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LUBRICANT GREASE

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This invention relates to lubricant greases and methods for making the same.

This application is a continuation-in-part of our co-pending application Serial No. 38,966, filed September 3, 1935.

One of the main objects of this invention is to provide a method of making lubricant greases which will be stable at virtually every temperature and pressure at which their use may be required.

Another object is to provide a method of making greases such that the danger of cracking or oxidation of their mineral oil-content is materially lessened.

A further object is to provide a method of making greases of uniform consistency and texture which may vary from extreme hardness to extreme softness, in accordance with the proportions of ingredients used.

A still further object is to provide a lubricant grease whose stability will be substantially higher than other greases known to the art, and which may be melted and cooled, distilled, or violently agitated without breaking down.

A still further object is to provide a truly anhydrous grease devoid of the fibrous or grainy structure which characterizes greases now commonly sold under the misnomer "anhydrous."

Further objects of the invention will in part be obvious and will in part appear hereinafter, it being understood that the described embodiments are to be taken as non-limiting examples given for illustrative purposes only.

In the course of the experiments made in connection with this invention, we have examined all of the so-called anhydrous greases known to the art. Without exception these greases were found to include a substantial volume of water ranging from .5% to 1% and more. Moreover, their physical characteristics appear those of an oil-in-water emulsion. The presence of this water is believed to be a primary reason for decomposition and failure of these greases at high temperatures and pressures.

No matter how carefully prior grease-making processes have been carried out, there has never been any definite assurance that all of the water originally introduced in one of the ingredients was eliminated. Grease production has been more or less of a hit-or-miss proposition, where human skill has been the real "control factor" and where even the most skilled operative has duplicated his best product but seldom.

The present method insures driving off substantially all of the water originally introduced

into the batch and produces a uniform anhydrous product without any need for employing skilled operatives. Some of the greases produced by this invention show no water-content whatever, although in others the water-content may range as high as about $\frac{1}{10}$ of 1%. In general, this last value of $\frac{1}{10}$ of 1% may be considered as virtually the highest water-content which will be found in the product of this invention.

A further result of the invention is that the emulsion phases are reversed in the product, and to the extent that there is any water, our grease is a water-in-oil emulsion, the water being in disperse phase and showing no tendency to separate out or cause decomposition. There appears to be a critical point somewhere between the water-content of the grease of the prior art and the $\frac{1}{10}$ of 1% water-content of our new grease. Just where this critical point lies has not been determined as yet, but by "anhydrous" in the claims, is meant grease whose water-content is below this critical percentage. The following test has been devised for determining whether or not this result has been attained:

To a sample of grease, there is added an approximately equal volume of kerosene, and the latter is then distilled off. The distillate is preferably condensed in a graduate, and any water coming off with the kerosene will separate by gravity and can be measured. If the percentage is in the order of $\frac{1}{10}$ of 1% or less, the conclusion is that the grease is anhydrous and hence stable.

However, there is a simpler means of reaching a conclusion from this test. If the water-content of the original grease was below the critical point, the product remaining after the distillation will congeal to a homogeneous pasty consistency. If on the other hand, the water-content had been above the critical point, the remaining product will break down to a consistency like that of sand and water. Of all the greases tested during the development of this invention, only the product of this invention passed this test.

The present invention is based to some extent on the discovery that the soaps used in grease manufacture will not hold water under partial pressure distillation with kerosene (or other hydrocarbon oils of the average boiling range of kerosene), and we make use of this discovery in the manufacture of anhydrous lubricant greases in the manner typified by the following general description of our manufacturing process.

Generally speaking, the process of the invention comprises mixing together a soap-forming substance, a hydroxide and a non-aqueous dilu-

ent of the nature of a hydrocarbon or its derivatives, saponifying the mixture under partial pressure of the diluent, and adding a lubricant oil to the resulting soap.

Among the soap-forming substances which may be used for the invention are fats such as tallow and coconut oil, fatty acids such as stearic, palmitic, oleic, etc., naphthenic acids, and various petroleum sludges.

Among the hydroxides that may be used are those of alkali metals such as sodium, potassium, lithium, etc., or metals such as calcium, aluminum, lead, magnesium, tin, zinc, etc. These hydroxides may be used either in the dry state or in solution, as will be explained hereinafter.

The diluent used depends in large measure upon the nature of the soap-forming substance. Thus for fats such as coconut oil, a kerosene oil will be satisfactory, and it is preferred to use a cut having a boiling range of approximately 180°-230° C. For fatty and naphthenic acids, a lower boiling diluent such as benzene, solvent naphtha or other light naphthas, or toluene may be used.

The purpose of the hydrocarbon diluent is three-fold. First, it may enable the saponification reaction to be carried out at a higher temperature, thus speeding the reaction. Second, it simplifies the removal of any glycerine formed as a by-product of that reaction. Third, it both makes possible and insures the production of a truly anhydrous grease as the final product, for in distilling off, the diluent carries with it the water which may have chemically combined with or been originally present in the other ingredients of the mix.

The various steps making up the process of the invention involve first mixing a quantity of a suitable soap-forming substance, such as those mentioned above, with at least an equal volume of a diluent suitable for that substance. Next there is added a suitable hydroxide, either dry or in solution, and preferably in the exact amount theoretically necessary to neutralize all the fat present.

The resulting mixture is distilled at least until sufficient of the diluent has been carried off to insure appreciably complete elimination of water. This point can easily be determined, for as long as water is present, the diluent will come off cloudy, and thereafter it will be clear. Substantially all the glycerine present will also come off with the diluent.

After the distillation has been carried to the point of insuring the elimination of water, and while the resultant gel is still hot, a lubricant oil is incorporated therewith, the nature and amount of such oil depending upon the lubricity required and the oil preferably being heated to approximately the temperature of the gel before being added.

It is important to note that the saponification process is carried out at such a high temperature (200° to 225° C.) that this neutralizing reaction to completion with no free acid or free alkali remaining in the grease. The grease is thus neutral and not corrosive. This is not possible with prior processes where the soap forming reaction is carried out at temperatures in the order of 100° C. At such a low temperature the reaction is slow and must be expedited by using an excess of reacting material, with the result that corrosive agents remain in the final product.

In cases where free neutral fat is required we may use slightly less alkali than the theoretical

amount, but we never use an excess. When acids, rather than fats, are to be neutralized, alkali or metal carbonates may be used as the neutralizing agent.

The following detailed example of our process is merely typical of one method of carrying out the invention and is not to be construed in a limiting sense.

Dissolve 400 parts of coconut oil in a still in 400 parts of kerosene, preferably of a cut having a boiling range of 180°-230° C. To this add 112 parts of caustic soda, preferably in flake or powder form. Heat this mass with continuous agitation. When it reaches a temperature between 180° and 200° C. the kerosene begins to distill off, carrying with it water and glycerine which stratify out in the distillate.

Continuation of the distillation until all water has been removed will result in distilling out about 350 parts of the kerosene originally present and roughly 30 of the 56 parts of glycerine produced by the reaction. Distillation may be continued until substantially all of the kerosene and glycerine has been driven out and recovered.

In the event that the kerosene contains fractions of higher boiling point the final distillation should be done under vacuum to assure the removal of these fractions without raising the still temperature high enough to cause decomposition of the grease.

When distillation is complete, there are added 800 parts of mineral lubricating oil of any desired viscosity and at a compounding temperature in the order of 200° C., and the agitation and heating are continued until a thoroughly homogeneous mixture has been formed. Upon cooling, the entire mass gels and forms a stable anhydrous grease.

By varying the proportions of soap to oil, the hardness or softness of the grease may be varied within wide limits. By substituting an alkali for a metal hydroxide or mixing alkali and metal hydroxides together, pure or mixed greases may be produced as desired.

This invention also makes possible the production of grease concentrates, consisting of various types of soaps protected by a relatively small amount of the hydrocarbon diluent.

Similarly, compound oil greases containing mineral and fixed oils in combination may be produced by adding various other oils or mixtures thereof to the mineral oil. These mixed oils may also be produced by using a smaller amount of alkali or metal hydroxide and thus failing to saponify a certain amount of neutral oil or fat.

The foregoing process is not only simpler and cheaper but produces a better product than is possible with the commonly accepted methods of grease manufacture. These methods, subject to only minor modifications are; (1) hydrous saponification of fat and the incorporation of mineral oil in the hydrous soap, heating to a low temperature being (fallaciously) relied upon to remove the water; (2) neutralization of fatty acids with hydrous alkali or metal hydroxides and the incorporation of mineral oil in the resultant soap; and (3) forming a metallic soap precipitate by using a metallic salt solution to effect double decomposition of soda soap and then incorporating mineral oil. None of these processes involve or are even capable of close or automatic control. They do not insure the removal of water or prevent the presence of corrosive agents in the grease. The recovery of glycerine as a

by-product of such processes is either quite expensive or entirely unfeasible, since it constitutes only about 5% of a lye and water solution after the separation of the soap.

With our process the glycerine or part of it may be left in the grease and that part distilled off is a pure product involving no extra expense to recover.

Our process requires no specialized type of apparatus for carrying it out. It results in a pure, neutral, stable, anhydrous grease and eliminates the need for elaborate, time-consuming and costly operations which are frequently undertaken in an attempt to remove such impurities as free alkali, free acid, metal salts and water from the greases made by standard methods.

Various changes and alterations might be made in our process without departing from the spirit of the invention or the scope of the appended claims, wherein the term "kerosene" is intended to include all mineral oils boiling at approximately the same temperature.

It will be understood, of course, that various details in the carrying out of the invention as described above may be modified in various ways as may become apparent to one skilled in the art without departing from the spirit and scope of the invention, and that therefore it is intended and desired to embrace within the scope of this invention such modifications and changes as may be necessary to adapt it to varying conditions and uses; and it is to be understood also that the following claims are intended to cover all of the generic and specific features of the invention as herein described, and all statements of the scope of the invention which as a matter of language might be said to fall therebetween.

We claim:

1. A process of making lubricant grease, which comprises mixing a soap-forming substance, at least an equal volume of a hydrocarbon diluent having a boiling point higher than that of water, and a caustic alkali hydroxide, distilling said mixture at a temperature within the boiling range of said diluent until the water combined with or liberatable by the reaction of the ingredients has been eliminated together with most of the diluent, and adding a lubricant oil to the saponified residue.

2. A process of making anhydrous lubricant grease, which comprises distilling a mixture of a soap-forming substance, a hydrocarbon diluent having a boiling point higher than that of water, and a metal hydroxide at a temperature within the boiling range of said diluent, the amount of said diluent being at least equal in volume to the amount of soap-forming substance used, and then adding a lubricant oil to the saponified residue from which most of the diluent is removed.

3. A process of making an anhydrous lubricant grease for use under high pressures and temperatures which consists in distilling a mixture of kerosene, a soap-forming substance including acids, and a metal hydroxide at a temperature within the boiling range of kerosene, to drive off with the oil the water combined with, or liberatable by the reaction of the ingredients, the amount of kerosene used being at least equal in volume to the amount of soap-forming substance used, and then adding a lubricant oil to the anhydrous saponified residue from which most of the kerosene is removed.

4. A process of making an anhydrous lubricant grease for use under high pressures and temperatures which consists in distilling a mixture

of kerosene, a soap-forming substance including acids, and dry metal hydroxide at a temperature within the boiling range of kerosene, to drive off with the oil the water combined with, or liberatable by the reaction of the ingredients, the amount of kerosene used being at least equal in volume to the amount of soap-forming substance used, and then adding a lubricant oil to the anhydrous saponified residue from which most of the kerosene is removed.

5. A process of making an anhydrous lubricant grease for use under high pressures and temperatures which consists in distilling a mixture of kerosene, a soap-forming substance including acids and a metal hydroxide solution at a temperature within the boiling range of kerosene, to drive off with the oil the water combined with, or liberatable by the reaction of the ingredients, the amount of kerosene distilled being at least equal in volume to the amount of soap-forming substance used, and then adding a lubricant oil to the anhydrous saponified residue from which most of the kerosene is removed.

6. A process of making an anhydrous lubricant grease for use under high pressures and temperatures which consist in distilling a mixture of kerosene, a soap-forming fatty substance and a metal hydroxide at a temperature between 180° C. and 230° C. to drive off with the oil the water combined with or liberatable by the reaction of the ingredients, the amount of kerosene used being at least equal in volume to the amount of fatty substance used, and then adding a lubricant oil to the anhydrous saponified residue in the still after heating said oil to a temperature in the order of 200° C.

7. A process of making an anhydrous lubricant grease for use at high temperatures and pressure which consists in distilling a mixture of kerosene, a metal hydroxide and a fatty glyceride including a fatty acid, said hydroxide being present in an amount no greater than the amount theoretically capable of neutralizing all of the fatty acid, distillation being carried out at temperatures in the order of 200° C. so that the reaction of the hydroxide with the fatty acid proceeds to completion and the water originally present or liberated by the reaction is driven off with the kerosene and at least part of the glyceride liberated in the reaction is likewise driven off, the amount of kerosene used being at least equal in volume to the amount of fatty substance used, then adding a lubricant oil to the non-alkaline saponified residue in the still, and agitating to produce a homogeneous anhydrous grease.

8. A method as set forth in claim 3, wherein the hydroxide is present only in sufficient amount to neutralize all of the fatty acid and the saponified residue of the distillation process contains only neutral fat.

9. A method as set forth in claim 3 wherein higher fractions present in the kerosene are eliminated by distillation under vacuum after the complete elimination of water.

10. An anhydrous grease for use at high lubricant temperatures and pressure, consisting of mineral lubricating oil homogeneously mixed with anhydrous soap, characterized by the fact that it contains not more than $\frac{1}{10}$ of 1% of water, and produced by distilling a mixture of a soap-forming substance, at least an equal volume of a hydrocarbon diluent having a boiling point higher than that of water, and a metal hydroxide until all chemically and mechanically combined water is removed, removing the excess diluent, and

adding said lubricating oil to the saponified residue.

11. An anhydrous grease for use at high lubricant temperatures and pressure, consisting of mineral lubricating oil homogeneously mixed with anhydrous soap, characterized by the fact that it contains not more than $\frac{1}{16}$ of 1% of water, said water being in disperse phase in said oil, and produced by distilling a mixture of a soap-forming

substance, at least an equal volume of a hydrocarbon diluent having a boiling point higher than that of water, and a metal hydroxide until all chemically and mechanically combined water is removed, removing the excess diluent, and adding said lubricating oil to the saponified residue.

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