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MANUFACTURE OF MILL GREASES

William E. Bradley, Los Angeles, Calif., assignor to Union Oil Company of California, Los Angeles, Calif., a corporation of California

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6 Claims. (Cl. 252—38)

The present invention relates to the preparation of lubricating compositions, and particularly pertains to the manufacture of a lubricant for use on bearings operating at high temperatures and pressures.

Many types of heavy machinery, as for example, those used in laundries, paper mills, cement mills, etc., are equipped with open bearings which operate under severe conditions of high temperature and/or pressure, as well as low journal speeds. Thus, the operating temperatures in such bearings range from 100° F. to 325° F. and even higher. In usual practice the bearings of such heavy machinery are lubricated by means of greases commonly known as mill greases which are usually formed into bricks. These bricked mill greases obviously must withstand the above excessive operating conditions without the production of hard films on the bearing surfaces and without melting or other decomposition. The water content of the final brick grease is also highly important. Thus, greases of this type may be broadly classified, depending on their texture, into three types: gel type, sponge type and the gum type. Although the question is a moot one, it is believed that the gel type brick grease is superior and is best suited for the economic and proper lubrication of bearings of the above-mentioned type of machinery. All of the greases, irrespective of type, have a fairly well defined upper water content limit above which the grease becomes mushy and soft and disintegrates rapidly at the operating temperatures and pressures. As to the gel type grease, it also has a rather definite minimum, so that if the water content of the grease drops below said minimum, the brick grease is granular and unsatisfactory. The range of water content depends on the particular type of grease. Thus, the gel type greases require and permit greater percentages of water as compared to the permissible water content of the sponge type of grease. However, as stated, this range is rather well defined for each type of grease. Although not limited thereby, the present invention will be described with particular reference to the manufacture of the gel type mill grease.

In the method of production of the brick or mill grease it is now customary to employ the open kettle method. Briefly, this method consists in charging the soap stock such as tallow, fat, fatty oil, rosin, rosin oil, degras, or similar substances, or certain of them, into an open kettle equipped with an agitating device and heated by any known means such as open fire, electric heating elements, steam jacket or steam coils. Simultaneously with the soap stock a certain portion of the mineral oil together with the necessary quantity of caustic soda in the form of an aqueous solution thereof are injected into the kettle. The saponification of the soap stock by the caustic soda occurs by reason of the application of heat to this mixture. After it has been determined that all of the soap stock has been thus saponified the remaining portion of the mineral oil is added to the mixture in the kettle, and the whole is further agitated and heated. This heating is primarily for the purpose of dehydrating the grease mixture, since as stated above the water content in the final grease must be comparatively small and within certain narrow limits. The dehydration being completed the produced grease is drawn into open pans of suitable size and allowed to cool and harden to form the bricks employed on mill bearings. The above described process of manufacturing brick grease is, however, attended with certain defects and difficulties. These primarily relate to the control of the operations of said process. Thus, the heating of the mixture in open kettles makes very difficult the dehydration of the grease to the proper degree so as to maintain the desired water content in the finished brick grease. One of the causes for the difficulty in controlling the dehydration is due to the impossibility in rapidly determining the water content of the grease while the latter is being heated in the kettle. In view of this the operators are usually forced to rely on their personal judgment and experience in estimating the degree of dehydration of the melted grease. Obviously, such procedure results in wide variations in the quality of the final product. Another defect of the open kettle method is caused by the inability to withdraw large batches of the melted grease from the kettle sufficiently rapidly to prevent marked differences in the water content between the first and last portions of the grease thus withdrawn.

Another objection resides in the excessive amount of time necessary for the complete saponification of the saponifiable ingredients. A still further defect is due to the fact that the dehydration of the grease is not uniform in the various parts of the open kettle in which the grease is made. Thus, the grease closest to the sides of the kettle is found to be excessively dehydrated although the water content of an average sample taken from such kettle may be

well within the allowable water content. The objection to this unequal dehydration resides in the fact that the grease thus excessively dehydrated does not re-dissolve in the wetter grease, so that, although the average water content of the grease is correct, one really obtains a mixture of greases, one of which is too dry and therefore granular, and another too wet and mushy.

The main object of the present invention is to provide a mill or brick grease which is able to withstand the severe operating conditions of high temperature and pressure. A further object of the present invention is to provide an improved method which will obviate the above enumerated defects and which will produce a brick grease of superior wearing and lubricating qualities. A still further object of the present invention is to provide a process for making brick or mill greases which can be easily controlled and which will insure uniformity in the resulting product.

It has now been discovered that the above and other objects may be attained by providing an improved process wherein the sequence of steps and operating conditions result in a grease uniformly superior in appearance, and of unusually smooth consistency, structure and texture, and which possesses greater resistance to the effect of heat. One of the features of the invention is the use of closed vessels for the saponification and heat treatment of the grease mixture. Another feature of the invention resides in the introduction of all the ingredients substantially together into the aforementioned still followed by the heating and agitation of the mixture thus produced under pressure. The saponification and heating of the grease mixture under pressure in a closed still eliminates the above described disadvantages since it prevents any dehydration of the melted grease prior to the time of drawing, thus permiting accurate control of the water content. In fact, operation according to the process to be described more fully hereinbelow permits accurate control of the water content in the final brick grease. This is due to the fact that the operation of the process of manufacturing brick grease in a closed still maintained under pressure will prevent uncontrolled vaporization of water and therefore excesive or insufficient dehydration of the grease being manufactured. In fact, the water content of the final product in such case will be a function of the degree of heating to which the grease is subjected in the still. Also, the maintenance of a pressure in the still during the manufacturing of the brick grease aids in producing a uniform grade of final product since it is possible to draw the grease rapidly from the pressure still into the cooling pans. Obviously, if desired, the grease may be drawn slowly from the pressure still. In fact, the use of the autoclave permits the grease to remain as an apparently true solution until it is drawn into the pans. This results in a grease having a smooth texture. Furthermore, the use of pressure during the heating of the mixture, as well as the ability to mix all of the ingredients at one time, considerably shortens the period of time necessary for the proper heating and preparation of the grease. In other words, the present process eliminates the present necessity of consecutive introduction of the various ingredients into the cooking still. The desired reduction in water content is obtained by "flashing" (evaporation of water consequent upon the release of pressure) when drawing the grease.

The invention includes reduction of water content to the desired percentage upon withdrawal of the grease from a pressure cooking vessel.

The invention may therefore be broadly stated as residing in a process of manufacturing brick mill grease wherein the ingredients forming the various component parts of the final product are introduced into a closed still, preferably heated under their own vapor pressure to the desired temperature, and then drawn into cooling pans. The invention further resides in a process wherein all or substantially all of the ingredients are placed in an autoclave which is thereafter sealed. The ingredients in the autoclave are then agitated and simultaneously heated to an optimum temperature and for a period of time sufficient to cause complete saponification of the saponifiable ingredients. The melted grease thus produced may then be drawn into pans or trays in which it cools and becomes hard.

The invention further resides in such processes whereby the dehydration of the grease is controlled within definite limits, preferably between about 0.1% and about 1% or at most 2% in the final product. The invention also extends to such products.

More specifically stated, the invention resides in a process of manufacturing brick grease comprising introducing the saponifiable materials, mineral oil and alkali into a still or autoclave, heating and agitating the mixture under pressure until the saponification of the saponifiable materials has occurred and until the grease mixture has attained the desired temperature, and withdrawing said melted grease from the pressure still or autoclave to allow the grease to cool and become hard. The invention still further resides in the rapid withdrawal or "flashing" of the melted grease from the pressure still. This permits controlled dehydration as well as uniformity between the various batches or trayloads of grease withdrawn from the still.

As previously stated, the ingredients which go into the making of a brick grease comprise one or more saponifiable materials, a mineral oil and an alkali. Also, a small and limited percentage of water must usually be present in the final grease. As the saponifiable material or soap stock, it is possible to use fat, fatty acids, tallow, lard oil, rosin, rosin oil, degras and/or cottonseed oil. In fact, the texture or consistency of the final grease depends to a considerable degree on the combination of saponifiable materials used in the making of the grease. Thus, the use of tallow in combination with rosin oil and degras tends to hold excessive amounts of water, resulting in a grease of an inferior quality. For this reason it has ben found desirable to add certain quantities of cotton seed oil, which permits easier dehydration. As to the degras, its use is advantageous because of the presence therein of high molecular weight alcohols which promote solubility between the soap and the mineral oil. The presence of rosin or rosin oil in the grease breaks the tendency toward formation of fiber thereby aiding in the production of a gel type grease. Although the cause is not known at the present time, it is presumed that the presence of terpene-like materials in the rosin and rosin oil assists in the above breaking of the fiber. The fiber breaking characteristics are also found in the degras due to the presence of the higher molecular weight alcohols.

The mineral oil used in the preparation of the grease may be either paraffinic in nature or comprise a western or a naphthenic base stock. Obviously, the viscosity, boiling point range and/or color of the mineral oil must be such as to produce the desired type of brick grease. It has been found that the well known paraffinic lubricating oils, either of the Pennsylvania type, or those produced by the solvent extraction of mixed base or similar oils, as for example, an S. A. E. 20 oil, produces a brick grease which may be satisfactorily employed for the lubrication of heavy machinery in laundry and cement mills wherein the operating temperatures are as high as 320–325° F. The western or naphthenic base crude oils do not ordinarily permit the greases containing the same to be used at the above high temperatures. Also, brick greases made with naphthenic or western oils ordinarily form a hard film on the surface exposed to the journal surface even when the operating temperatures are below the melting points of the grease. On the other hand the naphthenic base oils are more miscible with the soap and therefore do not produce a granular structure even when there is little or no glycerin in such grease. In fact, the presence of naphthene base oils allows the production of gel type greases of good texture even though substances such as cotton-seed oil or lime are not present. Caustic soda is the usual alkali employed for the saponification of the saponifiable materials going into the making of the brick grease. However, for particular uses a part of the caustic soda may be replaced by other alkalies as caustic potash or hydrated lime. Thus, the presence of lime tends to decrease somewhat the granular texture of a given brick grease. However, when lime is not present this granulation of the grease may be somewhat limited by increasing the ratios of cottonseed oil, or other liquid fat, to the other saponifiable materials of the class of tallow and other solid fats.

In carrying out the process all of the ingredients are first introduced into a still provided with an agitator and with any convenient heating means. After sealing of the still agitation is initiated and the temperature is gradually raised until saponification is completed. Due to the presence of water in the mixture, and since the temperature in the still is considerably above the boiling point of this water, the pressure in the still rises. The above mentioned water may be that introduced into the still or produced by hydrolysis during saponification. The temperature to which the mixture in the still or autoclave is heated depends on the composition of the ingredients and to a great degree on the amount of water added, as well as on the desired water content in the final product. Thus, when it is desired to produce a gel type brick grease, and when the aqueous caustic solution contains about 60% water (as in the examples given hereinbelow), the temperature is usually raised to and maintained at about 300° F. to 350° F. In some instances, the temperature may be raised as high as 500° F., and even above, particularly when greases of the sponge or gum types are to be produced. When a gel type grease is being made according to the present invention, the pressure generated in the still is between about 40 and 80 pounds per square inch.

After the desired degree of cooking has been attained, the melted grease is ejected (without any previous pressure release) into open pans or trays in which it is allowed to cool and become hard. During the discharge of the grease into the open pans or trays there occurs an evaporation of the water, this evaporation being caused by the reduction in pressure to atmospheric. Thus, it is possible to control the quantity of water remaining in the final product by accurately controlling the amount of water introduced into the kettle and regulating the temperature and therefore the pressure in the pressure still during the cooking operation, the excess water "flashing" out upon discharge.

As a specific example for carrying out this process, the following ingredients were introduced into a pressure kettle or still:

	Pounds
Hydrogenated foots from cottonseed oil fatty acids	80
Rosin oil	208
Degras	61
40% aqueous caustic soda solution	57.5
Mineral oil	540

In the specific example, a lubricating oil produced from a naphthene base or Western crude oil, and having a viscosity of 350 second Saybolt Universal at 100° F., a specific gravity of 0.931, and a viscosity-gravity constant of 0.886, was used. After sealing the kettle, the contents were agitated and heated for about three hours by steam applied to the kettle jacket at a pressure of 120 pounds per square inch, the temperature in the kettle being thus raised to about 320° F. while the pressure was about 40 pounds per square inch. At the end of the above period, the saponification was found to be complete, and the melted grease was drawn into open trays or pans for purposes of cooling and hardening. In the specific example, the water content of the grease thus produced was 1.0%, however this content could be varied so as to be as high as 2.0% by regulating the temperature and pressure at which the melted grease is drawn, and by controlling the amount or proportion of water introduced into the kettle or still. The grease thus produced is of the gel-type and has a smooth, satiny texture. It is highly suitable for the lubrication of bearings operating at between 50° and 210° F.

Although it was stated that all of the ingredients used in the making of the grease according to the present process are introduced simultaneously into the still or pressure kettle, it has been found advantageous to deviate slightly in this procedure when lime is used as one of the saponifying agents. This is due to the fact that caustic soda is a stronger saponifying agent. Therefore, when both lime and caustic soda are to be employed, the introduction of the caustic soda into the kettle is delayed but only for a period of time sufficient to permit the reaction between the saponifiable materials and the lime employed. The following is an example showing the procedure employed in the manufacture of a lime containing grease. In this case, the following ingredients in the specified proportions or quantities were used in the making of grease:

	Pounds
Prime tallow	35
Rosin oil	150
Degras	44
Cottonseed oil	23
Hydrated lime	1.8
Caustic soda (dry)	22
Mineral oil	490
Water	33

It is to be noted at the outset that the quantity

of alkali indicated will vary with the saponification numbers of the saponifiable materials. This is because the quantity of alkali is to be controlled so that it is substantially just sufficient to saponify all of the fat and rosin oil. In the instant case, the quantities of alkali given hereinabove were calculated on the basis of the following saponification numbers:

Saponifiable Material:	Saponification Number
Prime tallow	197
Cotton seed oil	195
Rosin oil	99
Degras	105

In operation, all of the tallow, rosin oil, degras and cottonseed oil were introduced into the pressure still together with a slurry containing the hydrated lime and 50 pounds of the mineral oil (which in the instant case was a Pennsylvania lubricating oil having a viscosity-gravity constant of 0.816 and a S. A. E. grade of 20). The mixture was heated for about 15 minutes to approximately 150° F. which is sufficient to cause the lime to react and form the calcium rosin oil soap. Thereafter, the balance of the above lubricating oil, together with the caustic soda dissolved in water to form a 40% aqueous solution thereof, were conveyed into the kettle, which was then sealed and heated by means of steam maintained in the kettle jacket at a pressure of about 100 pounds to 120 pounds per square inch. The heating and agitating of the mixture in this pressure still or kettle was continued for a period of about 3½ hours at approximately 300° to 320° F., at the end of which the saponification was found to be complete. The melted grease was then directly drawn into the cooling trays or pans. The final grease thus produced had approximately the following composition:

	Per cent by weight
Sodium fat soap	10.9
Sodium rosin oil soap	9.5
Calcium rosin oil soap	2.0
NaOH	0.1
Free fat	1.0
Water	0.5
Mineral oil and unsaponifiable material	76.0

The finished grease had an A. S. T. M. penetration of above 27 at 77° F. and an Ubbelohde melting point of 380° F. Depending on the temperature and pressure conditions in the pressure still, the water content may be varied between 0.3% and 1.0%. The brick grease is uniform in texture, consistency, etc., and is highly suitable for the lubrication of bearings operating at temperatures as high as 325° F.

It is believed to be clear from the description and the examples given that the present invention is applicable to the manufacture of various types and grades of brick or mill greases, and that a wide variety of formulae may be used within the workable ranges of proportions of the various ingredients shown hereinabove. Thus, as pointed out above, the heating under pressure to various temperatures may produce a brick grease which has more or less water. Also, the percentage of the soap and of the mineral oil in the final grease may be varied. Thus, for a brick grease of the type described in the second example the percentage of soap may vary from about 12% to 30% by weight of the grease, while the quantity of mineral oil employed may be between about 50% and 85% of the final product. The water content of gel type brick greases manufactured according to the present process may vary from about 0.3% to 1.5% or even 2%, while for greases of the other types said water content may be as low as 0.1%. Similarly, the percentages of lime, and of the various fats, fatty acids and rosin oil may be changed depending on the characteristics desired. It is to be noted, however, that the quantity of alkali or alkalies used should be substantially sufficient to saponify all of the saponifiable ingredients employed. From about 3% to 20% of the grease are incidental unsaponifiables.

It is also apparent that many modifications may be made in the process described herein. Thus, to prevent excessive foaming during the drawing of the melted grease, the latter may be conveyed into a flash chamber which is also heated and maintained under a pressure, the grease being then withdrawn therefrom into the pans from the bottom of said chamber. This will eliminate the presence of foam in the trays since the froth remains in the upper part of the flash chamber.

The above description is not to be taken as limiting the invention, but merely as illustrative of the invention and the best mode of carrying out, and many changes can be made within the scope of the following claims.

I claim:

1. A process for the manufacture of a brick type soap and oil grease which comprises introducing into a saponification stage all of the saponifiable material, alkali and mineral oil required in the final product, heating the mass at superatmospheric pressure in the presence of a relatively small proportion of water to a temperature above the boiling point of water and for a time to cause saponification of the saponifiable ingredients and produce a grease mixture, maintaining the grease mass under pressure and regulating the temperature of the mass at an elevated degree with respect to the water content of the mass and the water content required in the final product, and, without having previously reduced the pressure, withdrawing the heated grease mass from the saponification stage while reducing the pressure to flash off water, thereby causing reduction in the water content to that required in the final product, and cooling the product.

2. A process for manufacturing a soap and oil grease which comprises introducing into a saponification stage all of the saponifiable material, alkali, water and mineral oil required in the final grease, heating the mass in a closed zone to a temperature and for a time to effect saponification and place the zone under superatmospheric pressure in the presence of water, adjusting the temperature of the saponified mass under pressure with respect to the water content of the mass and the water content required in the final product so as to reduce the water content upon pressure release to that required in the product, and withdrawing the grease from said pressure zone with reduction of pressure to substantially atmospheric pressure to cause flashing off of water and consequent evaporation to the content required in the final product.

3. A process for making a grease containing soap and mineral oil which comprises introducing into a saponification stage all of the saponifiable material, water, alkali and mineral oil required in the final product, heating the mixture in the presence of water for a period of time under atmospheric pressure and at a temperature

to cause saponification of the saponifiable material and yield a grease mixture, adjusting the temperature of the mass at an elevated temperature to produce superatmospheric pressure in the saponification stage by evaporation of water from the mass, the adjusted temperature being such with respect to the water content in the mass that the desired water content in the final product will result upon flashing the mass, and withdrawing the grease mixture from the saponification stage with reduction of pressure to substantially atmospheric pressure to flash off water and reduce the water content to that required in the final product.

4. A method according to claim 2 wherein the water content is reduced to less than 2% of the grease mixture.

5. A method according to claim 3 wherein the water content is reduced to less than 2% of the grease mixture.

6. A method according to claim 1 wherein the water content is reduced to between 0.1% and 1.0% of the grease mixture.

WILLIAM E. BRADLEY.

CERTIFICATE OF CORRECTION.

Patent No. 2,222,589. November 26, 1940.

WILLIAM E. BRADLEY.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, first column, line 50, for "excesive" read --excessive--; same page, second column, line 60, for "ben" read --been--; page 4, second column, line 75, claim 3, for "atmospheric" read --super-atmospheric--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 22nd day of April, A. D. 1941.

Henry Van Arsdale,
Acting Commissioner of Patents.

(Seal)