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LUBRICANT

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This invention has to do with lubricants and more particularly has to do with lubricants characterized by a high order of effectiveness under severe operating conditions.

It is well known that lubricants generally lose some or all of their effectiveness when subjected to high temperature and pressure conditions, and this is particularly true of greases exposed to such conditions. In lubricating machine parts with a grease, it is essential to provide a grease which will substantially retain its character under the foregoing conditions; failure to do so results in high consumption of the lubricant and frequent servicing. In general, available greases suffer from a marked tendency to change in character when used over a wide range of temperature. For example, the consistency of a conventional grease is prone to change over a wide range of temperature. Certain soda base greases, for example, have a pronounced and undesirable property of changing from a short to a long fibre structure, with temperature change. Some conventional greases are also characterized by excessive softening when exposed to elevated temperatures, thereby being extruded too rapidly from the area being lubricated to provide efficient lubrication. In addition, some greases tend to lose their oil content when used at elevated temperatures and pressures; this condition is generally referred to in the art as "bleeding."

We have now discovered a grease which is characterized by a high order of effectiveness under severe operating conditions and which is substantially free from the shortcomings discussed hereinabove. The greases contemplated herein contain as a characterizing or reinforcing agent, a small amount of a water-dispersible, or oil-insoluble and water soluble, thermoplastic material, representative of which are cellulose derivatives such as methyl-, hydroxyethyl-, and sodium carboxymethyl-cellulose; polymerized glycols typified by polyethylenet glycols and esters thereof; esters of polyhydric alcohols and polyvalent inorganic acids; polyvinyl alcohols; natural gums such as gum arabic; proteins; etc. Preferred of such materials, however, are the water-dispersible cellulose ethers and, of this class, water-soluble methyl-cellulose containing from 1 to 1.2 methoxy groups per glucose unit is particularly preferred. All of these materials are well known in the art and it is believed to be unnecessary to discuss them further.

The greases containing one or more of the aforesaid characterizing agents are of the soda base type. These greases have a high soap content of the order of 36–50 per cent on the basis of the final product. They are generally prepared by heating a fat or a fatty acid, or a mixture thereof, with a mineral oil and thereafter heating the

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resultant mixture with caustic soda at elevated temperatures, to obtain a substantially anhydrous soda soap grease. The final product generally contains: 31–44 per cent fat or fatty acid; 4.8–6.8 per cent caustic soda (dry basis); 50–64 per cent mineral oil. In obtaining the grease, the mixture may be cooked, for example, at temperatures of 250–400° F. Available steam cooked greases do not maintain their shape at elevated temperatures and fire cooked greases are typified by "bleeding" under such conditions.

The mineral oil constituent of the greases contemplated herein may vary considerably in character and includes residual or distilled oils. Preference is accorded, however, to oils having a viscosity (S. U. V.) of 100–250 seconds at 210° F. Similarly the fats and fatty acids which may be used are those generally found in soap type greases. Representative of such materials are vegetable, animal and fish fatty oils, and hydrogenated fatty materials thereof. While sodium hydroxide is preferred in view of cost considerations, other metal hydroxides may be used in our greases, preferably where the soap constituent of the grease is prepared by direct saponification in aqueous phase. Accordingly, such other metal hydroxides as those of lithium, potassium, magnesium, calcium, barium, etc., are contemplated here, with the alkali metal hydroxides preferred. It will be understood, therefore, that the term "soap type grease" used herein, and in the appended claims, is used generically and includes such greases wherein metal constituents such as those mentioned above are present in place of sodium.

We have further discovered that the characterizing or reinforcing agents described hereinabove are most advantageously incorporated in soap type greases prior to or during the saponification stage, as illustrated by the following procedure. A fat or fatty acid, or mixture thereof, is added to mineral oil in order to provide a blend thereof; if necessary the materials may be heated in order to thoroughly distribute the fatty material in the mineral oil. A characterizing material, such as oil-insoluble, water-soluble, methyl cellulose, is dissolved or dispersed in water, and the resulting water solution is dispersed or emulsified with the blend of fatty material and mineral oil. The resulting dispersion or emulsion is thereafter saponified with a metal hydroxide solution, such as caustic soda. Following the latter treatment, water—of solution and of reaction—is evaporated by heating and stirring the saponified mixture at a temperature above about 220° F., preferably at 260° F. In this evaporation operation, the saponified mixture may also be steam cooked at temperatures of the order of 250–330° F., or fire cooked at temperatures in the range of 300–450° F. The grease is then withdrawn from the vessel in

which it was prepared and run into a suitable mold. The grease is allowed to cool whereupon it solidifies. The solid grease may then be cut into cakes of the desired size. By following this preferred procedure, the characterizing agent is incorporated in the grease in an extremely fine dispersed state and the grease is provided with a high order of effectiveness.

The greases of this invention are illustrated by the following typical example.

EXAMPLE

One part (by weight) of an oil-insoluble, water-soluble, methyl cellulose having about one methoxy group per glucose unit, was dissolved, with stirring in 30 parts of cold water. Sixteen parts of mineral oil (S. U. V.=165 seconds at 210° F.) and 11 parts of a fatty material were then added, with stirring, to the methyl cellulose solution and heated. The fatty material is a mixture of hydrogenated fatty acids (9 parts) obtained by hydrogenation of fish oil fatty acids, and hydrogenated fat (2 parts) obtained by hydrogenating fish oil fat. The methyl cellulose-oil-fat solution was heated at about 160° F. and agitated vigorously whereupon an emulsion was obtained. Caustic soda solution (45 per cent aqueous solution, 3½ parts) was added to the emulsion at 160° F., addition being completed in ten minutes. The mixture thus obtained was heated, and agitated, for three hours at 280° F. to complete the saponification, and was substantially dehydrated (water content, 0.1 per cent) thereafter by heating or fire cooking to a temperature of 330° F. The grease was then allowed to cool, whereupon it solidified. The final product, hereinafter referred to as grease I, is identified by the following characteristics, all figures being on a dry basis:

Grease I	Per Cent
Hydrogenated Fatty Acids	30.3
Hydrogenated Fat	7.3
Sodium Hydroxide	5.7
Mineral Oil	53.9
Methyl Cellulose	2.8

The outstanding character of the aforesaid grease is shown by established deformation and "bleeding" tests shown hereinafter which were run on samples of the said grease, and comparable and typical soda soap greases known in the art. The latter greases (II and III) are identified by the following:

	II (Fire Cooked)	III (Steam Cooked)
Hydrogenated Fatty Acids	31.8	31.3
Hydrogenated Fat	7.7	7.6
Sodium Hydroxide	6.0	5.8
Mineral Oil	54.5	55.3
(Methyl Cellulose	none	none)

Greases II and III were prepared by direct saponification of fatty materials and mineral oil with a strong caustic solution in the proportions indicated above, and both were dehydrated to a water content of about 0.5 per cent. Grease II was dehydrated by fire cooking and grease III by steam cooking.

One inch cubes were cut from one inch thick grease slabs which were representative cross sections of the grease blocks under test. The cubes were placed on an open tray and the tray was placed on a shelf in a constant temperature electric oven. The cubes were heated at 250° F. for one hour, at 300° F. for one hour and finally at 350° F., without being removed from the oven. After each of the aforesaid heating periods, the top side of each cube was slightly pressed down with the flat side of a spatula to determine whether there was any deformation or "slumping" of the cube. In order to pass the test, the sample should show only a slight spreading at the base of the cube and no free oil at the base of the cube, after the third test period of 350° F. The results (average of 4 tests) of these tests are tabulated in Table I below.

Table I

Grease	Dimensions of Test Cubes		Per Cent Increase in Base Area	Per Cent Decrease in Height	Oil at Base
	Before Heating	After Heating			
I	Base 1.031" x 1.031" Height 1.031"	1.109" x 1.094" 0.970"	14	6	None.
II	Base 1.000" x 1.063" Height 1.000"	1.156" x 1.140" 0.880"	24	12	Considerable.
III	Base 1.047" x 1.047" Height 1.000"	1.360" x 1.282" 0.813"	59	19	None.

It will be apparent from the foregoing results that grease I, which contains methyl cellulose, is greatly superior to grease II in its resistance to bleeding as shown by the oil extruded from the latter grease, and also in its substantially smaller degree of deformation. Grease I is also far superior to grease III in regard to deformation.

High temperature penetration tests were also carried out with grease test cubes, three inches on a side, after heating the same in an oven of the aforesaid type for three hours at 300° F. At the end of the heating period, A. S. T. M. penetration values (using a 300 gram load) were obtained at 300° F. Concurrently, measurements of each cube were taken to determine the degree of deformation. Test results are shown in Table II.

Table II

Grease	A. S. T. M. Penetration (300 grm. load) @ 300° F. (dms.)	Dimensions of Test Cubes		Per Cent Increase in Base Area	Per Cent Decrease in Height	Remarks
		Before Heating	After Heating			
I	249	Base 3" x 3" Height 3"	3¼" x 3" 2¾"	3	3	Slight deformation.
III	353	Base 3" x 3" Height 3"	5" x 3" 2½" x 1½"	94	33	Slumped more on one side.

Here again, test results effectively demonstrate the outstanding character of greases of the type contemplated herein.

It will be apparent from the foregoing that the greases contemplated herein have considerable application. For example, they are outstanding greases for lubricating locomotive driving journals where high temperatures and pressures are encountered. They also find application as antifriction bearing greases, for example, ball bearing and roller greases. Other applications will be apparent to those skilled in the art.

The characterizing materials of this invention may be used in various amounts in our greases. In general, from about ½ per cent to about 10 per cent will provide satisfactory greases, with preferred amounts being of the order of ½ per cent to 3 per cent. Accordingly, the various constituents (on a dry basis) of our greases may vary within the following limits:

	Per cent
Fatty material	30–50
Alkali	4½–7½
Mineral oil	40–60
Characterizing material	½–10

It is to be understood that the greases of this invention may also contain other characterizing agents and fillers. For example, they may contain grease antioxidants such as amines, phenols, sulfides, etc.; fillers such as asbestos, graphite, mica, talc, etc.; and lubricity improving agents such as free fat, free fatty acids, sulfurized fats, lead soaps, etc.

It is to be understood, moreover, that the foregoing specific examples are but representative of the greases contemplated herein. The present invention, therefore, is not to be construed as limited thereto but is to be broadly construed in the light of the claims appended hereto.

We claim:

1. A lubricating grease composition characterized by resistance to deformation and "bleeding" comprising the reaction products of the following ingredients in the indicated proportions:

	Per cent
Fatty material	30–50
Alkali	4½–7½
Mineral oil	40–60
Water-dispersible, cellulose ether	½–10

2. A lubricating grease composition characterized by resistance to deformation and "bleeding," comprising the reaction products of the following ingredients in the indicated proportions:

	Per cent
Hydrogenated fatty acids	30.3
Hydrogenated fat	7.3
Sodium hydroxide	5.7
Mineral oil	53.9
Water-dispersible methyl cellulose	2.8

3. A lubricating grease composition prepared from about 30 to about 50 per cent of a fatty material, about 4.5 to about 7.5 per cent of an alkaline material selected from the group consisting of alkali metal hydroxides and alkaline earth metal hydroxides, about 0.5 to about 10 per cent of a water-dispersible cellulose ether and the balance, mineral oil.

4. A lubricating grease composition prepared from about 30 to about 50 per cent of a fatty material, about 4.5 to about 7.5 per cent of an alkaline material selected from the group consisting of alkali metal hydroxides and alkaline earth metal hydroxides, about 0.5 to about 10 per cent of water-dispersible methyl cellulose and the balance, mineral oil.

5. A lubricating grease composition prepared from about 30 to about 50 per cent of a fatty material, about 4.5 to about 7.5 per cent of an alkali metal hydroxide, about 0.5 to about 10 per cent of a water-dispersible cellulose ether and the balance, mineral oil.

6. A lubricating grease composition prepared from about 30 to about 50 per cent of a fatty material, about 4.5 to about 7.5 per cent of an alkaline earth metal hydroxide, about 0.5 to about 10 per cent of a water-dispersible cellulose ether and the balance, mineral oil.

7. A soda soap lubricating grease characterized by resistance to deformation and "bleeding," comprising a sodium soap content of from about 36 to about 50 per cent and from about 0.5 to about 10 per cent of a water-dispersible cellulose ether.

8. A soda soap lubricating grease characterized by resistance to deformation and "bleeding," comprising a sodium soap content of from about 36 to about 50 per cent and from about 0.5 to about 10 per cent of water-dispersible methyl cellulose.

9. A soda soap lubricating grease characterized by resistance to deformation and "bleeding," comprising a sodium soap content of from about 36 to about 50 per cent and from about 0.5 to about 3 per cent of water-dispersible methyl cellulose.

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