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BARIUM SOAP GREASE COMPOSITIONS AND METHOD OF PREPARATION

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This invention relates to improvements in the manufacture and production of barium soap greases and particularly to an improved method in the preparation of normal barium soap greases and the grease compositions produced thereby.

It has been recognized for some time that barium soap greases possess certain inherent characteristics which are not attained by the conventional base grease. These characteristics include the combination of high melting point normally associated with the sodium soap greases and water-resistant properties characteristic of the calcium soap greases. The preparation of these greases has been extensively investigated and the investigators have concluded that the desirable characteristics of the barium soap greases are only attained by the basic and/or complex barium soap greases. The normal barium soap greases have been prepared only with great difficulty and require the presence of water for stabilization which substantially nullifies the improvement over the conventional base greases. Even the basic and complex barium soap greases cannot be prepared without resort to involved compounding procedures and special cooling and milling operations.

It has now been found possible to prepare stable barium soap greases, using a simplified manufacturing technique which eliminates the requirement of special gelling, static cooling, and cold working. Although this discovery is generally applicable to all barium soap greases, including the mixed base greases in which barium is one of the components, it is particularly applicable to the normal barium soap greases and affords a method of preparation which produces a stable normal barium soap grease possessing exceptional water and heat-resistant properties.

In accordance with the invention, it has been found that the use of a soap-forming hydroxy fatty acid as the acidic component of the barium soap not only permits the use of a simplified and more efficient method of manufacture but also provides improved texture stability in the resulting barium soap grease. In order to obtain these improvements the hydroxy fatty acid need not be the sole acid component of the soap but must be the predominant acid constituent. Thus, blends of conventional fats and fatty acids, with the hydroxy fatty acids, may be used provided the ratio of hydroxy fatty acid to fatty acid is greater than one.

The hydroxy fatty acids contemplated herein are those containing at least 12 carbon atoms and one or more hydroxyl groups separated from the

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carboxyl group by at least one carbon atom. From the standpoint of economy and availability, the most practical examples of these acids are 12-hydroxy stearic acid and the hydroxy acids produced by catalytic oxidation of hydrocarbon oils and waxes which have been extracted and fractionated to the desired molecular range. Although repeated reference has been made to the use of hydroxy fatty acids in the method of manufacture, the glycerides thereof may also be used and the only difference in composition of the resulting grease will be the possible inclusion of small amounts of glycerine.

In preparing the normal barium soap greases, the following general procedure has been found most effective. The desired amount of hydroxy fatty acid or the glycerides thereof are charged into a grease kettle with an equal weight of lubricating oil and a chemical equivalent amount of barium hydrate added. The temperature of the mass is maintained within the range of 170°–210° F. until saponification is complete, usually 4–5 hours. The saponification time may be shortened by the addition of water with the reactants. During the saponification and throughout the following steps of manufacture, continuous stirring is maintained.

It is furthermore preferable to utilize a steam-heated kettle since the temperatures used are well within the range of this type of equipment and a more accurate control thereover can be obtained. After saponification the reaction mass is heated to 260–300° F. for dehydration. It is necessary to avoid temperatures in excess of 300° F. since local overheating may cause the grease to melt and molten grease is conducive to the formation of lumps in the body of the grease. When the dehydration is substantially complete, the grease is gradually cooled by the progressive addition of the main body of lubricating oil. As previously mentioned, continuous stirring is maintained. The lubricating oil is added until the desired consistency is obtained and the grease is then cooled to a temperature below 200° F. and drawn as the finished product. The drawing temperature is necessarily below 200° F. and preferably around 130° F. because of the fact that high drawing temperatures promote setting up and false consistency.

The composition of the greases prepared in accordance with the invention naturaly depends upon the type of service and conditions of lubrication for which the grease is intended. Aside from the variations in composition obtained by the use of combinations of other metallic con-

stituents with barium and blends of other fats and fatty acids with the hydroxy fatty acids, an important factor to be considered is the oil component of the grease. In its broadest aspect, the oils contemplated by the invention may be defined as an oleaginous component of lubricating viscosity. This includes the conventional lubricating oils, synthetic oils, and the oleaginous organic compounds together with mixtures and/or blends thereof. The classification of oleaginous organic compounds embraces the liquid organic compounds which possess lubricating qualities and may be substituted for the conventional lubricating oils to impart improved torque-temperature characteristics to the grease. The composition of the compounds falling within this classification includes such divergent polar compounds as aliphatic dibasic diesters, aromatic, mono and dibasic esters, and aliphatic ethers. Specific examples thereof are the high molecular weight diesters of sebacic acid, benzoic and phthalic acid esters, and normal hexyl ether.

Outstanding results have been obtained by the use of extracts from the solvent refining of mineral lubricating oils as the oil component of barium soap greases designed for non-floating under-water lubrication. These extracts and particularly the extracts obtained from the sulfur dioxide or Edeleanu refining process provide grease compositions possessing certain characteristics which are not obtained by the use of other mineral oils of comparable density. For the preparation of the non-floating type of grease, it is desirable to select an Edeleanu extract with a 10.0° maximum A. P. I. gravity; that is, a density not less than 1.0. Also, for the best lubricating properties it is preferred to select extracts from naphthenic lubricating stocks of intermediate viscosity, which extract or blend thereof has a Saybolt Universal viscosity of approximately 300–340 seconds at 100° F.

For certain types of service where a high degree of oxidation stability is required an oxidation inhibitor may be incorporated in the grease. The aromatic amine type of inhibitor is considered to be the most efficacious. Of this class of inhibitors the polynuclear aromatic amines, such as tetramethyl diamino diphenyl methane, diphenyl amine, and phenyl alpha naphthylamine are preferred.

In order to illustrate the method of manufacture and grease compositions previously outlined, the following example is presented. 15 lbs. of hydrogenated castor oil and 15 lbs. of an Edeleanu extract, which is a blend of 44% extract from 80 Pale Stock and 56% extract from 300 Pale turbine stock, were charged to a steam heated kettle and stirred at a rate of 27 R. P. M. 8.1 lbs. of barium hydrate, having a barium carbonate content of 2.9% were then added to the mixture through a 12 mesh sieve followed by the addition of 10 lbs. of water. The ingredients were added in this order to minimize formation of barium carbonate from the hydroxide which is undesirable in the reaction since the barium carbonate reacts very slowly with fatty acids. The physical tests on the charged ingredients are as follows:

Hydrogenated castor oil

Saponification No.	179
Neutralization No.	2.4
Iodine No.	3
Titer °C.	76.2
Hydroxyl value	160

Edeleanu extract

Gravity °A. P. I.	9.6
Equivalent density at 60° F.	1.003
Flash C. O. C., °F.	340
Fire C. O. C., °F.	395
Viscosity S. U. at 100° F.	316
Pour A. S. T. M., °F.	−5
Carbon residue, per cent	0.09

The temperature was raised rapidly to 190° F. and maintained approximately constant for five hours to effect saponification. After saponification, heating was resumed and the product dehydrated over a five hour period, the temperature reaching 288° F. The dehydrated product was a smooth, dark brown, doughlike mass. Care was exercised to avoid heating above 300° F. because the product might melt and become lumpy. After dehydration, heating was discontinued but stirring was maintained as an additional 12 lbs. of Edeleanu extract were added to make a total of 27 lbs. in the batch and yield theoretically 37.8% normal barium soap. At this stage the product was a smooth, dark, glossy grease, the temperature being 240° F. A sample was removed for control penetration test and was found to have a non-worked penetration of 41 and a worked penetration of 155. Since the product was harder than desired, two more pounds of Edeleanu extract were added to give a theoretical soap content of 36.0%. Stirring was then continued until the temperature dropped to 130° F., when the grease was drawn from the kettle as a dull, grayish, adhesive, buttery grease.

Physical properties of this grease were as follows:

Barium soap, per cent (theoretical)	36.0
Water, per cent	None
Mineral oil, per cent	60.0
Dropping point, °F.	315
Oil separation at 150° F., per cent	None
Penetration, unworked	133
Penetration, worked	203
Density	1.07
Corrosion	None

Although hydrogenated castor oil was used as the source of the soap-forming hydroxy fatty acids in the above example, other hydroxy fatty acids or their glycerides may be used without departing from the fundamental steps of the process. Furthermore, blends of these hydroxy fatty acids with the conventional fats or fatty acids such as tallow and stearic acid may also be used. The oleaginous component has no effect upon the conditions of manufacture of the grease and therefore other oils such as the conventional lubricating stocks and the oleaginous organic compounds may be substituted for the Edeleanu extract of the example.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. The method of preparing an anhydrous normal barium soap grease having good water and heat resistant properties and stable against oleaginous liquid and soap separation, which comprises saponifying in the presence of oleaginous liquid of lubricating viscosity constituting a fractional part of the oleaginous liquid employed in

the final grease, an acidic soap-forming material containing a predominant proportion of soap-forming hydroxy fatty acids with barium hydrate at temperatures within the range of 170–200° F. to produce a saponified product of normal barium soap, heating the saponified product to effect substantially complete dehydration at temperatures above 260° F. but below 300° F., adding additional oleaginous liquid of lubricating viscosity in substantial amount to obtain a stiff adhesive grease consistency while cooling the dehydrated product to a temperature below 200° F., all of said operations being conducted with continuous stirring, and finally drawing the resulting anhydrous grease composition at a temperature below 200° F.

2. A method of preparing an anhydrous normal barium soap grease having good water and heat resistant properties and stable against oil and soap separation, which comprises saponifying in the presence of mineral lubricating oil constituting a fractional part of the lubricating oil employed in the final grease, an acidic soap-forming material containing a predominant proportion of hydrogenated castor oil with barium hydrate at temperatures within the range of 170–200° F. to produce a saponified product of normal barium soap, heating the saponified product to effect substantially complete dehydration at temperatures above 260° F. but below 300° F., then adding additional mineral lubricating oil to obtain a stiff

adhesive grease consistency while cooling the dehydrated product to a temperature below 200° F., all of said operations being conducted with continuous stirring and finally drawing the resulting anhydrous grease composition at a temperature below 200° F.

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