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PRODUCTION OF GREASES

Arnold A. Bondi, New Orleans, La., assignor, by mesne assignments, to Shell Development Company, San Francisco, Calif., a corporation of Delaware

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1 The present invention relates to the production of sodium soap lubricating greases.

It has hitherto been proposed to saponify the fatty acid component of a grease-making batch with metallic sodium, but the reaction has not been carried to completion, and the resulting grease has been of poor quality and relatively unstable.

The present invention has for its primary object the production of a sodium soap lubricant grease which eliminates the disadvantages above set forth.

Another object of the present invention is the production of a sodium soap lubricant grease wherein the time of cooking of the grease is shortened while there is simultaneously produced a grease which keeps its penetration value after relatively long periods of milling.

A further object of the present invention is the production of a sodium soap lubricant grease where the fatty acid component of the grease-making batch is saponified by metallic sodium in the presence of an addition agent which facilitates the completion of the grease-making reaction, and produces a grease which is firm, stable, shows little tendency to bleed, and has the penetration properties which characterize a normal grease as more fully hereinafter pointed out.

A still further object of the present invention is the production of a sodium soap lubricant grease wherein the saponifiable component of the grease-making batch is saponified by metallic sodium in the presence of an organic addition agent which prevents the formation of shells of hard reaction product around the globules of metallic sodium formed on the heating of the sodium, said organic addition agent preferably having a labile hydroxy group attached to a secondary or tertiary carbon atom.

Another object of the present invention is the production of a grease containing a polyglycol having a molecular weight varying from about 400 to 6000, said polyglycol being preferably a polyethylene glycol. The preferred molecular weight is about 1500. The polyethylene glycol may be defined as a dihydroxy polyalkylene ether.

In carrying out the present invention the alkylene radical of the dihydroxy polyalkylene ether may comprise 2 to 6 carbon atoms. When the polyalkylene glycol is used in the making of grease to promote the reaction between the metallic sodium and the fatty acid component, it should be used in amounts varying from 1% to 25%, but preferably between 1% to 15%, based

2 on the amount of the saponifiable fatty acid component present in the grease making mixture.

It is not intended to limit the amount of polyethylene glycol that is being added to the grease to the percentages above set forth as many desirable properties are conferred upon the grease when the polyalkylene glycols are present in amounts from 0.2% to 25% based on the weight of the saponifiable matter present in the grease, or stated differently, a soda base grease having valuable properties may be produced by incorporating in the grease .02% to 1.0% of a polyalkylene glycol and preferably a polyethylene glycol, said percentage range being taken on the weight of the soda base grease. Usually said soda base greases contain between 3% to 15% of a sodium soap which is usually derived from stearic acid or other fatty acids of between 16 and 24 carbon atoms or their glycerides.

In accordance with one form of the present invention, the saponification of the saponifiable medium of the grease-making batch is effected in the presence of an addition agent while the batch is being heated, said addition agent comprising an organic compound containing a labile hydroxy group or groups, said addition agent functioning during the grease-making reaction to inhibit or prevent the production of hard shells of a grease-making reaction product around the sodium globules. The addition agent may comprise hydrogenated sugars and their fatty acid esters, the polyethylene glycols, and blown fatty oils which contain hydroxylated fatty acid esters formed by oxidation during the blowing step.

In one form of the invention the addition agent is selected from a hydroxy stearic acid or a hydroxy stearic acid ester. The addition agent may be a monohydroxy stearic acid or a monohydroxy stearic acid ester. Very satisfactory results have been obtained by using an addition agent selected from a group consisting of 12-hydroxy stearic acid or a 12-hydroxy stearic acid ester including the glycerides. While hydrogenated ricinoleic acid or the glyceride thereof may be used as the addition agent, ordinary ricinoleic acid and the ester thereof, that is, castor oil and castor oil fatty acid may be used.

The addition agent may be a composite addition agent as, for example, 12-hydroxy stearic glyceride may be mixed with sorbitan monostearate or with sorbide-monostearate, the 12-hydroxy stearic glyceride predominating. Alternatively, the 12-hydroxy stearic acid or its ester may be mixed with a polyalkylene glycol, as for example,

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polyethylene glycol having a molecular weight varying between 400 to 6000, but preferably having a molecular weight of about 1500.

The lubricating grease may have incorporated therein as an addition agent polyhydroxy-stearic acids. For example, there may be added to the batch hydroxy stearic acids in which the hydroxy groups are in the 9 or 10 carbon position. The polyhydroxy stearic acid may be prepared by sulfonation and subsequent hydroxylation of oleic acid.

The present invention will be illustrated in connection with the following examples:

Example 1

42 grams of metallic sodium are emulsified with a small amount of lubricating oil in any appropriate type of emulsifier. Preferably 42 grams of metallic sodium are emulsified with 100 grams of 2000 vis. at 100° F. coastal lubricating oil at a temperature of about 220° F. The amount of metallic sodium used is the stoichiometric amount required to neutralize the fatty acid component of the batch. A greater amount may be used if desired. The resulting emulsion of metallic sodium in the lubricating base is added to a mixture of about 480 grams of commercial stearic acid and 800 grams of 2000 vis. at 100° F. coastal red oil. The reaction is exceedingly sluggish and is not completed even after cooking for 1 hour at 380° F. To the mass there was added 6000 grams of lubricating oil of the same kind above set forth and the resulting batch heated to about 370° F. Thereafter the grease was poured into cooling pans and cooled in the usual manner. The grease was a soft and mushy product substantially valueless for use as a commercial grease. The acidity of the resulting mass was 3 mg. KOH indicating that 13% of the total amount of fatty acid present had failed to react with the metallic sodium.

The above run was repeated utilizing the identical procedure, but instead of using 480 grams of stearic acid, there was substituted therefor a mixture of 360 grams of stearic acid and 120 grams of 12-hydroxy stearic acid. In the presence of the 12-hydroxy stearic acid the saponification reaction and other grease making reactions proceeded smoothly and rapidly being completed within 10 minutes, this being indicated by the sudden subsidence of foam and the increase in viscosity of the melt.

The resulting grease had the normal properties of a good commercial grease. More specifically, the properties of the grease were as follows:

Penetration at 77° F.: After 60 strokes, 244, and after 300 strokes, 284. The micro penetration before rolling in a shell-roller-tester was 140; after 4 hours' rolling, the micro penetration was 175, all of the penetration figures being expressed in deci-millimeter units.

Example 2

A mixture of 480 grams of tristearin and 800 grams of 2000 viscosity coastal lubricating mineral oil was mixed with 42 grams of metallic sodium. This amount of metallic sodium is the stoichiometric amount required to neutralize the fatty acid component of the grease batch including the addition agent. Of course, there may be a slight excess up to about 5% of sodium or of fatty acid. The resulting mixture of oil and glyceride and metallic sodium is emulsified at 320° F. The reaction proceeded very sluggishly and was not completed even after 90 minutes of cooking

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as apparent by the non-subsidence of the foam and the insignificant change in viscosity of the melt. The so-produced mass was then mixed with about 6000 grams of the same kind of mineral oil and poured and cooled in the usual manner. The resulting grease was mushy and non-coherent.

The above procedure was repeated employing the conditions set forth, but instead of employing 480 grams of the tristearic glyceride there was substituted therefor 360 grams of tristearic glyceride and 120 grams of tri-12-hydroxy stearic glyceride. The resulting grease was firm, coherent, and had about the same stability as the grease produced in accordance with Example 1.

Example 3

The procedure set forth in Example 2 was followed, but instead of employing 480 grams of tri-stearic glyceride, there was substituted therefor a mixture of 360 grams of stearic acid and 120 grams of tri-12-hydroxy stearic glyceride. The resulting grease was firm and had normal mechanical properties. The penetration in deci-millimeter units at 77° F., after 60 strokes, was 220; after 180 strokes, 246; and after 300 strokes, 260.

Example 4

A mixture of 480 grams of stearic acid, 70 grams of tri-12-hydroxy stearic glyceride, 20 grams sorbitan monostearate in 700 grams of 2000 vis. coastal mineral lubricating oil was reacted with 47 grams of metallic sodium emulsified in 100 grams of 2000 vis. mineral lubricating oil while the mixture was heated at about 390° F. The reaction proceeded smoothly. The 20 grams of the sorbitan ester replaced the function of 70 grams of hydroxy stearic glyceride. After diluting to 7000 grams with lubricating oil the mixture was poured into pans and cooled in the usual manner to produce a firm grease. The penetration in deci-millimeter units at 77° F. after 60 strokes was 245; after 180 strokes, 262; and after 300 strokes, 276.

Example 5

Using the same procedure set forth in Example 4 but replacing the sorbitan monostearate by 23 grams of sorbitide-monostearate, a good stable firm grease is obtained which had a penetration at 77° F., after 60 strokes, of 245; after 180 strokes, 276; and after 300 strokes, 288.

Example 6

A mixture of 275 grams of stearic acid and 10 grams of polyethyleneglycol (molecular weight, 1500) in 600 grams of 2000 vis. coastal mineral lubricating oil was reacted with an oil emulsion containing 22 grams of metallic sodium, the reaction being effected at about 320° F. The reaction proceeded smoothly and was completed after 15 minutes. The resulting mass was diluted with lubricating oil to produce 6000 grams of finished product. After cooling a typical light chassis lubricant was obtained. The penetration at 77° F. after 60 strokes was 332, and after 300 strokes, 350, all expressed in deci-millimeters.

The above procedure was repeated using a mixture of 62 grams of stearic acid, 35 grams of tri-12-hydroxy stearate, 10 grams of polyethyleneglycol (molecular weight, 1500) the mixture being reacted with 19 grams of sodium in an oil emulsion. The resulting grease which had excellent mechanical properties had a penetration at 77° F. after 60 strokes of 296; after 180 strokes,

316; and after 300 strokes, 318 expressed in decimillimeters.

Example 7

A mixture of 60 grams of stearic acid, 10 grams of rapeseed oil containing about 10% of hydroxylated esters and 10 grams of 12-hydroxy stearic acid, together with 150 grams of oil, was reacted with 7.2 grams of sodium emulsified in 80 grams of 2000 vis. mineral lubricating oil and the mass heated to 360° F. The reaction proceeded smoothly and rapidly. The mass was diluted with a mineral oil of the same viscosity to produce a final mass of 1000 grams, and thereafter cooled to produce a firm grease with good mechanical properties and showing no tendency to bleed.

It is desired to point out that some materials furnishing 12-hydroxy stearic acid, which may be the acid or an ester thereof, are soluble in the oil component of the lubricating grease and, therefore, require no blending agent for their incorporation in the grease. Other materials furnishing the 12-hydroxy stearic acid radical may be only slightly soluble or soluble with difficulty in mineral oils, animal oils, and vegetable oils usually used in the manufactured greases; therefore, it may be necessary to provide a dispersion agent for the same.

The 12-hydroxy stearic acid radical in one form may be provided by hydrogenated ricinoleic acid.

While usually the oil base is a mineral oil base, it is obvious that the mineral oil base may have mixed therewith any of the well known lubricating bases including hydrogenated fish oil, animal oils, vegetable oils and/or rosin, or synthetic ester, or plurality thereof, all as well known in the grease-making art. The oil base may be a vegetable oil base or an animal oil base or a mixture as well known in the art. The term "12-hydroxy stearic acid" is used in accordance with the common usage which means that the hydroxy group is on the twelfth carbon atom from the carboxyl group.

The emulsion of the metallic sodium is produced by high speed stirring of the hot oil with sodium, the temperature of the oil being above the melting point of sodium which is 97° C. The molten sodium is agitated in the hot oil by any known means. Preferably the emulsion of molten sodium in the oil is produced by homogenizing which is a well known method of producing emulsions. It may be stated that it has been observed that the emulsion reacts best when it has been stirred in the presence of an oxygen-containing atmosphere as, for example, the air, for a sufficient length of time until the emulsion acquires a bluish-gray appearance. While in the herein examples, the emulsion is produced by dispersing the molten sodium in mineral oil, it is obvious that other types of dispersion medium may be used so long as the dispersion medium is substantially chemically inert relative to the molten sodium provided the dispersion medium has a boiling point above the melting point of the sodium. Any hydrocarbon or ether having a boiling point above the melting point of the sodium and chemically inert with respect to the sodium may be used as the dispersion medium. The emulsion can be prepared in a concentration of 1 to 70% of sodium, the preferred range being between 10% to 30%. The amount of molten sodium used is a function of the amount and molecular weight and basicity of the fatty acids employed including the addition agent. In other words, the amount of sodium used depends on the amount of acid material to be

saponified. Usually the amount of sodium used is that stoichiometric amount required to exactly neutralize the fatty acid. In some cases a slight excess usually varying from ¼% to 5% may be employed of either the sodium or fatty acid in order to impart certain desirable properties to the finished grease.

In carrying out the present invention the saponifiable component of the grease making batch may be any of the components usually used in the production of grease. The fatty acids usually used in grease making are the saturated fatty acids containing from 14 to 32 carbon atoms and the unsaturated fatty acids containing from 18 to 22 carbon atoms. Instead of using the said fatty acids, the glycerides thereof may be used as well as the monohydric alcohol esters of certain fatty acids or the wax esters of said acids.

The addition agent may comprise any of the materials herein set forth including 12-hydroxy stearic acid, degreas which contains hydroxy palmitic acid; 9 and/or 10 hydroxy stearic acid, the 9, 10 hydroxy stearic acid containing two hydroxy radicals being known as the 9, 10 dihydroxy stearic acid; blown rapeseed oil; hydrogenated sugars and their stearic and other fatty acid esters, glycols and related compounds.

In producing grease in accordance with the present invention, the grease making batch may be heated to a temperature varying from 200° F. to 600° F.; however, the preferred heating range varies from about 290° F. to about 400° F.

In forming or producing grease in the presence of metallic sodium, water is not produced by the saponification of the higher fatty acids or the esters. Since no water is formed during the saponification step, it is not necessary to drive any water off, and, therefore, there is a shorter time cycle for the grease formation. Further, there is considerable saving in heat energy required because of the shorter time cycle in order to produce the grease; therefore, for a given size of grease-making kettle, more grease can be made for each 24-hour period.

Utilizing the metallic sodium and the addition agent of the present invention, the grease-making reaction is completed in a time period varying from about 10 to 25 minutes which compares with the usual time period of from about 50 minutes to 90 minutes when using sodium hydroxide instead of metallic sodium. In general, using metallic sodium and the addition agent of the present invention, the time period varies from about one-fifth to one-half of the time period which is necessary to react a batch of equivalent weight when using sodium hydroxide.

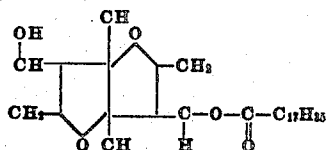
The present invention sets forth a method of manufacturing a soap base grease comprising forming a mixture of a lubricating base; which may be any of the usual lubricating bases used in the manufacture of grease, a material furnishing a saponifiable grease making component which is usually a fatty acid or an ester thereof, or any of the materials herein set forth; metallic sodium which acts as a saponifying agent; and an addition agent having a hydroxy fatty acid component which is intended to be a generic expression to include hydroxy fatty acid, itself, or an ester thereof. After forming a grease-making batch of a character set forth, the mixture is heated at the temperatures herein set forth or equivalent temperatures until saponification and the grease making reaction are substantially completed. It is, of course, to be understood that any of the usual temperatures used in making grease

may be used in carrying out the present invention provided the temperature is above the melting point of sodium. Any saponifiable medium usually used in grease-making, including the saponifiable vegetable and animal oils and fats may be utilized in carrying out the present invention; in short, any of the prior art saponifiable media may be used which are set forth in Klemgard's book entitled "Lubricating Greases; Their Manufacture and Use" (1937), published by the Reinhold Publishing Company, New York. Usually the saponifiable grease-making component is a fatty acid or an ester, but any saponifiable medium may be used which has hitherto been used in the making of grease and saponified by sodium hydroxide or an equivalent saponifying agent.

When the addition agent is a hydroxy fatty acid, the amount thereof may vary between 15% and 30% taken on the weight of the saponifiable grease-making component present in the grease. When the addition agent is a polyhydroxy compound as, for example, any of those set forth in Examples 4, 5, and 6, the amount of addition agent used may broadly vary from about 1% to 25%, but preferably from about 3% to 5% taken on the weight of the saponifiable grease-making component present in the grease. Referring to Example 6, good results have been obtained using polyethylene glycol as the addition agent, but a somewhat better result is obtained when using a mixture of 12-hydroxy stearic acid and polyethylene glycol, the latter being present in minor proportion as, for example 10% to 35%, the balance being 12-hydroxy stearic acid.

In practicing the present invention the addition agent when it comprises a higher hydroxy fatty acid ester may be present in an amount varying between 10% and 50% based on the weight of the saponifiable fatty material. When the addition agent comprises the 12-hydroxy fatty acid or the 12-hydroxy stearic acid, said addition agent may be present in the grease-making batch in an amount varying between 10% and 50% based on the weight of the saponifiable fatty material. Similarly, when using tri-12-hydroxy stearin glyceride, the latter may be present in an amount between 10% and 50% taken on the weight of the saponifiable fatty material. Further, when using the higher hydroxy fatty acids and esters thereof, in one form of the invention, these materials may be present in a grease-making batch in an amount varying between 15% and 30% taken on the weight of the saponifiable grease-making material.

Sorbitan monostearate has the following formula:



Grease made in accordance with the present invention may be compounded in the cold with other greases, as, for example, calcium soap greases, aluminum stearate greases, and the like.

Wherever the term "polyalkylene glycol" or the term "polyethylene glycol" is used in the present specification, it is intended to cover those glycols having a molecular weight varying from 400 to 6000.

The polyalkylene glycols herein referred to are produced by the polymerization of alkylene oxides.

In making a soda base grease, in accordance with the present invention, instead of using sorbitan monostearate as the addition agent whereby the formation of shells of hard reaction product around the sodium particles are inhibited, other sorbitan esters may be used, as for example, sorbitan oleate, sorbitan myristate, and sorbitan laurate; and in general the oil-soluble fatty acid esters of sorbitan, the fatty acid radicals of which have more than 8 carbon atoms.

In general, in accordance with the present invention, there is provided a soda base grease containing 2% to 1.0% of a sorbitan ester of a fatty acid having at least 8 carbon atoms in the fatty acid radical. Preferably, the fatty acid which is combined with the sorbitan radical may have anywhere from 8 to 29 carbon atoms. It is not known whether the promoting action of the sorbitan addition agent is due to the presence of the sorbitan radical in its hydrolyzed or in its anhydrous state.

Having thus described the invention, what is claimed as new and desired to be secured by Letters Patent is:

1. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof; and heating said mixture until saponification, and the grease making reaction are substantially completed.

2. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent comprising a higher hydroxy fatty acid ester; and heating said mixture until saponification, and the grease making reaction are substantially completed.

3. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent comprising a 12-hydroxy fatty acid; and heating said mixture until saponification, and the grease making reaction are substantially completed.

4. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent comprising 12-hydroxy stearic acid; and heating said mixture until saponification, and the grease making reaction are substantially completed.

5. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent comprising tri-12-hydroxy stearic glyceride; and heating said mixture until saponification, and the grease making reaction are substantially completed.

6. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, an emulsion of metallic sodium, and an addition agent selected from the group consisting of higher hydroxy fatty acids

and esters thereof which insures the reaction's being carried to completion; and cooking said mixture until saponification and the grease making reaction are substantially completed.

7. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, an emulsion of metallic sodium in lubricating oil, and an addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof which insures the reaction's being carried to completion; and cooking said mixture until saponification and the grease making reaction are substantially completed.

8. The method of manufacturing a soda base grease comprising emulsifying molten sodium in a lubricating oil at a temperature above the melting point of the sodium, mixing the emulsion with a lubricating base, a non-hydroxy saponifiable fatty material, and an addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof, and heating the resulting mass until saponification and the grease making reaction are substantially completed.

9. The method of manufacturing a soda base grease comprising emulsifying molten sodium in a lubricating oil while the latter is being agitated in the presence of an oxygen-containing atmosphere, said emulsifying being carried out at a temperature above the melting point of the sodium, mixing the emulsion with a lubricating base, a non-hydroxy saponifiable fatty material and an addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof; and heating the resulting mass until saponification and the grease making reaction are substantially completed.

10. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a material furnishing a non-hydroxy saponifiable fatty material, metallic sodium, acting as a saponifying agent, and 10 to 50% of an addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof, said percentage being taken on the weight of the saponifiable fatty acid; and heating said mixture until saponification, and the grease making reaction are substantially completed.

11. The method of claim 2 wherein the higher hydroxy fatty acid ester is present in an amount varying between 10% to 50% based on the weight of the fatty acid material.

12. The method of claim 3 wherein the 12-hydroxy fatty acid is present in an amount varying between 10% to 50% based on the weight of the fatty acid material.

13. The method of claim 4 wherein the 12-hydroxy stearic acid is present in an amount varying between 10% to 50% based on the weight of the fatty acid material.

14. The method of claim 5 wherein the tri-12-hydroxy stearic glyceride is present in an amount varying between 10% to 50% based on the weight of the fatty acid material.

15. The method of claim 1 wherein the addition agent is present in an amount varying between 15% and 30% taken on the weight of the saponifiable fatty material.

16. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a non-hydroxy saponifiable fatty acid material, metallic sodium, acting as a saponifying agent, and an addition agent selected from the group consisting of the higher hydroxy fatty acids

and esters thereof; and heating said mixture to a temperature varying between 200° F. and 600° F. until saponification, and the grease making reaction are substantially completed.

17. In the method of manufacture of a sodium base grease from a mixture containing a saponifiable fatty material and a lubricating base, the step of substantially completely saponifying and neutralizing the fatty material by molten sodium in the presence of an addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof, said saponifying and neutralization step being carried out at a temperature above the melting point of the molten sodium.

18. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a non-hydroxy saponifiable fatty material, metallic sodium, and an organic addition agent selected from the group consisting of the higher hydroxy fatty acids and esters thereof which prevents the formation of shells of hard reaction product around the sodium globules on the heating of the sodium above the melting point thereof; and heating said mixture until saponification and the grease-making reaction are substantially completed.

19. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent comprising a mixture of 12-hydroxy fatty acid and a polyethylene glycol having a molecular weight above 400; and heating said mixture until saponification, and the grease making reaction are substantially completed.

20. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a saponifiable fatty material, metallic sodium, acting as a saponifying agent, and an addition agent comprising polyethylene glycol having a molecular weight above 400; and heating said mixture until saponification, and the grease-making reaction are substantially completed.

21. The method of manufacturing a soda base grease comprising forming a mixture of a lubricating base, a non-hydroxy saponifiable fatty material, metallic sodium, and an organic addition agent which prevents the formation of shells of hard reaction product around the sodium globules on the heating of the sodium above the melting point thereof, said addition product being selected from the group consisting of the higher hydroxy fatty acids and esters thereof; and heating said mixture for a period of time varying between 10 minutes and 25 minutes.

ARNOLD A. BONDI.

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