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GREASE COMPOSITION AND METHOD OF MANUFACTURE

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This invention relates to a method of manufacturing sodium myristate greases and particularly such greases as are applicable to lubrication under high temperature conditions, together with the grease compositions resulting therefrom.

This is a continuation-in-part of our copending application Serial No. 590,314, filed April 25, 1945 (now abandoned).

It has been found that the sodium myristate greases possess certain characteristics which render them of particular value for high temperature lubrication of anti-friction bearings. Besides possessing a dropping point in excess of 400° F., they are also resistant to the leaching effect of water and provide satisfactory lubrication with negligible texture or phase change even at temperatures above 300° F. These characteristics are particularly unusual for sodium base greases of the same consistency and yield and accordingly distinguish myristic acid from the conventional fatty acids used in grease manufacture.

In general, the preparation of these greases involves saponification of the myristic acid with a sodium hydroxide solution in the presence of small amounts of mineral oil and water, dehydrating the resulting soap base and adding sufficient mineral oil to obtain the desired consistency. Although the invention is broadly applicable to a wide range of mineral oils as the oil component of the subject grease, it has been found particularly desirable to use a paraffinic oil and, preferably, a blend of a distillate and residual oil.

For the purpose of the invention mixtures of low molecular weight fatty acids containing at least 70% myristic acid may be used in the preparation of the subject greases. It is preferred, however, that myristic acid of around 90% purity be used to insure the desirable characteristics of the finished grease composition. Although the invention is directed to the use of the acid itself, it is to be understood that glycerides thereof containing at least 70% myristic acid in the fatty acid component may also be used.

The grease kettle manufacture of the sodium myristate greases may be illustrated by the following general procedure: A kettle is charged with a mixture of the desired amount of myristic acid and equal amounts of mineral oil and water. The selection of the mineral oil to be used will depend upon the type of lubricating service for which the grease is intended. It is preferable, however, that the oil be of a paraffin base stock. If a blend of a distillate and residual oil is used, it is desirable that the lower viscosity distillate

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oil be charged initially to facilitate the saponification and the higher viscosity residual oil added after dehydration as such or blended with the distillate oil. For convenience of operation, the amount of mineral oil and, accordingly, the amount of water initially charged is generally less than the amount of fatty acid charged and is usually around two-thirds of the weight of the fatty acid. The mixture is heated to around 170–190° F., with continued stirring at an intermediate rate of speed of around 26 R. P. M., and the fatty acid saponified by the addition of a sodium hydroxide solution added as rapidly as possible without boiling the mixture. In the preparation of the high temperature water-resistant greases of the invention it is desirable to add an excess of sodium hydroxide over that required to saponify the fatty acid so that the final product contains an amount of free alkali in the range of 0.05–1.0%.

After saponification is complete the stirring speed is reduced to around 18 R. P. M. and the temperature raised to 295–310° F. to effect dehydration. The soap base is maintained at 295–310° F. for at least two hours before oil addition is started. The addition of the mineral oil to the soap base is then begun and the temperature allowed gradually to decrease. The reduction of the temperature is controlled during the oil addition so that a total amount of mineral oil equal to at least twice the weight of the fatty acid is charged before the temperature is allowed to drop below 260° F. The amount of oil added above 260° F. will necessarily depend upon the amount initially charged with the fatty acid. If, for example, the mineral oil initially charged were equal to two-thirds the weight of the fatty acid then the amount of oil incorporated above 260° F. must be at least four-thirds the weight of the fatty acids. After this amount of mineral oil has been incorporated above 260° F., the temperature may be reduced and sufficient oil then added to bring the grease composition to the desired consistency. The resulting product is then drawn from the kettle as the finished sodium myristate grease.

In preparing the sodium myristate grease particular care must be taken to maintain the soap base at temperatures above 295° F. for a period of time to insure completion of the dehydration reaction. It has been found that the soap base should be maintained at dehydration temperatures of 295–320° F. for at least two hours, preferably about 2½ hours, before oil addition is begun.

Another factor in the preparation of these greases is the amount of mineral oil incorporated into the soap base at temperatures above 260° F. In order to obtain satisfactory yields the amount of mineral oil incorporated during the oil addition stage at temperatures above 260° F. should be sufficient to provide an oil content in the soap-oil mixture at this point of at least twice the weight of the fatty acid initially charged. Thus, the sum total of the oil charged initially and that incorporated in the oil at the oil addition stage, at temperatures above 260° F., should at least be twice the weight of the myristic acid content.

Example

Fifteen pounds of myristic acid (saponification No. 245, neutralization No. 242, titer °C. 45.7), ten pounds of a solvent-refined, dewaxed paraffin base lubricating oil of an SAE 20 grade, and 9.9 pounds of water were charged to a steam-jacketed kettle. Heating was begun with stirring at 18 R. P. M. At temperatures between 170 and 198° F. 6.57 pounds of a 49.9% solution of sodium hydroxide were added. After the addition of the sodium hydroxide solution the temperature was raised and the saponification product dehydrated. Toward the end of the dehydration stage the soap base was held for two hours at temperatures of 297–303° F. After the completion of the dehydration reaction the temperature was gradually lowered and addition of the oil started. The temperature was maintained above 260° F. with stirring until 20.0 pounds of oil had been added. Thereafter the temperature was allowed to drop while the oil addition was continued. After an additional 16.5 pounds of oil had been incorporated a sample was removed for control penetration (unworked =160 at 78° F., worked =183 at 78° F.). Oil addition was continued with stirring until eight pounds of oil were further incorporated, resulting in 54.5 pounds of oil contained in the batch. Control penetration at this point showed a worked penetration of =220 at 78° F. and unworked =213 at 77° F. The grease was then drawn and screened through a Viking pump and a double 60-mesh screen. The resulting sodium myristate grease was of a very light color possessing a smooth almost buttery texture.

An analysis of the subject grease is as follows:

Sodium soap, per cent	22.7
Free alkali (NaOH), per cent [1]	0.86
Free fatty acid (Oleic), per cent [1]	None
Free neutral fat, per cent	0.38
Water, per cent	0.4
Penetration at 77° F. (ASTM)	
Unworked	205
Worked	230
Dropping point, °F	417
Dynamic water resistance, per cent [2]	15

[1] "The Institute Spokesman," National Lubricating Grease Institute, January 1944. "Test Methods for Determining Free Acid and Free Alkali in Greases."
[2] AN–G–5a—Dynamic Water Resistance Test.

While the grease of the foregoing example has a sodium soap content of 22.7%, it will be understood that the soap content for greases of various grades for ball and roller bearing lubrication will generally fall within a range in excess of about 15% but less than about 25% by weight on the basis of the final product. Whereas sodium base greases of soft consistency adapted for antifriction bearing lubrication generally have a fibrous texture and are known as "fiber greases," the sodium myristate grease of the present invention is unique in being a smooth soft grease of a buttery non-fibrous texture. In order to obtain this type of grease in satisfactory yield, it has been found essential to follow the foregoing method of preparation involving essentially the lower temperature saponification in the presence of small amounts of the mineral lubricating oil and water, the high temperature dehydration at 295–320° F. before further oil addition to reduce the water content to less than about 0.5% by weight on the basis of the final product, and finally the two-stage oil addition with temperature control as specified. In the saponification step, the amount of oil initially added should be less than the weight of fatty acids initially charged but should be at least about one-half the weight of the fatty acids. Likewise, the amount of water initially added should be roughly equivalent to the amount of lubricating oil specified above. The outstanding and unusual properties of this particular sodium base ball and roller bearing grease are the high dropping point, the exceptional resistance to water washing or leaching from the bearing by a stream of water as shown by the above listed Dynamic water resistance test, and the ability to provide satisfactory lubrication of anti-friction bearings at temperatures above 300° F. without undergoing phase change, such as oil or soap separation, and without change in texture or consistency to a substantial extent, such as represented by a large increase or decrease in penetration.

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 in the manufacture of a smooth soft sodium myristate ball and roller bearing grease of buttery non-fibrous texture, which comprises mixing a quantity of low molecular weight fatty acids containing at least 70% by weight of myristic acid with a small amount of distillate mineral lubricating oil less than the quantity of fatty acids charged but equivalent to at least about one-half of the weight of the fatty acids, and also with a small amount of water roughly equivalent to the amount of lubricating oil, specified above, agitating the mixture at a mildly elevated temperature of about 170–190° F. with the addition of an excess of an aqueous sodium hydroxide solution to effect saponification of the fatty acids and provide in the final product a free alkali content in the range of 0.05–1.0% by weight, then heating the resulting saponified mass with continued agitation at 295–320° F. for at least two hours before oil addition is started to effect dehydration to a water content less than 0.5% by weight on the basis of the final product, then adding additional mineral lubricating oil in two stages with control of temperature of the mass and continued agitation, the first stage of oil addition being while the mass gradually cools from the said dehydration temperature but is maintained above 260° F. and wherein the amount of oil added is sufficient to bring the total lubricating oil content of the mass to at least twice the weight of the fatty acids initially charged, and the second stage of oil addition being at temperatures below 260° F. to bring the mass to the consistency desired, and then drawing as a

final product a smooth soft grease of a buttery non-fibrous texture having a soap content in excess of 15% but less than 25% by weight on the basis of the final product, said grease having a dropping point in excess of 400° F., and possessing the property of satisfactorily lubricating ball and roller bearings at temperatures above 300° F.

2. The method according to claim 1, wherein the fatty acids initially charged contain at least 90% by weight of myristic acid, and the mineral lubricating oil used is a paraffin base mineral lubricating oil.

3. The method according to claim 2, wherein the amounts of paraffin base mineral lubricating oil and water initially charged are each roughly about two-thirds of the weight of the fatty acids in the charge, and the amount of

paraffin base mineral lubricating oil added in the first stage after dehydration and at a temperature above 260° F. is at least four-thirds of the weight of the fatty acids in the charge.

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