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[54] **GREASE COMPOSITIONS**

[75] Inventors: **Joseph J. Dickert, Jr.**, Lower Makefield Township, Pa.; **El-Ahmadi I. Heiba**, Princeton, N.J.

[73] Assignee: **Mobil Oil Corporation**, New York, N.Y.

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[58] **Field of Search** 252/33

[56] **References Cited**

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Primary Examiner—Delbert E. Gantz
Assistant Examiner—I Vaughn
Attorney, Agent, or Firm—Charles A. Huggett; Raymond W. Barclay; Benjamin I. Kaufman

[57] **ABSTRACT**

Grease compositions are provided comprising a major proportion of an oil of lubricating viscosity and a thickening amount of a salt of a C-alkyl or alkenyl succinimidoarylsulfonic acid having from about 14 to about 30 carbon atoms in the alkyl or alkenyl group.

12 Claims, No Drawings

GREASE COMPOSITIONS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to grease compositions and relates more particularly to grease compositions containing, as thickening agents salts of C-alkyl or alkenyl succinimdoaryl-sulfonic acids.

2. Description of the Prior Art

The prior art has heretofore provided grease formulations containing various types of thickening agents, particularly greases thickened with animal or plant-derived fatty acid salts or with modified clays. It is found, however, that reliance on natural products is not always expedient and also that such thickening agents often fail to provide the complete range of thickening desired in commercial greases where the grease must undergo widely varying temperature changes during the course of operating conditions.

SUMMARY OF THE INVENTION

In accordance with the present invention, there are provided improved grease compositions comprising a major proportion of an oil of lubricating viscosity and a thickening amount of a salt of a C-alkyl (or alkenyl) succinimidoarylsulfonic acid having from about 14 to about 30 carbon atoms in the alkyl or alkenyl group. The presence of these thickening agents results in greases which can effectively perform their function over a wide temperature range and thus represent improvement over natural thickening agents, e.g. animal or plant-derived fatty acid salts or modified clays as heretofore employed.

A critical requirement of the above-described salts, as thickening agents, is that the C-alkyl or alkenyl succinimidoarylsulfonic acid contains from about 14 to about 30 carbon atoms in the alkyl or alkenyl group. If the alkyl or alkenyl group contains less than 14 carbon atoms, the resulting grease composition is not sufficiently stable to perform satisfactorily under conditions of application. On the other hand, if the alkyl or alkenyl group contains more than about 30 carbon atoms, the resulting thickener becomes excessively soluble in the oil and does not provide sufficient thickening power to obtain an effective grease.

Within the aforementioned limitations, various salts of C-alkyl or alkenyl succinimidoarylsulfonic acids, containing from about 14 to about 30 carbon atoms in the alkyl or alkenyl group may be effectively employed as thickening agents. Thus the salts may include salts of C-alkyl or alkenyl succinimidobenzenesulfonic acids or C-alkyl or alkenyl succinimidonaphthalenesulfonic acids, the sodium-lithium salt of C-n-octadecylsuccinimidonaphthalenedisulfonic acids, the sodium-calcium salt of C-n-octadecylsuccinimidohydroxybenzensulfonic acid, the triethylamine salt of C-n-docosenylsuccinimidohydroxybenzenesulfonic acid, the sodium salt of C-n-docosenysuccinimidohydroxybenzenesulfonic acid, or the lithium salt of C-n- docosenylsuccinimidohydroxybenzenesulfonic acid.

These salts of C-alkylsuccinimidoarylsulfonic acids may be synthesized by commercially well-known methods, in oil solution. Many metals may be effectively employed for synthesizing the desired metal salt and particularly include alkali or alkaline earth metal salts represented by lithium, sodium, potassium, rubidium, cesium, calcium, strontium or barium.

Oils employed in the greases of this invention can be mineral or synthetic oils of lubricating viscosity. Suitable mineral oils have a viscosity from about 45 SSU at 100°F. to about 6,000 SSU at 100°F. or a viscosity range from about 50 SSU at 210°F. to about 25 SSU at 210°F.

Synthetic vehicles can be used instead of mineral oils, or in combination therewith. Typical synthetics are: polypropylene, polypropylene glycol, trimethylol propane esters, neopentyl and pentaerythritol esters, di(2-ethyl hexyl) sebacate, di(2-ethyl hexyl) adipate, dibutyl phthalate, polyethylene glycol di(2-ethyl) hexoate), fluorocarbons perfluoroalkyl-polyethers, silicate esters, silanes, esters of phosphorus-containing acids, liquid ureas, ferrocene derivatives, hydrogenated mineral oils, chain type polyphenyl ethers, siloxanes, and silicones (polysiloxanes) fluorosilicones, alkyl-substituted diphenyl ethers typified by a butyl-substituted bis-(p-phenoxy phenyl) ether, and phenoxy phenyl ethers.

Other hydrocarbon oils include synthetic hydrocarbon polymers having improved viscosity indices, which polymers are prepared by polymerizing an olefin, or mixture of olefins, having from 5 to 18 carbon atoms per molecule in the presence of an aliphatic halide and a Ziegler-type catalyst.

It is to be understood, however, that the compositions contemplated herein can also contain other characterizing materials. For example, antioxidants such as phenyl alphanaphthylamine (PAN), corrosion inhibitors, extreme pressure agents, viscosity index agents, and fillers can be used. Among such materials are colloidal silica, and molybdenum disulfide. Such characterizing materials do not detract from the lubricating value of the compositions of this invention, nor do they detract from the beneficial character of the polymers; rather, the characterizing materials serve to impart their customary properties to the particular compositions in which they are incorporated.

The greases of this invention can be prepared in accordance with conventional grease manufacturing procedures, as by any mixing technique wherein solid particles are wetted by a fluid.

An essential characteristic of the above-described salts of the present invention is that these grease thickening agents have relatively short alkyl chains (viz. from about 14 to about 30 carbon atoms) attached to the alpha carbon atom of the succinimide. The sulfonic acid portion of the molecule may be mono- or polysulfonic, and may contain one or more rings either condensed or simmply attached linearly, and/or may be otherwise substituted (for example with OH^-, CL^- and the like).

The novel greases of the present invention, as hereinbefore indicated, may be prepared in accordance with conventional grease manufacturing procedures, or by any mixing technique in which solid particles are wetted by a fluid. The manufacture of typical representative greases, in accordance with the invention, is illustrated by the procedures of the following examples.

DESCRIPTION OF SPECIFIC EMBODIMENTS

Example 1

Preparation of the Imide

About 0.31 mole (about 125 g.) of the triethylamine-sodium salt of 7-amino-1, 3-naphthalene disulfonic acid, about 0.33 mole (about 118 g.) of C-n-octadecylsuccinic anhydride and about 727 g. (sufficient to

make about a 25% solution) of 500 SSU solvent refined naphthenic neutral oil were mixed in an appropriate reactor under nitrogen. The mixture was stirred and heated at about 160°C for 15–20 hours; at this time the infra-red spectrum showed a strong absorption for imide carbonyl group. The cooled product was mixed vigorously to insure homogeneity.

Example 2

Sodium-lithium salt thickened grease

About 0.17 mole (about 487 g. of the 25% imide-oil solution) of the product from the above described reaction was placed in a small, open, externally-heated reactor which was stirred by counter-rotating blades to insure good mixing. About 0.35 mole (14.8 g.) of $LiOH.H_2O$ and about 150 ml. of water was added.

The stirring mixture was heated slowly to about 360°F., then allowed to cool slowly to room temperature (while stirring). The product was homogenized by milling twice in a 3-roll mill. The ASTM ½ scale penetration (worked 60 strokes) of the product at room temperature was 67. 150 g. of the aforementioned neutral oil was added to the product in the previously described reactor. The mixture was stirred and heated slowly to about 380°F, then stirred while cooling to room temperature. The product was homogenized by 2 passes through a "Tri-Homo" mill. The ASTM ½ scale penetration of this product was 138. (worked 60 strokes). After working 10,000 strokes in a motormatic worker (¼inch holes), the ASTM ½ scale penetration was 149. The thickener produced in accordance with the above-described procedure comprised the sodium-lithium salt of C-n-octadecylsuccinimidonaphthalenedisulfonic acid.

Example 3

A di-triethylamine salt of 2-amino-1-phenol-4-sulfonic acid was reacted with C-n-octadecylsuccinic anhydride in the aforementioned neutral oil in the manner described in Example 1 above. This imide was then reacted with 1.1 mole $Ca(OH)_2$ per mole, in the presence of water as described in the reaction of the imide in Example 2 with $LiOH.H_2O$. The ASTM ½ scale penetration (worked 60 strokes) of the initial product was 93; after the further oil addition the value was 188. The product produced in accordance with the above-described procedure comprised the sodium-calcium salt of C-n-octadecylsuccinimidohydroxybenzenesulfonic acid.

Example 4

Preparation of the Imide

About .54 mole (about 210 g) of the triethylamine salt of 2-amino-1-phenol-4-sulfonic acid and about 0.55 mole (about 223 g) of C-n-docosenylsuccinic anhydride and about 1300 g (sufficient to make 25% solution) of 500 SSU solvent refined naphthenic neutral oil were mixed in an appropriate reactor under nitrogen. The mixture was stirred and heated at about 150°C for 15–20 hours. At this time, the infra-red spectrum showed a strong absorption for imide carbonyl group.

Example 5

Calcium Salt-thickened Grease

About 0.16 mole (about 500 g of 25% imide-oil solution) of the product from the above-described reaction was placed in a small, open, externally-heated reactor which was stirred by counter-rotating blades to insure good mixing. About 0.19 mole (15 g of 93%) of $Ca(OH)_2$ and about 25 ml of water were added.

The stirring mixture was heated to 350°F. then allowed to cool to room temperature. The product was homogenized by milling twice in a "Tri-Homo" mill. After cooling to room temperature the ASTM ½ scale penetration (worked 60 strokes) of the product was 89. About 285 g. of above described neutral oil was added to the product in the previousy-described reactor. The mixture was stirred and heated to 350°F., then cooled to room temperature. After milling twice in a "Tri-Homo" mill, the ASTM ½ scale penetration of the producct (worked 60 strokes) was 114. The thickener produced in accordance with the above-described procedure comprised the calcium salt of C-n-docosenylsuccinimidohydroxybenzenesulfonic acid.

Example 6

Sodium Salt-thickened Grease

About 0.16 mole (about 500 g of 25% imide-oil solution) of the product of Example 4 above, was placed in the reactor described in Example 5. About 0.352 mole (14.4 g of 98%) NaOH and 25 ml of water were added. The stirring mixture was heated to 350°F, then cooled to room temperature and milled twice in a "Tri-Homo" mill. After cooling to room temperature the ASTM ½ scale penetration (worked 60 strokes) of the product was 160. The thickener produced in accordance with the above-described procedure comprised the sodium salt of C-n-docosenylsuccinimidohydroxybenzenesulfonic acid.

Example 7

Lithium Salt-thickened Grease

About 0.16 mole (about 500 g of 25% imide-oil solution) of the product from Example 4 above, was placed in the reactor described in Example 5. About 0.352 mole (14.9 g) LiOH and 25 ml of water were added. The stirring mixture was heated to 350°F, then cooled to room temperature and milled twice in a "Tri-Homo" mill. After cooling to room temperature the ASTM ½ scale penetration was 100. The thickener produced in accordance with the above-described procedure comprised the lithium salt of C-n-docosenysuccinimidohydroxybenzenesulfonic acid.

Example 8

Preparation of the Imide

Above 0.54 mole (about 230 g) of the triethylamine salt of 7-amino-1, 3-naphthalene disulfonic acid, monosodium salt and about 0.55 mole (about 231 g) of a C-polybutenylsuccinic anhydride (mole wt. 420) and about 1380 g (sufficient to make about 25% solution) of the neutral oil of Example 1 were mixed in an appropriate reactor under nitrogen. The mixture was stirred and heated at about 150°–180°C for 15–20 hours then allowed to cool to room temperature.

Example 9

Calcium Salt-thickened Grease

About 0.15 mole (about 500 g of 25% imide-oil dispersion) of the product from the above reaction (Example 8) was placed in the reactor. About 0.083 mole of $Ca(OH)_2$ (6.64 g of 93%) and 5 ml of water were added. The stirring mixture was heated at 185°–200°F for about ½ hour, then to 350°F. The mixture was cooled to room temperature. The ASTM ½ scale pene-

tration (worked 60 strokes) was 159. The thickener produced in accordance with the above-described procedure comprised the calcium sodium salt of C-polybutenylsuccinimidonaphthalenedisulfonic acid.

Example 10

Preparation of the Imide

About 0.54 mole (230 g) of the triethylamine salt of 7-amino-1, 3-napthalene disulfonic acid monosodium salt and about 0.55 mole (223 g) of n-docosenylsuccinic anhydride and about 1360 g of the neutral oil of Example 1 were mixed in an appropriate reactor under nitrogen. The mixture was stirred and heated at about 150°C for 15 to 20 hours. The infra-red spectrum of the cooled reaction mixture showed a strong absorption for an imide carbonyl group. The thickener produced in accordance with the above-described procedure comprised the triethylamine-sodium salt of C-n-docosenylsuccinimidonapthalenedisulfide acid.

Example 11

Lithium Salt-thickened Grease

About 0.15 mole of the above product (Example 10) (500 g of 25% imide-oil solution) was placed in the reactor. About 0.15 mole (6.5 g) of $LiOH.H_2O$ and about 10 ml of water were added. The stirring mixture was heated to 210°F ±10° for about 20 minutes there to 365°F. The cooled mixture was milled once through a "Tri-Homo" mill. The ASTM ½ scale penetration (worked 60 times) was 110.

Example 12

Calcium Salt-thickened Grease

About 0.15 moles (about 500 g of 25% imide-oil solution) of the product from the above reaction (Example 10) above was placed in the reactor described in Example 5. About 0.076 mole (6.1 g of 93%) $Ca(OH)_2$ and about 10 ml of water were added.

The stirring mixture was heated to 420°F, cooled and then milled twice through a "Tri-Homo" mill. After cooling to room temperature, the ½ scale ASTM penetration (worked 60 strokes) was 101. The thickener produced in accordance with above example comprises the calcium-sodium salt of C-n-docosenylsuccinimidonapthalenedisulfonic acid.

Example 13

Amine-Sodium-Imide; one-step preparation

About 1360 g of the above-described neutral oil was placed in an appropriate reactor under nitrogen. To this was added a mixture of 0.538 mole (189 g based on titration of acid groups) of 7-Amino-1, 3-naphthalenedisulfonic acid, monosodium salt, 250 ml water and 80 ml of triethylamine. The stirred mixture was heated slowly to about 150° to 160°C to remove the water and excess triethylamine. The mixture was cooled slightly. About 0.55 mole (about 223 g) of n-docosenylsuccinic anhydride was added. The mixture was stirred and heated at about 150°C for about 15 to 20 hours. The mixture was cooled to room temperature. The infrared spectrum of the product showed a strong absorption for imide carbonyl group.

A sample of this product was milled twice in a 3-roll mill. The ASTM ½ scale penetration was 113. The thickener produced in accordance with the above-described procedure comprised the triethylamine sodium salt of C-n-docosenylsuccinimidonaphthalenedisulfonic acid.

The above results illustrate the improved properties realized from grease compositions containing the above-described salts as thickening agents. While the invention has been described with reference to preferred compositions and components therefor, it will be understood, by those skilled in the art, that departures from the preferred embodiments can be effectively made and are within the scope of the preceding specification.

We claim:

1. A grease composition comprising a major proportion of an oil of lubricating viscosity and a thickening amount of a salt of a C-alkyl or alkenyl succinimidoarylsulfonic acid having from about 14 to about 30 carbon atoms in the alkyl or alkenyl group.

2. A grease composition as defined in claim **1** wherein said acid is a C-alkyl or alkenyl succinimidobenzenesulfonic acid.

3. A grease composition as defined in claim **1** wherein said acid is a C-alkyl or alkenyl succinimidonaphthalenesulfonic acid.

4. A grease composition as defined in claim **1** wherein said salt is the sodium-lithium salt of C-n-octadecylsuccinimidonaphthalenedisulfonic acid.

5. A grease composition as defined in claim **1** wherein said salt is the sodium-calcium salt of C-n-octadecylsuccinimidohydroxybenzenesulfonic acid.

6. A grease composition as defined in claim **1** wherein said salt is the triethylamine-sodium salt of C-n-docosenylsuccinimidonaphthalenesulfonic acid.

7. A grease composition as defined in claim **1** wherein said salt is the sodium salt of C-n-docosenylsuccinimidohydroxybenzenesulfonic acid.

8. A grease composition as defined in claim **1** wherein said salt is the lithium salt of C-n-docosenylsuccinimidohydroxybenzenesulfonic acid.

9. A grease composition as defined in claim **1** wherein the oil lubricating viscosity is a mineral lubricating oil.

10. A grease composition as defined in claim **1** wherein the oil of lubricating viscosity is a synthetic lubricating oil.

11. A grease composition as defined in claim **1** wherein the oil of lubricating viscosity has a viscosity range from about 45 SSU at 100°F. to about 6,000 SSU at 100°F.

12. A grease composition as defined in claim **1** wherein the oil of lubricating viscosity has a viscosity range from about 50 SSU at 210°F. to about 250 SSU at 210°F.

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