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METHOD OF MANUFACTURING
LUBRICATING GREASE

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This invention relates to a method of manufacturing aluminum soap greases and particularly to a method by which aluminum stearate greases of superior quality may be produced effectively and economically.

It is well known that certain aluminum soap greases, for example greases prepared from mineral oil thickened with aluminum stearate, have desirable characteristics which make them useful for many purposes. Until recent years such greases were prepared by heating the aluminum soap in the mineral oil to a temperature of the order of 300° F., the mixture being agitated during heating. Thereafter in order to obtain a good grease structure it was necessary to pour the grease into pans in shallow layers to permit rapid chilling because if the grease is not chilled fairly rapidly it is not of good consistency. For example, if an aluminum stearate grease is poured directly into 400 pound drums it has been found that that portion which is next to the outside of the drum and cools quickly is a good consistency, but the center portion is semifluid and grainy in appearance. This condition is due to the difference in the rate of cooling. It requires about a week for the grease in the center of a 400 pound drum to cool from 300° F. down to ordinary room temperatures.

In recent years, methods have been developed for manufacturing aluminum soap greases in rapid continuous cooling mechanisms where the desired structure may be obtained by feeding the heated grease through a jacketed mixer chilled externally with cold water. This process has been fairly satisfactory where continuous process equipment is available, but there are many grease plants which are equipped with and still use large grease cooking kettles operated by the batch process. The present invention has as an object the use of an existing batch equipment for producing aluminum soap greases of good quality.

Aluminum soap greases, and particularly aluminum stearate greases, have two peculiar properties which must be taken into consideration in the preparation of these compositions. When aluminum stearate is mixed in mineral oil in grease forming proportions and the mixture is heated to a temperature of the general order of 300° F., for example 275° to 350° F., it forms a rubbery plastic mass. When the resulting grease is cooled, it reaches a temperature or temperature range, usually around 100° to 130° F., where the rubbery mass changes in structure to a gel or regular grease structure. This temperature is known as the transition temperature. The tran-

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sition temperature varies somewhat with the type of oil and the amount of soap used, but in general it ranges between the upper limit of about 160° F. and the lower limit of 90° F., most greases falling within the narrower transition range of 100° to 130° F., mentioned above.

On further cooling through and beyond the transition range, if this is accomplished with prolonged agitation, the gel tends to be broken down to a stringy semiliquid product having grainy characteristics. Such a product is not a satisfactory grease. On the other hand, if agitation is stopped just a few degrees below the transition temperature, a firm gel of desirable grease consistency and texture is obtained.

The time required for the change from rubbery plastic to gel varies considerably and may be only a few minutes or as much as ten hours or more. This time depends, among other things, upon the batch size and the composition of the grease. It appears that the change from a rubbery structure to a gel structure is a process related to or resembling crystallization. The growth or formation of the grease structure or micelle structure is materially affected by the rate of temperature change as well as by the mechanical working.

In order to obtain the maximum consistency of grease, that is the grease having the hardest consistency for given soap content, it has been found that two factors are important, especially in aluminum soap grease manufacture and more particularly in the manufacture of aluminum stearate grease. In the first place the gelation temperature, which must be as low as and preferably somewhat below the transition temperature, is important. In the second place the time required for bringing the grease through the transition range and down to the temperature selected has an important effect upon the eventual grease structure. This is particularly true where, as is usually the case, cooling is accompanied by mechanical working. The present invention has to do with the modification and control of this second factor.

In order first to illustrate the effect of these two variables on grease consistency, two experiments were conducted.

Example 1

A grease composition was prepared consisting of 9% by weight of aluminum stearate, 89.5% mineral lubricating oil, of 45 viscosity index and 185 S. S. U. viscosity at 210° F., and 1.5% of diamylphenol. The diamylphenol is a crystallization modifier which is useful to improve the texture of aluminum soap greases and prevent me-

chanical breakdown during cooling, as set forth in the patent to Sproule and Zimmer, No. 2,394,567. Obviously, other phenolic materials may be substituted as described in said patent.

The grease of the foregoing example had a transition temperature range of between about 110° and about 120° F. The effect on consistency of the grease of various gelation or final chilling temperatures is shown in the following data:

Gelation temperature, °F.	75	90	100	110
Worked penetration	299	314	348	377

The results shown in the above data indicate that the worked penetration of the grease decreases, that is the grease becomes harder as the difference between gelation temperature and transition temperature is increased. Upon reaching the transition temperature in each test, the grease was allowed to stand without further agitation.

Example 2

A grease of precisely the same ingredients as in Example 1 was prepared to illustrate the second factor mentioned above, that is, the effect of time of cooling through the transition to a given gelation or setting temperature. The grease of Example 2 was cooled to a final gelation temperature of 95° F. One portion was rapidly cooled by an external jacket of cold water while the other portion was allowed to cool slowly in the air. The rapidly cooled portion, which was brought down from its transition temperature of about 120° F. to 95° F. in one hour had a worked penetration number of 330 as compared with a worked penetration number of 380 for the other portion which required three hours to cool. These data indicate that a shortened time for cooling the grease from transition to gelation temperature improves the hardness or consistency to a marked degree.

The data given above in connection with Examples 1 and 2 show first that a low gelation temperature is desirable to obtain the maximum consistency or hardness and also that the time of cooling must be as short as possible. Mechanical working is continued throughout the cooling process. With rapid cooling mixing may be continued without breaking down the grease or causing oil separation whereas with slow cooling mechanical working in the form of prolonged agitation of the paddles in a standard grease mixing kettle tends to break down the gel structure into a soft oily grease.

In the batch or kettle grease making process, the plant water supply usually available for cooling purposes, particularly in the summer months, does not provide adequate heat transfer and will not cool the greases through their transition to gelation temperature in a short enough time. Therefore, particularly where large batches of grease are being processed, an internal refrigerant must be added to speed up the cooling process. Such a refrigerant is preferably an inert solid refrigerant capable of absorbing large quantities of heat as it changes from the solid state. It was found that by adding suitable quantities of finely divided solid carbon dioxide, for example 0.5 to 3%, based on the weight of the grease batch, the grease cooling period could be shortened sufficiently to obtain the desired final grease structure, such rapid cooling permitting the use of the conventional batch type grease kettle. By this process the use of such special apparatus as the close clearance film chiller and the like is not necessary. Hence by this process conventional equipment can be used efficiently for preparing superior types

of aluminum soap greases. It should be noted, however, that irrespective of the process employed the aluminum soap greases, and particularly the aluminum stearate greases, should not be agitated or worked mechanically for long periods during the transition in structure. For this reason rapid cooling appears to be essential for efficient production since fairly continuous mechanical working is usually necessary to obtain a uniform product. Mechanical working can be discontinued for part of the cooling process, however.

Example 3

Another composition was prepared by mixing together 9.0% by weight of aluminum stearate, 0.75% diamylphenol, and 90.25% of mineral oil of 85 S. S. U. viscosity at 210° F. with viscosity index of 45. The grease so prepared had a transition temperature of between 110° and 120° F. A 5,000 pound batch was produced in a kettle having a capacity of 6,000 pounds. In this example, the ingredients were mixed together and heated to about 300° F. Thereafter they were cooled down to a temperature of about 105° F. by passing service water of a temperature of about 70° F. through the jacket of the mixer. The time of cooling from 120° F. to 105° F. was 2 hours. The grease was then allowed to stand without further agitation for transition from its rubbery structure to the final gel structure. Thereafter the grease was worked in a mixer for a few minutes to render it homogeneous. It was found to have a worked penetration number of 353 at 77° F.

Example 4

Another batch identical with that of Example 3 was prepared by heating the ingredients to 300° F., cooling them to 120° F. with service water of about 70° F. temperature and thereafter cooling further by mixing therein solid carbon dioxide. At the temperature of 120° F. 75 pounds of powdered solid CO₂ was slowly added, resulting in lowering the temperature to 102° F. in three-quarters of an hour. The grease was then allowed to stand without mixing for transition from rubbery to gel type structure and thereafter the contents of the kettle were mixed for a few minutes to render them homogeneous. The worked penetration of this grease was 328 as compared with 353 for a grease of the same ingredients cooled more slowly by the use of water. A comparison between Examples 3 and 4 thus indicates that a grease identical in composition but 25 points harder in worked penetration was obtained by the more rapid cooling resulting from the use of solid carbon dioxide.

The quantities of carbon dioxide which may be required will vary somewhat with the cooling water and the type of facilities available. In general, amounts of 0.5 to 3% based on the weight of the grease batch are suggested. In order to cool a 5,000 pound batch of grease from a temperature of 120 to 100° F. it is necessary to remove about 47,000 B. t. u. The addition of 75 pounds of solid CO₂ as in Example 4 above is sufficient to absorb about 21,600 B. t. u. In this example the water in the jacket of the mixer absorbed the remainder. If desired, greater quantities of CO₂ may be used or if the water available for chilling is quite cold it may be possible to use relatively small quantities of CO₂ to obtain the desired results.

It is preferred to use carbon dioxide snow or an equivalent finely divided inert solid refrigerant

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and preferably the particle sizes thereof should not exceed about $\frac{1}{4}$ inch. A finer subdivision of the refrigerant is desirable. The CO_2 should be added gradually and mixed thoroughly in the grease to obtain the desired uniformity in texture. As indicated above, the quantity employed may be varied depending upon the type of grease, the difference between transition temperature and gelation temperature, the size of the batch, temperature of cooling water available, and the like. In any case, cooling should be sufficiently rapid to prevent syneresis and graininess which result from excessively slow cooling and also to prevent the need for long continued mixing which tends to break down the grease structure.

The process described above has particular application to aluminum stearate greases although greases containing mixed aluminum soaps formed from mixtures of fats and fatty acids may be similarly prepared. However, the invention has some application also to the production of soda soap greases, such as those used for high temperature lubrication. In the manufacture of soda greases the process may involve heating to a high temperature, for example of the order of 500 to 525° F. The grease thereafter may be cooled in the ordinary manner to a temperature of about 190° F. for example, with agitation. Such cooling ordinarily requires about 24 hours for a 5,000 pound batch. By the use of internal refrigeration, employing solid carbon dioxide as described above, the cooling time may be reduced considerably. Among other things this will reduce or eliminate oxidation which is likely to occur when a grease is maintained at a high temperature over a long period of time.

It will be understood that the transition temperature, the aluminum stearate content, the type of mineral oil employed, and the additives and modifiers contained in the grease, are all variable factors which may be modified to suit the type and purpose of lubricant to be manufactured. Ordinarily the aluminum stearate will be used in quantities of 5 to 15% by weight, based on the finished grease composition. The lubricating oil will usually have a viscosity of not less than 50 S. S. U. at 100° F. nor more than 1000 S. S. U. at 210° F.

In general, the transition point for aluminum stearate greases lies between 90° and 160° F. and it is desirable to reduce the temperature from this transition temperature to a gelation temperature which is preferably 10° to 40° or more lower than the transition temperature in a relatively short period of time. For the sake of economy, external or jacketed refrigeration will be used as far as practicable. Where more rapid cooling is requisite, the use of internal refrigerant, preferably accompanied by water jacket cooling, facilitates manufacture in conventional grease kettle equipment.

It will be understood that the temperature to which the greases are raised in their preparation may be varied considerably although as a rule aluminum soap greases, specifically the aluminum stearate greases, are heated to a temperature between about 275° and 350° F. As indicated

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above, high temperature soda soap greases to which the invention also has some application, are prepared at substantially higher temperatures.

The use of phenolic compositions as crystallization modifiers has been suggested in the foregoing examples and as noted above is covered in detail in the Sproule and Zimmer patent, No. 2,394,567. Such modifiers may be various types of phenolic compounds and they may be added in various proportions as described in said patent. Ordinarily the quantity of modifier used will be between about 0.5 and 2.5%, based on the finished lubricant. In addition, other conventional additives may be employed, as is well known to those skilled in the art. Thus, antioxidants, tackiness agents, viscosity index improvers, extreme pressure additives, and the like may be employed in usual proportions.

What is claimed is:

1. A method of preparing aluminum soap base grease which has a tendency to break down mechanically upon prolonged mechanical working during its transition from rubbery structure to gel structure, comprising heating aluminum soap with mineral lubricating oil at a grease-forming temperature, thereafter cooling said grease rapidly to a temperature approximating its transition temperature, accompanied by mechanical working, said cooling being accomplished by using an external cooling medium and thereafter mixing solid carbon dioxide into the grease with continued mechanical working to lower the grease temperature rapidly below said transition temperature at the latter part of the cooling operation.

2. A method of preparing a lubricating grease composition which comprises combining about 5 to 15 parts by weight of aluminum stearate, 0.5 to 2.5 parts of a phenol modifier and 94.5 to 82.5 parts of mineral lubricating oil, heating said composition to a temperature range between about 275° and 350° F. to form a grease, cooling said grease rapidly by means of external water accompanied by mechanical working to a temperature approaching its transition temperature, and thereafter mixing 0.5 to 3 parts by weight of solid carbon dioxide into said grease to reduce its temperature rapidly towards the end of the cooling operation.

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