

# UNITED STATES PATENT OFFICE

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## PRODUCTION OF GREASES USING REACTION PRODUCTS OF ALPHA-HYDROXY FATTY ACIDS

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This invention relates to an improved process for the production of greases.

In one of its aspects the invention relates to the preparation of certain fatty acid reaction products for use in the production of gel type lubricating greases. In another of its aspects the invention relates to fatty acid reaction products useful in the preparation of gel type lubricating greases with little or no working to produce a smooth grease with little tendency to bleed. In still another of its aspects the invention relates to novel gel type lubricating greases having little tendency to bleed.

It is well known that greases can be made by dissolving or dispersing under suitable conditions a fatty acid soap in a suitable hydrocarbon oil. The particular type of grease produced depends, of course, upon the method by which it is produced. A fiber grease is usually produced by mixing the proper proportions of oil, fat, and aqueous caustic soda and heating to saponify the fat and to evaporate the water formed as well as any water which may be present. The resulting mixture is usually about 50 per cent each of oil and fatty acid soap, and a fiber grease is then produced by slow cooling and mixing with additional oil. A gel grease is produced by mixing a suitable fatty acid soap, usually in an amount from 5 to 20 per cent, with a hydrocarbon oil and heating to a temperature somewhat above the solution temperature. Upon effecting solution, the material is cooled sufficiently rapidly that substantially no separation of soap and oil takes place, and the resulting cooled material is then worked to produce a smooth grease.

In making gel greases, the soap is usually an alkali metal salt of a fatty acid of 18 carbon atoms or higher. The sodium and potassium salts of palmitic acid (16 carbon atoms) can only occasionally be used, and those of less than 16 carbon atoms do not appear to be compatible with the oils in so far as making grease is concerned.

Lithium salts of fatty acids having 16, 14, and sometimes 12 carbon atoms per molecule can often be used. The oil used in the manufacture of the grease determines how short a chain can be successfully used.

I have now found that metal salts of the reaction product of an hydroxy fatty acid, especially an alpha-hydroxy fatty acid, and a fatty acid are excellent grease gelation agents, and that those acids which are not, per se, suitable grease gelation agents may be changed so that they can be used to produce lubricating greases.

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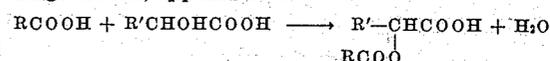
I have also found that the reaction product, formed upon esterification of the hydroxy group of the hydroxy fatty acid with one or more molecules of such an acid, when converted to a metal salt, is an excellent gelation agent for lubricating oil greases.

Thus, according to the invention, a juncture product of a hydroxy fatty acid, preferably an alpha-hydroxy fatty acid, with at least one other molecule of such an acid, and/or with another fatty acid, is produced, converted to a metal salt and then used in the production of a gel type lubricating grease. In place of the fatty acid other acylating agents can also be used. For example, the acid anhydrides or the acid chlorides of the fatty acids can be used. Also a polymerized hydroxy fatty acid can be reacted to form a mixture of a metal salt of the polymer and a metal salt of a fatty acid and said mixture can then be used as a grease gelation agent.

The following description and examples read in conjunction with the preceding portion of this specification will indicate fully and clearly the scope of the appended claims to the invention so that one skilled in the grease production art will be enabled to prepare any of the products to which reference is made. A now preferred gelation agent of the invention is that which is formed by the reaction of an alpha-hydroxy fatty acid with a fatty acid. Therefore, the following portion of the description relates to said reaction.

The general process for the production of the gelation agents from an alpha-hydroxy fatty acid and a fatty acid comprises heating the fatty acid and alpha-hydroxy fatty acid in the liquid phase for a sufficient length of time to effect reaction, usually from 1 to 12 hours. A condensation catalyst such as zinc chloride can be used. The reaction temperature is usually in the range of 125° C. to 250° C., preferably 150° C. to 225° C., and the pressure such that the water present and/or formed is allowed to distill off but sufficient to maintain the acids in substantially liquid phase. Operation at atmospheric pressure is ordinarily preferred, but elevated pressure can be used where necessary.

The reaction which takes place, at least to a large extent, appears to be



wherein R and R' represent alkyl groups and the total number of carbon atoms in R'+R is at least 16 and preferably at least 18.

Several specific procedures, not necessarily

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completely equivalent methods, for carrying out the reaction can be used. In some instances one or more of these methods will be found to be preferred over another.

The simplest method for carrying out the illustrated reaction is to heat a mixture of the fatty acid and hydroxy fatty acid, the fatty acid being present at least in an amount molecularly equivalent to the hydroxy fatty acid. The product is then neutralized to produce the sodium salt, and the neutral product is used to produce a gel grease with a hydrocarbon oil.

In a second method, the reaction vessel initially contains only a heated quantity of fatty acid, and the alpha-hydroxy fatty acid is then slowly added with agitation. In this manner, the concentration of the reactive hydroxy acid is kept low and the reaction is directed toward juncture of the fatty acid and hydroxy fatty acid, because the opportunity for two hydroxy fatty acid molecules to react has been minimized. After the reaction has been effected, neutralization of the acid constituents to form sodium salts and the making of the grease is carried out.

The reaction may also be carried out in a continuous system. In such a method, the fatty acid and hydroxy fatty acid are continuously charged to a reaction zone in the desired proportions, either as separate streams or as a mixture. A stream of the reaction mixture equal to the materials charged is continuously drawn off. Neutralization and making of the grease is carried out as before.

In some instances, it may be desirable to effect even further direction of the reaction to that of the fatty acid with the alpha-hydroxyl group. This may be done by maintaining at all times the fatty acid in molecular excess over the unreacted alpha-hydroxy fatty acid in the reaction zone. The excess fatty acid may be removed from the reaction mixture, withdrawn and recycled to the reaction zone prior to neutralization. However, in those instances where this excess is not objectionable in the final utilization, none or only a part may be removed prior to neutralization.

In choosing the starting reactants, it is desirable to choose a pair which will yield a juncture product of at least 16 carbon atoms per molecule, and preferably 18 or more carbon atoms per molecule. As noted, a partial esterification of the alpha-hydroxy fatty acid with itself can occur. However, these dimers should be considered on the basis of total available monomer, disregarding any dimerization, when calculating the moles in any given quantity of hydroxy fatty acid.

Although not as desirable a method as those previously described, an alpha-hydroxy fatty acid can be polymerized and subsequently reacted to form a mixture of the sodium salts of the polymer and a fatty acid, and the mixed salts then used as grease gelation agents, as stated. In such instances, it is preferred that the alpha-hydroxy acid have at least eight carbon atoms per molecule. Or a condensation product of two or more molecules of the hydroxy acid may be condensed with an unsubstituted fatty acid to produce an acid that may be converted to a soap suitable for use in a grease.

These gelation agents can also be used with the synthetic ester, diester and polyester lubricating oils, or with polyalkalene oxide lubricating oils, although it is noted especially that an ester oil is not required and that according to the inven-

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tion greases with lubricating oils can be prepared with little or no working by simple admixture and heating of the gelation agent and a lubricating oil.

It is to be noted that the gelation agents of this invention produce greases of excellent resistance to bleeding.

#### Example I

Approximately equimolecular parts of lauric acid and alpha-hydroxydecanoic acid primarily 2-hydroxy-4,6,6-trimethylheptanoic acid were heated together at atmospheric pressure for three hours at 150° C. and one additional hour at 200° C. A small amount of zinc chloride was used as a catalyst. The reaction mixture was cooled, and the organic material neutralized with sodium hydroxide. The neutralized organic material was mixed and heated with a hydrocarbon lubricating oil and dissolved therein. On quick cooling the solution, a grease was produced.

#### Example II

In a second trial, it was attempted to produce a gel grease from the mixed sodium salts of lauric acid and alpha-hydroxydecanoic acid. This mixture of salts did not produce a grease. These salts individually are not gelation agents.

#### Example III

In another test, alpha-hydroxydecanoic acid as the only reactant was heated for three hours at 150° C. and one hour additional at 200° C. The resulting material was neutralized with sodium hydroxide and this sodium salt mixed with sodium laurate. The mixed salts were then used successfully as a gelation agent in making a gel grease from a hydrocarbon oil.

#### Example IV

Portions of alpha-hydroxydecanoic acid were separately reacted by the previously-described procedure in mixture with stearic acid and in mixture with oleic acid. Each product was converted to the sodium salt and then successfully used as the gelation agent for making a gel grease from hydrocarbon oil.

From Examples I and II it can be noted that the neutralized reaction product of lauric acid and alpha-hydroxydecanoic acids acted as a gelation agent whereas a mixture of the sodium salts of these acids did not so act. However, in Example III, where the alpha-hydroxydecanoic acid was the only reactant and it was reacted with itself, then neutralized and then mixed with sodium laurate a successful gelation agent was obtained. Example IV shows that substitution of stearic and oleic acids, respectively, for the lauric acid of Example I was successful.

Although this specification is directed mainly to the use of sodium soaps of the disclosed condensed acids, it should be understood that other metal soaps may be used. These include soaps of other alkali metals, such as potassium and lithium, soaps of the alkaline earth metals, such as calcium, strontium, and barium, and also soaps of aluminum and of magnesium. The soaps of the different metals are not necessarily full equivalents, and this fact must be borne in mind when specific applications of the invention are contemplated. Mixtures of the soaps of two or more metals may be used as grease gelation agents within the broad scope of this invention.

Other alpha-hydroxy acids usable in the process of the invention are alpha-hydroxyoctanoic

acids, alpha-hydroxydodecanoic acids, and alpha-hydroxy hexadecanoic acids.

Variation and modification are possible within the scope of the foregoing disclosure and the appended claims to the invention the essence of which is that certain hydroxy fatty acid, preferably alpha-hydroxy fatty acid, reaction products have been prepared, and when converted to sodium salts, these products have been found to produce smooth gel type lubricating greases with little or no working being required and the grease having little or no appreciable tendency to bleed; this without necessary recourse to any ester oil.

I claim:

1. The production of a gel type lubricating grease which comprises effecting an esterification juncture of an hydroxy fatty acid with at least one of an hydroxy fatty acid and another fatty acid to produce a juncture product having at least sixteen carbon atoms to the molecule; converting the juncture product to a metal salt; and admixing said salt with a suitable lubricating oil to produce said grease.

2. A production according to claim 1 wherein the juncture is effected at an elevated temperature sufficient to cause reaction to occur and the reaction mass is maintained at said temperature for a time sufficient to complete the reaction.

3. A production according to claim 2 wherein the temperature is in the range 125° to 250° C.

4. A production according to claim 3 wherein the temperature initially is lower than finally and the initial temperature is maintained for a substantial period of the reaction.

5. The production of a gel type lubricating grease which comprises effecting an esterification juncture of alpha-hydroxydecanoic acid (2-hydroxy-4,6,6-trimethylheptanoic acid) with at least one material selected from the group consisting of said acid itself and another fatty acid to produce a juncture product having at least sixteen carbon atoms to the molecule; converting the juncture product to sodium salt; and then admixing said salt with a suitable lubricating oil to produce said grease.

6. A production according to claim 5 wherein the other fatty acid when used is selected from the group consisting of lauric, stearic and oleic acids.

7. A production according to claim 6 wherein the juncture is effected at a temperature in the approximate range 125° to 250° C.

8. A gel type lubricating grease the essential gelation agent of which is a metal salt of an esterification juncture product of an hydroxy fatty acid with a material selected from the group consisting of an hydroxy fatty acid and another fatty acid said product containing at least sixteen carbon atoms.

9. A grease according to claim 8 consisting essentially of said gelation agent and a suitable lubricating oil.

10. A grease according to claim 8 wherein the hydroxy fatty acid is 2-hydroxy-4,6,6-trimethylheptanoic acid and the other fatty acid is selected from the group consisting of lauric, stearic and oleic acids.

11. A gel type lubricating grease the gelation agent of which is an admixture of the sodium salt of an esterification juncture product of alpha-hydroxydecanoic acid (2-hydroxy-4,6,6-trimethylheptanoic acid) with itself and sodium laurate.

12. The preparation of a gel-type lubricating grease which comprises heating an hydroxy fatty acid with at least one of an hydroxy fatty acid and another fatty acid, in the liquid phase at a temperature in the approximate range 125-250° C., for a time in the range of from about 1 hour to about 12 hours, to produce a product having at least 16 carbon atoms to the molecule; converting the product thus produced to a metal salt; admixing the salt thus obtained with a lubricating oil; heating the admixture to form a blend of said salt and said lubricating oil; and then cooling said blend sufficiently rapidly, so that no substantial separation of the salt and oil can occur, to form said grease.

13. A preparation according to claim 12 wherein the hydroxy fatty acid is at least one acid selected from the group consisting of alpha-hydroxy-octanoic acid, alpha-hydroxydecanoic acid, alpha-hydroxydodecanoic acid, and alpha-hydroxyhexadecanoic acid and the said another fatty acid is at least one acid selected from the group consisting of lauric acid, palmitic acid, stearic acid and oleic acid.

14. A preparation according to claim 12 in which a small amount of zinc chloride is employed as a catalyst during the first heating step.

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#### REFERENCES CITED

The following references are of record in the file of this patent:

#### UNITED STATES PATENTS

Number	Name	Date
1,927,296	Powers	Sept. 19, 1933
2,334,274	Meadows	Nov. 16, 1943
2,375,530	De Groote et al.	May 8, 1945
2,397,956	Fraser	Apr. 9, 1946
2,450,220	Ashburn et al.	Sept. 28, 1948
2,452,092	Ault	Oct. 26, 1948

#### FOREIGN PATENTS

Number	Country	Date
615,188	Great Britain	Jan. 3, 1949