 30 carbon atoms, Ar is an aromatic radical preferably a monocyclic hydrocarbon radical, R′ is either hydrogen or a lower aliphatic hydrocarbon radical and at least one-half of R′ is hydrogen, x is at least 0.25, y is at least .25, the sum of x and y is from 1.5 to 2.5 and z is from 0.5 to 1.5.

Suitable aluminum alcoholates for use in the preparation of the greases are those in which the alcohol precursor is ethyl alcohol, propyl alcohol, isopropyl alcohol, any of the butyl alcohols, or mixtures of the foregoing alcohols.

Suitable higher fatty acids for use in the preparation of the complex basic aluminum soap greases are caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, 12-hydroxy stearic acid, arachidic acid, melissic acid, oleic acid and linoleic acid. Phenyl-substituted fatty acids such as phenyl decanoic acid and naphthenic acids containing at least 12 carbon atoms may also be employed as the "fatty acid" reactant.

Aromatic acids suitable for use in the preparation of the basic complex aluminum grease are benzoic acid, the toluic acids, benzoic acids substituted with one or more ethyl, propyl or butyl groups, amino-substituted benzoic acids, chlorobenzoic acids and naphthoic acids. The oil component of the grease may be either a hydrocarbon oil of lubricating viscosity derived from petroleum or a synthetic oil as described in U.S. Patent 2,768,138.

The process for the production of the complex basic aluminum soap greases is carried out in two steps.

The first step is carried out at a temperature in the range 150°–300° F. and ordinarily at atmospheric or subatmospheric pressure to facilitate evolution of alcohol from the reaction mixture. The reaction mixture heated during the first step will contain approximately 2 molar parts of a mixture of fatty acid and aromatic acid, approximately 1 molar part of a lower aluminum alcoholate and the large molar excess of an oil of lubricating viscosity. The reaction mixture may or may not include water. If water is included the quantity of water may range from a very small amount up to about 1.5 molar parts of water. When one or more molar parts of water is present in the reaction mixture subjected to the first reaction step, substantially all of the aluminum alcoholate is decomposed and no aluminum alkoxy linkages are present in the reaction product obtained at the end of the first step.

The second step in the process is carried out at temperatures in the range of 200° to 500° F. and the reaction mixture is held at temperatures in this range usually for one-half hour or longer. Generally the reaction time in the second step will be in the range ½ hour to 5 hours and the time will vary inversely with the temperature. Usually about ½ hour is required for completion of the second step, but the upper limit of time is not critical to the properties of the final product. The pressure in the second reaction step is generally atmospheric, but higher or lower pressures are not precluded. After the heating at 200°–500° F. has been completed, the mixture is vigorously agitated as by circulation through a Charlotte mill and simultaneously cooled to a suitable packaging temperature usually in the range 150°–180° F.

It is necessary that water be added to the reaction mixture at the beginning of the second step. The quantity of water added must be at least 0.05 molar part and must be at least sufficient to bring the total water employed in the process in the two reaction steps to at least about 0.5 molar part and preferably to at least 1 molar part. Unless water is added at the beginning of the second reaction step, a liquid or semi-liquid product rather than a grease is obtained.

The process of the invention is illustrated by the following examples:

Example 1

46.2 grams (0.165 mol) of hydrogenated tallow acid, 20.1 grams (0.165 mol) of benzoic acid, and 200 grams of a solvent-refined paraffinic oil having a viscosity of 500 SSU at 100° F. were stirred together at a temperature of 200° F. for 30 minutes until the acids had dissolved. 33.6 grams (0.165 mol) of aluminum isopropoxide and 225 grams of oil were heated at 220° F. while stirring for 30 minutes until a clear solution resulted. The oil solution of the mixed acids was added to the solution of aluminum isopropoxide and a thick spruplike material formed with the rapid evolution of isopropanol. The mixture was heated at 300° F. for 40 minutes after which time all of the alcohol had been removed. The mixture was cooled to 200° F. and 2.9 ml. (0.165 mol) of water was added. This resulted in the liberation of additional alcohol, and a heavy gel was formed after heating to 290° F. 250 grams of additional oil was added, and this mixture was heated to a temperature of 400°–410° F. for 3½ hours. The resulting grease was still grainy. 400 grams of additional oil was added to 600 grams of this grease, and the resulting grease was milled. This resulted in a grease containing 5.8% thickener having the folowing properties:

ASTM dropping point, ° F.	470
P_0	265
P_{60}	293
$P_{100,000}$	376

Example 2

1820 grams of hydrogenated tallow acid, 793 grams of benzoic acid, and 20,000 grams of a solvent-refined paraffinic lubricating oil were charged to a kettle and circulated by means of a pump at a temperature of 180° F. until the acids had dissolved. 117 ml. of water was added. A solution of 1326 grams of aluminum isopropoxide in 9000 grams of oil was heated to 180° F. and added to the kettle. 1000 grams of oil was added to wash the lines, and the mixture was held for 30 minutes at 200° F. It was heated to 210° F. and 35 ml. of water was added. The mixture was heated to 330°–340° F. and stirred for 1 hour. It was recycled through a Charlotte mill at 300° F. and 800 grams of zinc oxide and 1000 grams of oil were added. The resulting grease had an unworked penetration of 233 and a worked penetration (60 strokes) of 292.

Example 3

A solution of 1820 grams of hydrogenated tallow acid and 793 grams of benzoic acid was prepared in 29,000 grams of a lubricating oil having a viscosity of 500 SSU at 100° F. This was heated to a temperature of 225° F. and 1326 grams of aluminum isopropoxide was added and the batch stirred for one-half hour. 152 ml. of water was added at a temperature of 220° F. and the mixture stirred for one-half hour. It was then heated to 400° F. and held for one-half hour and milled through a Charlotte mill. 800 grams of zinc oxide was added with stirring and 3500 grams of oil was added while milling. A smooth grease resulted.

Example 4

A solution of 2045 grams of hydrogenated tallow acid and 1074 grams of benzoic acid was prepared in 15,000 grams of white oil heavy at a temperature of 150° F. The mixture was heated to 190° F. and 144 ml. of water was added. 1632 grams of aluminum isopropoxide was dissolved in 7000 grams of white oil heavy and this solution was added to the oil solution of the acids at a temperature of 190° F. It was held at this temperature for one-half hour. An additional 15 cc. of water was added at a temperature of 338° F. and the grease gelled. It was cooled and recycled through a Charlotte mill. This grease had an unworked penetration of 264 and a worked penetration (60 strokes) of 310.

Example 5

A 1600 pound batch of grease was prepared by the process of the invention. The components of the batch were 64.8 pounds of stearic acid, 36.2 pounds of benzoic acid, 53.1 pounds of aluminum isopropoxide, 1320 pounds of white oil, 80 pounds of sodium benzoate, 32 pounds of zinc oxide, 8 pounds of sodium nitrite, and 6.2 pounds of water. The sodium benzoate stabilizes the grease at elevated temperature, the zinc oxide is a filler and coloring agent, and the sodium nitrite is a rust inhibitor. The stearic acid, benzoic acid and 150 gallons of white oil were charged to a mixer and heated to 220° F. with agitation. After the acids were dissolved, the solution was circulated from the mixer through a Charlotte mill and back to the mixer. The aluminum isopropoxide was added to the mixer over a 30-minute period while agitation in the mixer was maintained and circulation of the mixture through the Charlotte mill was maintained. The circulation and agitation were continued for 45 minutes while the temperature was maintained at 210°–220° F. At the end of 45 minutes, the 6.2 pounds of water were added and the resultant mixture was slowly heated to 320° F. During the heating the materials in the mixer were agitated, but circulation through the mill was discontinued. After the temperature of 320° F. was reached (approximately 1 hour), the reaction mixture was slowly cooled with continuing agitation to 230° F. During the cooling the remainder of the white oil was added. When the temperature of 230° F. was reached the sodium nitrite mixed with 3 gallons of water was added to the reaction mixture which was then re-heated to 260° F. and held at that temperature for about 30 minutes to dehydrate the reaction mixture. The reaction mixture was then cooled to 200° F. and the sodium benzoate and the zinc oxide were added. The resultant mixture was circulated through the Charlotte mill and slowly cooled to 160° F. When the temperature reached 160° F. it was withdrawn from the mixture and passed through a filter screen and packaged.

In the foregoing examples a large molar excess of oil was employed in each case. In general, satisfactory greases are obtained when the soap content of the grease is in the range about 4 to 16% by weight.

Example 6

1 molar part of benzoic acid and 1 molar part of hydrogenated tallow acid were added to a molar excess of a solvent-refined paraffinic lubricating oil having a viscosity of 500 SSU at 100° F. The mixture was heated until the acids dissolved and then 1 molar part of aluminum isopropoxide and 1.33 molar parts of water were added. The mixture was heated to 220° F. and agitated at that temperature for approximately 60 minutes. The mixture was then heated to 400° F. without the addition of more water and held at 400° F. for 30 minutes and then slowly cooled. The resultant product was a fluid, not a grease, even though its content of complex basic aluminum soap was 9.6% by weight, a quantity sufficient to give an excellent grease if water had been added during the second heating step pursuant to the invention.

We claim:

1. A process for producing complex basic aluminum soap greases, which comprises mixing approximately 1.5 to 2.5 molar parts of a mixture of higher fatty acids and aromatic carboxylic acids in which the mol ratio of higher fatty acid to aromatic carboxylic acid is in the range from about 0.25:1 to 4:1, approximately 1 molar part of a lower aluminum alcoholate, from 0 to 1.5 molar parts of water and a large molar excess of an oil of lubricating viscosity, heating the resultant mixture to a temperature in the range about 150° to 300° F. until reaction of the acids with the alcoholate is substantially complete, then

adding a quantity of water in the range 0.05 to 10 molar parts and at least sufficient to bring the total water introduced to about 0.5 molar parts and vigorously agitating the resultant mixture at a temperature from about 200° F. to 500° F.

2. A process for producing complex basic aluminum soap greases, which comprises forming a substantially water-free mixture of approximately 2 molar parts of a mixture of higher fatty acids and aromatic carboxylic acids in which the mol ratio of higher fatty acid to aromatic carboxylic acid is in the range from about 0.25:1 to 4:1, approximately 1 molar part of a lower aluminum alcoholate and a large molar excess of an oil of lubricating viscosity, heating the resultant mixture to a temperature in the range about 150°–300° F. until reaction of the acids and the alcoholate is substantially completed, then adding a quantity of water at least 0.5 to 10 molar parts, and vigorously agitating the resultant mixture at a temperature from about 200° to 500° F. for at least one-half hour.

3. The process as described in claim 1, wherein the mol ratio of higher fatty acid to aromatic carboxylic acid is approximately 1.

4. The process as described in claim 3, wherein all of the water employed in the process is added to the reaction mixture after the reaction between the acids and the aluminum alcoholate is complete.

5. A process for producing a grease, which comprises heating about one molar part of a lower aluminum alcoholate, 0.4 to 1.6 molar parts of a higher fatty acid, 0.4 to 1.6 molar parts of an aromatic carboxylic acid, the total molar parts of higher fatty acid and aromatic carboxylic acid being in the range 1.5 to 2.5 molar parts, 0 to 1.5 molar parts of water and a molar excess of an oil of lubricating viscosity to a temperature in the range about 150° F. to 300° F. until reaction of the acids and alcoholate is substantially complete, then adding a quantity of water in the range 0.05 to 10 molar parts and sufficient to bring the total water introduced to about 0.5 molar parts, vigorously agitating the mixture at a temperature from about 200° F. to 500° F. for at least about one-half hour, and then simultaneously vigorously agitating and slowly cooling the mixture to a temperature in the range about 150°–180° F.

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DANIEL E. WYMAN, *Primary Examiner.*

I. VAUGHN, *Assistant Examiner.*