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METHOD FOR DEGASIFYING AN ORGANOPHILIC CLAY-BODIED LUBRICANT

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This invention relates to bodied lubricants. In one aspect this invention relates to a method for preparing bodied lubricants. In another aspect this invention relates to a method for preparing bodied lubricants wherein an organophilic clay employed to body said lubricant is degasified prior to being dispersed in the lubricating liquid.

In the production of bodied lubricants, it is customary to prepare solutions or dispersions of soaps in lubricating oils, the function of the soaps being to body up the oils to grease consistency. Soaps commonly used for this purpose are the metal salts of long carbon chain saturated and unsaturated fatty acids as, for example, potassium oleate, magnesium stearate, aluminum palmitate, etc. It is commonly observed that lubricants consisting of these soap-in-oil dispersions have a relatively high thermal coefficient of viscosity and that at temperatures often encountered in machinery under normal operating conditions these greases become fluid, resulting in loss of the lubricant and damage to the machinery. This is to a considerable extent a function of the melting point of the soap. Thus, the magnesium, zinc, or aluminum soaps would not be desirable for high temperature applications because of their low melting points. The alkali soaps, while possessing higher melting points, up to 175° C., are subject to hydrolysis by moisture, which gives rise to deleterious alkalinity, and are, therefore, not entirely suited to some conditions of use. Other additives, such as clay and asphaltic materials, are sometimes used to increase the load bearing capacity and the high temperature viscosity of greases; the clays, however, introduce abrasive material, and the organic bodying agents increase the low temperature consistency to such a degree that starting of cold machinery is difficult.

One more recently developed type of bodied lubricant is one formed by colloidally dispersing an organophilic clay, commonly called "Bentone," in an organic lubricating liquid, such as a petroleum lubricating oil, a synthetic lubricating oil, such as a diester or heavier polyester, or a mixture of such lubricating oils, in a manner to obtain a gel or "grease." The clays which are useful as starting material for making the modified clay in accordance with this invention are those exhibiting substantial base-exchange properties, and particularly those exhibiting comparatively high base-exchange properties and containing cations capable of more or less easy replacement. Some clays, characterized by an unbalanced crystal lattice, are believed to have negative charges which are normally neutralized by inorganic cations. As found in nature, therefore, they exist as salts of the weak clay-acid with bases, such as the alkali- or alkaline-earth metal hydroxides.

The clays particularly contemplated herein include the montmorillonites, viz., sodium, potassium, lithium, and other bentonites, particularly of the Wyoming types; magnesium bentonite (sometimes called hectorite) and saponite; also nontronite and attapulgite, particularly that of the Georgia-Florida type. Bentonites which are

particularly useful are the swelling bentonites of the Wyoming type and the swelling magnesium bentonites of the hectorite type.

An organophilic bentonite, commonly used in the manufacture of bodied lubricants from mineral lubricating oils, is formed by replacing the exchangeable inorganic cations of the bentonite with organic cations from salts of aliphatic, cyclic, aromatic and heterocyclic amines, primary, secondary and tertiary amines and polyamines, and quaternary ammonium compounds, such as dimethyldicetylammonium hydroxide. The bentonite is first hydrated in a very dilute dispersion to separate the unit particles and is then purified by super-centrifuging to achieve complete removal of non-clay impurities which would impart an abrasive character to the finished lubricant. The reactive exchangeable inorganic cation, such as sodium or potassium, of the dispersed bentonite is replaced by the addition of the salt referred to above and the modified clay is then filtered, washed, dried and ground into a fine powder. Increasing the chain length of an alkyl ammonium cation used to replace the exchangeable inorganic cation increases the organophilic properties of the modified bentonite. A more complete disclosure of the preparation of organophilic bentonite will be found in United States Patent 2,531,427, issued to E. A. Hauser.

Bentonite greases are customarily made from Bentone 34, and other organophilic clays in three general steps: (1) a slurry is prepared by stirring the clay into an organic lubricating liquid; (2) a dispersion aid is generally added to the slurry and the stirring is continued until the mixture forms a weak "preliminary gel"; (3) the preliminary gel is then run through a colloid mill wherein a final gel (grease) is formed. Such greases are very frequently "cloudy" or "hazy" in appearance. This cloudy or hazy appearance is generally due to the presence of dispersed gas, generally air, in the grease. Not only are such greases lacking in a pleasing appearance but when the dispersed gas is air, said air serves as foci for the beginning of deteriorative oxidation.

I have found that in most instances the dispersed gas in such a grease is air and results from air entrapped in the dry clay. I have further found that said air can advantageously be removed prior to colloidally dispersing the clay in the organic lubricating liquid. Thus, broadly speaking, my invention comprises an improved method for the preparation of bentone greases wherein the clay is degasified or deaerated prior to colloidally dispersing said clay in the organic lubricating liquid.

An object of this invention is to provide an improved lubricant. Another object of this invention is to provide an improved bodied lubricant. Another object of this invention is to provide an improved lubricant bodied by an organophilic clay. Another object of this invention is to provide an improved method for the preparation of a lubricant bodied by an organophilic clay wherein said clay is degasified or deaerated prior to being colloidally dispersed in the organic lubricating liquid which forms the base of said lubricant.

Still other aspects, objects and advantages of the invention will be apparent to those skilled in the art upon reading this disclosure.

Thus, according to the invention, there is provided an improved method for the preparation of a bodied lubricant from an organic lubricating liquid and an organophilic clay which comprises degasifying or deaerating said clay prior to colloidally dispersing said clay in said organic liquid.

It is to be noted that the clay is degasified or deaerated prior to colloidally dispersing the clay in the organic lubricating liquid. While it may be that some of the clay is dispersed in the organic lubricating liquid dur-

ing the slurrying step, any gel thus formed is a weak "preliminary gel" which can be degasified according to the invention. Thus the deaeration or degasification of the clay can be carried out at any time prior to the formation of the final gel (grease), i.e., at any time prior to step 3 (above) in which all of the clay is colloidally dispersed in the organic lubricating liquid.

A presently preferred method for deaerating or degasifying the clay is by the application of vacuum to said clay either with or without agitation. Such deaeration can be batch or continuous and can be applied to the dry clay per se or to a slurry of the clay in the organic lubricating liquid. However, since the clays employed to body these types of lubricants are finely divided, it is generally less convenient to deaerate the dry clay. Therefore, a presently preferred method according to the invention comprises slurrying the organophilic clay in the organic lubricating liquid; subjecting said slurry to vacuum conditions; and then colloidally dispersing said clay in the organic liquid to form the bodied lubricant. When the slurry is deaerated the deaeration can be carried out by applying vacuum during the slurrying operation or after completion of the slurrying operation. The mixture of clay and lubricating liquid can also be degasified by applying vacuum prior to the stirring in the slurrying step.

In some instances, it is advantageous to heat the mixture or work the mixture in a milling operation. However, such steps are not always necessary. In most instances it will be desirable to add a dispersion agent to the slurry. The mixture is then usually passed through a colloid mill in order to thoroughly disperse the clay in the lubricating liquid. Again, the final dispersion can be accomplished rather easily while using a cold oil. The final dispersion can be made even more readily, however, by heating the oil to a temperature below its decomposition point but preferably to a temperature only slightly above atmospheric temperature.

As stated, when operating according to my invention, I deaerate the organophilic clay by the application of vacuum prior to colloidal dispersion of said clay in the organic lubricating liquid. If desired the dispersion step can also be carried out under vacuum conditions, or for that matter, the entire operation can be carried out under vacuum conditions. However, neither of these is necessary for the preparation of an improved lubricant. It is only necessary to deaerate the clay prior to its colloidal dispersion in the organic lubricating liquid. Therefore, for economic reasons I prefer to carry out the deaeration at only one point in the process, i.e., either on the dry clay or preferably on the clay after it has been slurried with the organic lubricating liquid but prior to being colloidally dispersed in said liquid.

In addition to the previously mentioned improved appearance and enhanced oxidation stability of lubricants prepared according to my invention there are other advantages to preparing lubricants according to the method of my invention, i.e., deaerating the clay prior to formation of the final gel (grease) rather than deaerating the final grease. These advantages are: (1) When the vacuum is applied to a slurry of the clay in the organic lubricating liquid the bubbles of air (expanded by the vacuum) rise to the surface of the slurry unimpeded due to the non-gelled (or at most only slightly gelled) nature of the slurry. As will be understood by those skilled in the art, deaeration of a gel is a much more difficult procedure due to the low mobility of the trapped bubbles in the gel. (2) The removal of air bubbles from the slurry results in better contact between the clay and the organic liquid, and therefore improved wetting of the clay by said organic liquid is brought about by the evolution of the bubbles in the clay. The improved wetting and contact between the clay and the liquid results in improved dispersion of said clay in said liquid when the final dispersion step is carried out.

Still another advantage, in those instances where a dispersion aid is employed, is that less dispersion aid is required for the same degree of dispersion when deaeration is effected prior to the final dispersion step.

My invention can be employed in connection with any organophilic clay capable of gelling an organic lubricating liquid. Organophilic bentonites, prepared as described above, are presently preferred clays. Examples of such organophilic bentonites are "Bentone 18" and "Bentone 34" both of which are commercially available from the Baroid Sales Division of the National Lead Company, New York, N.Y. The percentage of organophilic bentonite clay which can be used to produce a satisfactory lubricant according to my invention usually lies within the range of 2 to 35 weight percent based on the finished lubricant. I prefer to use a clay loading in the range of 3 to 15 weight percent, based on the finished lubricant, since such loadings result in excellent bodied lubricants and it is economically desirable to use as low clay loadings as possible and yet obtain the desired degree of bodying.

Any organic lubricating liquid capable of being bodied or gelled to a grease of the desired consistency can be used in preparing bodied lubricants according to my invention. The organic lubricating liquid comprises the major ingredient of a bodied lubricant. A presently preferred organic lubricating liquid is a mineral lubricating oil, more preferably a petroleum lubricating oil; however, the organic lubricating liquid can be a mixture of a lubricating oil with other oleaginous substances. Suitable mineral lubricating oils include petroleum lubricating oil; polyester oils; silicone fluids, which are usually oxidant resistant; synthetic lubricants produced by the Fischer-Tropsch, Synthol, Synthine and related processes; synthetic lubricants produced by the Bergius process such as by hydrogenation of coal, peat, asphalt, petroleum residues and the like; organic synthetic lubricants such as alkyl esters of various organic acids; and synthetic lubricants produced by polymerization processes including silicon polymers.

A presently preferred dispersion agent is a nitroparaffin selected from the group consisting of nitromethane, nitroethane, 2-nitropropane and 1-nitropropane as disclosed and claimed in my co-pending application Serial No. 453,721, filed September 2, 1954, now Patent 2,833,720.

Other dispersion agents disclosed by the prior art and which can be employed in connection with my invention are ethyl acetate, acetic acid, acetone, methyl alcohol, ethyl alcohol, benzoyl chloride, butyl stearate, coconut oil, cyclohexanone, ethylene dichloride, ethyl ester, furfural, isoamyl acetate, methyl ethyl ketone, and nitrobenzene. The preferred dispersion agents can be used alone or in combination with the above mentioned other dispersing agents.

The amount of dispersing agent which is utilized in a grease prepared according to my invention is dependent upon not only the specific dispersing agent, but is also dependent upon the concentration of organophilic clay, the type of said clay, such as "Bentone 18," "Bentone 34," etc., and the specific organic lubricating liquid which is used. The amount of dispersing agent which is used varies above 0.25 weight percent, generally within the range of from 0.25 to 6 weight percent, the specific amount once again being dependent upon the clay concentration, the type of clay used, and the organic lubricating liquid used.

Minor proportions of conventional oxidation inhibitors, such as mercaptoaryl thiazoles, aminophenols, phenylene diamines, and phenyl-α-naphthyl amine, can be incorporated in the grease for the purpose of improvement of the oil base.

The amount of vacuum employed according to the invention will depend upon the amount of entrapped air or other gas in the clay. Any degree of vacuum from a

partial vacuum to substantially complete vacuum can be used. Generally the degree of vacuum employed will be within the range of less than 1 millimeter of mercury to 20 millimeters of mercury.

Better understanding of the invention will be obtained upon reference to the following specific examples which are meant to be exemplary and not to be unduly limiting of the invention.

Example I

A grease was prepared by mixing 5.5 parts by weight of "Bentone 34" with 92.5 parts by weight of a high V.I. SAE 20 lubricating oil blending stock at room temperature. 2 parts by weight of nitromethane were then added and the mixture was stirred with a mechanical mixer for 30 minutes to form a weak "preliminary gel." The slurry thus formed was then passed through a laboratory model Charlotte ND–1 colloid mill at a clearance of 0.003 inch to disperse the clay into the lubricating oil and form the final gel or grease. This grease, designated as grease No. 1, was "cloudy" in appearance due to the presence of a great number of air bubbles ranging in size from microscopic to approximately ⅛ inch in diameter.

Example II

A second grease was prepared by mixing 5.5 parts by weight of "Bentone 34" with 92.5 parts by weight of a high V.I. SAE 20 lubricating oil blending at room temperature. The mixture was then deaerated by applying vacuum to the mixing vessel. During about the first 10 minutes of the deaeration period only a partial vacuum could be applied because the mixture tended to "bubble up" or "boil." The mixture was then subjected to a vacuum less than 1 mm. of mercury for about 5 minutes to complete the deaeration. 2 parts by weight of nitromethane were then added, the mixture was then mixed for 30 minutes as before. The resulting weak "preliminary gel" or slurry was then milled on the same mill, in the same manner as for grease No. 1, to form the final gel or grease. This grease, designated as grease No. 2, was clear and "bright" and possessed a pleasing appearance. There were no air bubbles in grease No. 2.

Example III

Samples of grease No. 1 and grease No. 2 were placed in small flat bottom glass dishes on a hot plate for 5 minutes and heated to a temperature of approximately 300° F. Grease No. 1 appeared to form additional bubbles, "puffed up," and became opaque. Grease No. 2 remained clear and "bright."

The above examples demonstrate the superiority of grease No. 2 prepared according to my invention, i.e., deaerating or degasifying the organophilic clay prior to dispersing same into the lubricating liquid. Greases thus prepared according to my invention not only have a more pleasing appearance, as shown by a comparison of Examples I and II, but are also superior lubricants because of the absence of gas bubbles which cause the grease to "swell" or "puff up" when heated to mild temperatures, such as would be encountered in many ordinary services. The presence of such gas bubbles could cause a failure of the mechanism being lubricated.

Greases prepared according to the prior art can be deaerated or degassed after the final milling step. However, as previously mentioned, such deaeration or degassing is much more difficult to carry out, and consequently usually not as complete, due to the low mobility of the grease.

Various modifications of the invention can be made or carried out in the light of the above disclosure without departing from the spirit or scope of said disclosure or the appended claims.

I claim:

1. In a process for manufacturing a bodied lubricant wherein an organophilic clay is colloidally dispersed in an organic lubricating liquid the improvement which comprises degasifying said clay prior to colloidally dispersing said clay in said liquid.

2. In a process for manufacturing a bodied lubricant wherein an organophilic clay is colloidally dispersed in an organic lubricating liquid the improvement which comprises degasifying a mixture of said clay and said liquid prior to colloidally dispersing said clay in said liquid.

3. A method for the manufacture of a bodied lubricant which comprises slurrying an organophilic clay in an organic lubricating liquid; subjecting said slurry to vacuum conditions; and then colloidally dispersing said clay in said liquid to form said lubricant.

4. A method according to claim 3 wherein said slurry is subjected to said vacuum condition during the slurrying operation.

5. A method according to claim 3 wherein said slurry is subjected to said vacuum condition after the slurrying operation.

6. A method according to claim 3 wherein said clay is an organophilic bentonite and said organic lubricating liquid is a mineral lubricating oil.

7. A method according to claim 6 wherein said bentonite is employed in an amount between 65 and 98 weight percent and said mineral lubricating oil is employed in an amount between 2 and 35 weight percent, both of said amounts being based on the finished lubricant.

8. A method for the manufacture of a bodied lubricant which comprises the steps of: slurrying a gelling amount of an organophilic clay in an organic lubricating liquid under vacuum conditions so as to simultaneously deaerate said slurry during its formation; incorporating a dispersing agent in said slurry; and then colloidally dispersing said clay in said liquid to form said lubricant.

9. A method according to claim 8 wherein said clay is an organophilic bentonite; said organic lubricating liquid is a mineral lubricating oil; and said dispersing agent is a nitroparaffin selected from the group of nitromethane, nitroethane, 2-nitropropane and 1-nitropropane.

10. A method according to claim 9 wherein said bentonite is employed in an amount between 65 and 97 weight percent, said mineral lubricating oil is employed in an amount between 2 and 35 weight percent, and said nitroparaffin is employed in an amount between 0.25 and 6 weight percent, said amounts being based on the finished lubricant.

References Cited in the file of this patent

UNITED STATES PATENTS

2,257,945 Fraser ---------- Oct. 7, 1941

OTHER REFERENCES

Manufacturing and Application of Lubricating Greases, Boner, Reinhold Pub. Corp., N.Y. (1954), pp. 724, 726, 728–730, 740, 227, 228, 278 and 279.