Patented May 18, 1943 2,319,405

UNITED STATES PATENT OFFICE

2,319,405

MANUFACTURE OF GREASES

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No Drawing. Application August 3, 1940, Serial No. 351,117

10 Claims. (Cl. 252—39)

This invention relates to novel greases that have a wide variety of uses and more particularly to lubricating greases, and methods for their manufacture.

Lubricating greases as ordinarily made contain water or glycerine and usually both of these materials, which, in many cases, are highly objectionable in a grease. Both of these constituents might be looked upon with reason as objectionable impurities. Either or both of them may cause complete breakdown of a lubricating film in a grease with the tendency for bad effects becoming more severe with increase in temperature or load. Both water and glycerine act as solvents for one part of the grease and not for the remaining part, and they lower the melting point of the grease and aggravate separation of the grease constituents. In those exceptional cases where glycerine may not be objectionable or may, under unusual circumstances, be desirable, a very careful control of the quantity thereof is not only essential but mandatory to the production of a grease suitable for the purpose desired. Due to the varying types of oils employed in grease manufacture, either in the soap for use in the grease or otherwise, accurate control of the glycerine content of lubricating greases has not heretofore been possible.

Having in mind the defects of the prior art methods and compositions, it is an object of the invention to provide a new and improved method of manufacturing lubricating greases. Another object of the invention is to provide new and improved greases and lubricating compositions which give increased satisfaction in many places where lubricating compositions are now used or may be used. Another object of the invention is to simplify the manufacture of greases, while at the same time reducing the cost of manufacture. Still another object is the provision of a method for controlling uniformity and thus superiority in lubricating compositions. Yet another object is to provide a method for making lubricating greases free from objectionable impurities such as grit, glycerine and water, common to most lubricating greases.

Novel and unusual lubricating greases can be prepared by my new and improved method by combining in a molten state, anhydrous soap substantially free of glycerine with mineral oil and/or other lubricating adjuvants. The molten anhydrous soap substantially free of glycerine is advantageously prepared by the process described and claimed in my United States Patent No. 1,918,603 issued July 18, 1933, and United States Patent No. 1,951,511 issued March 20, 1934, wherein one or more of a great variety of different kinds of soap (water-soluble or insoluble) are treated to remove volatile impurities including glycerine with the aid of heat, usually with steam agitation and with substantial exclusion of air or other oxidizing influence.

The soaps are completely saponified and substantially pure. Where glycerine is formed in these soap-making processes it is substantially completely removed and recovered, and the soap produced is anhydrous and free from liquid water or water-containing emulsions unless water may be subsequently added. The anhydrous soaps described in my two patents referred to above and incorporated in my novel lubricating greases are characterized by an unusually low glycerine content or none at all, freedom from volatile impurities, and by a reduced tendency to oxidation and to other deterioration. By these processes it is possible to procure soaps of desired compositions in liquid condition by fusing the soap under conditions which do not cause any sensible decompositoin or deterioration. With the aid of the molten anhydrous relatively pure soap it has been found possible to make intimate and uniform mixtures of these soaps with other lubricating grease materials which it is desirable to incorporate with them. I have found that this is made possible from the fact that the relatively pure anhydrous soaps in molten condition become excellent solvents for many of these materials which at lower temperatures and in the presence of a liquid water phase do not become dissolved therein, but merely emulsified therewith. It should be explained that the solvent action referred to in the case of heated anhydrous materials and anhydrous soap is a mutual one. For instance a melted anhydrous soap free from water and glycerine will actually dissolve a high percentage of a high grade mineral lubricating oil without injuring it in any way, or conversely the mineral lubricating oil, being considered as the solvent, dissolves a high percentage of anhydrous soap. At temperatures sufficiently high for fusion of pure anhydrous soap, as for example in the case of some soaps 290° C. to 300° C., the mutal solution of the anhydrous soap and the mineral lubricating oil is substantially in all proportions, whereas with the use of lubricating oils thus dissolved with hot soap, a considerable

percentage of both soap and oil being present, there is a separation on cooling, to two phases, the lower containing, when cold, the greater concentration of soap and lesser concentration of the mineral lubricating oil, in some instances as little as 20% of mineral oil, while the upper layer will comprise mineral oil to a larger degree and anhydrous soap to a lesser degree.

It will be found that both of these layers, the one richest in anhydrous soap and the one richest in mineral lubricating oil, possess excellent properties as lubricating greases especially for severe duty requirements. Neither of these compositions will suffer physical or chemical breakdown under the most rigid requirements ordinarily met with in extreme cases. In any use at a temperature higher than that at which the two phases separate there will be but one phase, said single phase possessing good lubricating qualities. The two phases may be kneaded together at low temperatures with ease in the dry condition without addition, or with the incorporation of other lubricating material as for instance graphite and will make a superior lubricating grease consisting at ordinary temperatures of two solid phases (or in case graphite has been added, of three solid phases). This physical condition will give the composition a buttery, smooth consistency due to the tendency for one phase to slip against the other and with no tendency for the grease to separate or change in an unfavorable manner due to increase in temperature, as rise in temperature favors a mutual solution of the phases without breakdown of the film of grease.

I may for example in preparing a lubricating grease in accordance with the invention take tallow or lard or horse fat or any of the fats or oils commonly used for this purpose, or mixtures thereof, and make them into an anhydrous sodium soap with the use of dry soda ash and superheated steam agitation, at about 300° C. in the substantial absence of air or other deteriorating influences, and thus obtain a pure soap free from water and glycerine in thin molten condition. As soon as the glycerine has been removed, which takes but a comparatively short time, the desired amount, large or small, of a desirable mineral oil constituent may be run into the soap whereupon the two will mix rapidly and completely due to their substantially complete mutual solubility at the temperature employed. The melting point of the grease thus formed is lower than that of the pure anhydrous soap and it is therefore not necessary to maintain the same high temperature as in making the soap. After the grease has thus been made, agitation may be stopped with reduction of temperature while continuing to exclude air throughout the process as long as the grease is at a temperature above the boiling point of water. A proper degree of subsidence will suffice to permit any chance objectionable solid or gritty impurities to settle out so that with the use of a proper device the grease may be run out free from such impurities. The grease may be run into a proper tank and allowed to separate into two phases if desired, if its composition is such as to form such a separation, or it may be run at once to a cooling-mixer where it may be worked into a finished lubricant.

The process is very flexible while permitting accurate control of all steps and of the finished product. Thus a softer grease may be obtained by employing a higher proportion of mineral oil, and a harder grease by the employment of more soap.

The process gives desirable results which are surprising in the light of experience gained from ordinary greases. Thus, it is not customary to use much if any cocoanut oil in making lubricating greases. This is because soap from this oil is so soluble in water and glycerine water, and is melted and dissolved so readily in these two impurities that any ordinary lubricating grease containing water and glycerine and cocoanut oil soap would liquefy with rise in temperature in use and separate and result in a broken film with inferior lubricating qualities. Also, cocoanut oil yields 30% more glycerine than most other fats and oils and for this reason is not suitable for use in ordinary greases made in known ways owing to the larger amount of glycerine that would be left in such greases.

On the other hand, contrary to common belief, cocoanut oil soap when anhydrous and freed from glycerine is appreciably higher melting than anhydrous tallow soap, for instance, or many other anhydrous sodium soaps. Furthermore, anhydrous potash soaps are generally appreciably higher melting than anhydrous sodium soaps from corresponding fatty material. Potash soaps have increased solubility in the presence of impurities like water and glycerine with very much lowered melting points whereas the melting point of the anhydrous potash soap free from glycerine is very high.

Soaps may also be made from material made in accordance with my United States Patent No. 1,951,511 issued March 20, 1934, or by making higher melting anhydrous soap with suitable fatty material mixed with a large proportion of rosin. I have found that the melting points of some of these anhydrous soaps made from oxidized petroleum compounds by the process of my United States Patent No. 1,951,511 are appreciably higher than the melting points of most anhydrous soaps made from natural fats, also that such anhydrous petroleum soaps have somewhat lower melting points when made in the presence of, or when mixed with, certain high boiling petroleum materials like high boiling mineral lubricating oils, there being at these high temperatures a very considerable degree of mutual solubility. Also, anhydrous rosin soap made by itself in the absence of fatty soaps or fluxing materials has a very high melting point compared with anhydrous soaps made from fats which generally melt around 300° C. if decomposition is avoided.

Accordingly, the present invention permits of the use of these high melting soaps while deriving signal advantages therefrom, as it thus becomes possible to provide a plentiful supply of inexpensive and at the same time superior high viscosity greases for use in duty at high temperatures and particularly suitable for such duty where heavy loads must be continuously carried. These products may be made by incorporating high boiling lubricating materials at high temperatures with these high melting soaps, taking proper precautions to thoroughly agitate the heated mixture to avoid local overheating and to thoroughly exclude air. Before beginning to chill the greases so made, grit and other insoluble solid matter should be separated and removed by sedimentation or otherwise so that it may be excluded from the final product.

It should be at once evident that the ordinary processes of making greases in open grease kettles

could not be employed at all for the manufacture of such high melting, high viscosity, high duty greases, which by the present invention may be made without injury at temperatures that would result instantly in fire if the air were not effectively excluded.

These facts show how with the use of the present process and careful selection of materials employed, stable soaps and stable greases with unusually high melting points suitable for very heavy duty may be obtained, even from those materials previously considered wholly unsatisfactory for such purposes.

Lubricating greases are put to a great variety of uses and some greases are more suitable than others for some uses. For some purposes a grease containing lime soap, or calcium soap, will be found to be better than one made with a sodium or potash soap. The present process lends itself readily to the making of a soap with almost any desired base or bases. Thus for making a calcium soap from tallow I may mix with each 100 lbs. tallow about 50 lbs. good quality pulverized limestone and treat the mixture with superheated steam at about 300° C. in such a way as to exclude air, prevent local overheating, and distil off and collect the glycerine formed. For this purpose I may employ controlled indirect heating as for instance diphenyl vapor under pressure, or diphenyl oxide vapor, or a mixture of the two. In this way I may obtain a purer more completely saponified calcium soap than may be obtained by ordinary processes, and one free from the objectionable impurities, water and glycerine, and because of the fluidity of the soap I may free it completely from gritty impurities in a way that is not possible with ordinary calcium soaps and calcium soap greases. Calcium soap grease prepared by the present process with the aid of suitable mineral lubricating oil, e. g., 70%, 80%, or 90% of a lubricating oil possessing a Saybolt viscosity of 180–200 at 100° F., have special advantages for many purposes. For instance when prepared with proper consistency it is very well suited as a cup grease and it may be used both as a stiff and as a soft grease with grease guns. It is very suitable as a water proof grease for use in cups lubricating water pumps or pumps for other liquids, and is especially well suited to lubricate outboard bearings of propeller shafts.

It is not generally feasible to use other bases than the alkalies and calcium or mixtures thereof, though some others like aluminum and lead are worked into greases with difficulty. The present invention enables one to make special greases with greater ease than they may otherwise be made. I may for instance readily make a magnesium soap grease and find it especially easy to make a calcium-magnesium soap grease that has its own characteristic advantages, by employing a good grade of pulverized dolomite in proper amounts with a desired fatty material. Under the conditions which are well suited for this type of manufacture the saponification may be carried out in a still-like vessel with direct steam agitation at about 300° C. with or without vacuum so as to distil off the glycerine and prevent local overheating. The mixed calcium-magnesium soap melts with even greater ease than the calcium soap alone, and when incorporated with proper mineral oil lubricating fractions makes a very satisfactory grease.

One may have great leeway in selecting the mineral oil constituent for these greases according to their intended uses. Fractions obtained from petroleum oils by fractional distillation are suitable; so also are mineral oil lubricants obtained and purified from crude petroleum by solvent extraction processes. Where greases are to be employed for heavy duty and are required to operate at high temperatures or to sustain great loads it will generally be found desirable to employ the higher boiling or the more viscous fractions of mineral oil and frequently desirable to pick a soap base with a higher melting point, and in all cases it is desirable to choose a composition that will not give a two phase separation in use, with two quite dissimilar phases, so that an easily ruptured film will result and cause wearing surfaces to come into actual contact with one another and even to "seize."

For some uses, requirements are quite opposite from others. Thus, for lubricating curved rails to prevent flange friction, waterproof qualities are desirable so that the grease will not become detached by rain or snow; a more or less permanent consistency is desirable even on long exposure so that the grease will continue to function, and it must also display reasonably good lubricating qualities though certainly not the same as a grease used to lubricate a heavily loaded bearing housing, or a rapidly revolving axle, or a wrist pin. A good cheap grease may be made by the present invention with a cheap fatty matter mixed with cheap rosin to make an anhydrous soap which may be mixed with cheap petroleum products such as still tar or fuel oil or even with some asphalt and cheap oil.

For some purposes materials other than the ordinary soap-making materials may be employed in the process to advantage as for instance naphthenic acid neutralized by the process with a suitable base, or used in conjunction with fatty or rosin matter. Tall oil, waxes and wool fat soaps may also be used advantageously by the present novel process for some types of lubricating greases. I may make good greases by the present process using fatty acids with complete conversion of their acid qualities to "neutral" greases. Lubricating greases may be used with or without addition of graphite, chlorinated hydrocarbons, or the like. Greases made with one class of materials may be blended with greases made from other materials.

The process may be modified by making the soaps by some other method of saponification and then purifying the soap thus made by the heat-steam treatment before making the final lubricating grease. Although there is no special advantage in doing so, the saponification may be performed in the presence of some of the mineral oil product. The melt thus formed is lower melting than the one from pure soap but on the other hand some of the mineral oil product will distil over with the steam and will have to be collected to avoid loss, and the greater advantage is obtained when very little of the mineral oil product is distilled over.

Greases made by the present invention, owing to the absence of water and glycerine and objectionable acid impurities, exert practically no corrosive qualities on metals on which they are used at any temperature, though such action is a common fault of many ordinary greases.

While I have illustrated my invention with specific examples, it is to be understood that it is not to be restricted thereto, but is to be limited only by the broad principles of the disclosure and by the claims.

I claim:

1. The process of manufacturing lubricating grease which comprises making a substantially pure anhydrous soap in molten condition substantially free from glycerine, unsaponified, and volatile unsaponifiable matter, grit and other impurities, and mixing therewith a mineral base lubricating material while the two are in a substantially fused anhydrous condition, with substantial absence of air, and cooling the product with stirring with substantial absence of air.

2. The process of manufacturing lubricating grease which comprises saponifying a fat or fatty oil with a base and heating the mixture thus formed with the aid of thorough agitation and flow of steam and substantial exclusion of air to a temperature in excess of the melting point of the resulting anhydrous soap, volatilizing therefrom the glycerine that is formed, while continuing the agitation, flow of steam, and exclusion of air, and mixing with the resulting molten anhydrous soap in the substantial absence of air a high boiling mineral lubricating oil which is appreciably mutually soluble with said soap at the temperature employed for said mixing, and cooling the product with stirring.

3. The process of making lubricating greases which comprises intimately mixing in the substantial absence of air a fused anhydrous soap made from coconut oil and substantially free from glycerine with a high-boiling mineral lubricating oil.

4. The process of making lubricating greases which comprises mixing at a high temperature with the exclusion of air, a soap substantially free of water, volatile impurities and glycerine with a mineral oil lubricant in such proportion that said soap and said lubricant have a high degree of mutual solubility under the mixing conditions and in such proportion that the product thereof will form two separate grease phases upon cooling, agitating and maintaining a temperature above about the melting point of the anhydrous soap during said mixing to produce a high degree of fluidity and mutual solubility of said soap and said lubricant, cooling said mixture without harmful exposure to air to form two grease phases, and separating the two grease phases.

5. The process of making lubricating greases which comprises mixing at a high temperature in the substantial absence of air, a soap substantially free of water, volatile impurities and glycerine with a mineral oil lubricant in such proportion and at such temperature that said soap and said lubricant have a high degree of mutual solubility under the mixing conditions and in such proportion that the product thereof is capable of forming two separate grease phases upon cooling, agitating and maintaining a temperature above about the melting point of the anhydrous soap during said mixing to produce a high degree of fluidity and mutual solubility of said soap and said lubricant in the mixture, cooling said mixture without harmful exposure to air, and stirring during cooling to prevent segregation of said grease phases.

6. A lubricating grease substantially free from water, glycerine, volatile impurities and grit, comprising a mineral oil lubricant, calcium and magnesium soaps, the calcium and magnesium soaps being in approximately the same relative proportions as the calcium and magnesium occurring in dolomite limestones.

7. The process of manufacturing lubricating greases which comprises the joint incorporation therein, in the absence of liquid water and glycerine, of a mineral oil lubricant and of a high melting anhydrous soap made from oxidized petroleum hydrocarbons, said anhydrous soap having been modified by subjection to a temperature above about the melting point of the anhydrous soap and to thorough agitation in the absence of air with the aid of a current of steam for a period of time sufficient to effect the substantial removal of the hydroxyl groups from hydroxy soaps therein.

8. The process of manufacturing lubricating greases which comprises the joint incorporation therein, at elevated temperatures and above the melting temperature of said greases in the substantial absence of liquid water, volatile impurities and glycerine, of a high melting anhydrous rosin soap and a high boiling lubricating material.

9. A lubricating grease substantially free from water, glycerine, volatile impurities and grit, comprising a mineral oil lubricant and an anhydrous soap made by oxidation of petroleum hydrocarbons, which soap has been subjected to a temperature above about the melting point of the anhydrous soap, in the absence of air, for a period of time sufficient to effect the substantial removal of the hydroxyl groups from hydroxy soaps therein.

10. A lubricating grease substantially free from water, grit, glycerine and volatile impurities occurring in fats and fatty oils, comprising mineral lubricating oil and anhydrous soap made from naturally-occurring glycerides, which soap has been subjected to a temperature at least as high as the melting point of the anhydrous soap, in the absence of air, for a period of time sufficient to reduce the tendency of the soap to oxidize and otherwise to deteriorate.

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CERTIFICATE OF CORRECTION.

Patent No. 2,319,405. May 18, 1943.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, second column, line 52, for "mutal" read --mutual--; page 4, second column, line 32, claim 8, for "at elevated temperatures" read --at an elevated temperature--; line 33, after the word "greases" insert a comma; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 29th day of June, A. D. 1943.

Henry Van Arsdale,
Acting Commissioner of Patents.

(Seal)