Patented Nov. 17, 1953

2,659,695

UNITED STATES PATENT OFFICE

2,659,695

LUBRICATING GREASES

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No Drawing. Application January 6, 1951, Serial No. 204,827

8 Claims. (Cl. 252—33.2)

This invention relates to improvements in lubricating greases.

In the past many difficulties have arisen in the manufacture of greases in which saponification is accomplished by the use of aqueous slurries of an insoluble metal hydroxide at elevated temperatures. In such processes of manufacture the handicap of considerable quantities of water is encountered. This water is difficult to remove and necessitates prolonged cooking, which in many cases does not effect complete removal. The water retained after cooking is completed detracts from the efficiency of the grease and may induce corrosion of the metal surfaces to be lubricated. In the case of water-proof greases the retained water may affect the greases' water resistance. Such greases have a slimy and murky appearance and develop lumpiness requiring the finished grease to be strained.

One object of this invention is to overcome the above mentioned difficulties. This and further objects will become apparent from the following description and examples.

According to the invention a mineral lubricating oil solution of fats or fatty acids is intimately contacted with an insoluble metal hydroxide, which is dispersed throughout a water-in-oil emulsion containing a petroleum mahogany sulfonate. It is preferable that the petroleum mahogany sulfonate be of the same metal as the insoluble metal hydroxide.

The emulsion containing the insoluble metal hydroxide may be prepared in any manner provided that the emulsion is not formed before the addition of the insoluble metal hydroxide. It is preferably formed by dissolving the petroleum mahogany sulfonate in a carrier oil, adding the insoluble metal hydroxide to form a paste, and then adding required amounts of water.

The carrier oil, i. e., the oil used in making the emulsion, should preferably be the same mineral oil as is used in the grease. The viscosity of this carrier oil should preferably not substantially exceed 47 seconds Saybolt at 210° F. and 310 seconds Saybolt at 100° F. Thus, if an oil having a greater viscosity is used as the base oil for the grease, a different lighter oil should be used as the carrier oil. The viscosity range of the carrier oil in any case should preferably be from about 38 to 47 seconds Saybolt at 210° F. and 110 to 310 seconds Saybolt at 100° F.

The amount of the petroleum mahogany sulfonate used should be controlled so that the amount present in the emulsion is from about 3 to 6%. The amount of water present in the

emulsion should be from about 30 to 60%, and the amount of carrier oil present should be from about 17 to 25%.

In making the grease, fats or fatty acids are dissolved in a base oil, the base oil being a mineral lubricating oil having a viscosity range of from 110 to 225 seconds Saybolt at 100° F. and 38 to 91 seconds Saybolt at 210° F. If necessary, the mineral lubricating oil may be heated to dissolve the fats or fatty acids. A quantity of the emulsion, containing a substantially stoichiometric amount of the metal hydroxide in relation to the fats or fatty acid, is then added with stirring to the oil, whereup saponification takes place. No heat need be supplied during saponification. A smooth, homogeneous paste is formed, no lumping of the mixture occurs, and there is no water separation, as is the case in conventional grease working. All the water contained in the grease formed is contained in the internal phase.

The amount of base oil used in dissolving the fats and fatty acids varies from 5 to 75%, depending upon the type of grease, and is as high as 80% for water pump grease, and as low as 5% for viscose pressure greases. The metal hydroxide used in accordance with the invention may be any of the metal hydroxides conventionally known and used in the production of grease, such as calcium hydroxide (lime), lithium hydroxide, barium hydroxide and strontium hydroxide. The petroleum mahogany sulfonate may be any petroleum mahogany sulfonate, but it is preferable that it be of the same metal as the metal hydroxide. Thus, when using lime, calcium petroleum sulfonate should be used. The fats and fatty acids used are those conventionally known and used in the production of greases, these being fatty acids containing between 12 and 20 carbon atoms and their glycerides.

The greases made under the new process are superior to those made in the conventional manner; for example, the consistency (ASTM worked penetration) of water pump greases made under the new process varies from about 180 to 200 as compared to 240 to 340 for those made in the conventional manner. The retention of the consistency (ASTM worked penetration) of the same grease remains constant after six months for the grease made under the new process, while it changes to from 300 to 380 for the grease made by the conventional method. The water resistance of grease made in accordance with the invention, measured by putting a ball of the grease on the end of a thermometer placed in a 600 beaker filled with water, boiling the water and noting the time the grease can withstand the softening and disintegrating effects of boiling water, is over 25 minutes, while that made according to the conventional method is only about 5 minutes. This water resistance remains the same for the grease made by the new process when tested after six months, while for the grease made according to the conventional method, it drops to less than three minutes. The corrosion inhibition of the new greases excels that of the greases made according to the conventional method.

The following examples are given by way of illustration and not limitation:

EXAMPLE 1

A mixture of 210 pounds of tallow fatty acids, 20 pounds of hydrofol fatty acids (hydrogenated fish oil containing 52.6% stearic acid, 42.6 palmitic acid, 3.7% myristic acid, 0.8% oleic and .3% lauric acid), and 43 gallons of mineral oil were first charged into a steam-jacketed kettle and the temperature raised to 150° F. to melt the fatty acids. Then a lime slurry obtained by stirring together 60 pounds of hydrated lime and 11 gallons of water were run in while agitating the whole mixture. Though the lime slurry was strained prior to adding it to the fatty material, lumping of the mixture was observed. As the saponification proceeded a great quantity of water separated from the batch, amounting to 65% of the water added. Sampling of the grease in half hour intervals showed that maximum saponification was obtained after four hours, but it required two additional hours to cook out the separated water.

After six hours of cooking the balance of the mineral oil (43 gallons) was stirred in. Straining of the finished grease was necessary in order to free it from lumps. The finished grease had a mucky and slimy appearance and as comparative figures will show later, an inferior consistency and water-resistance.

EXAMPLE 2

A similar grease was made, using the same amount of fatty material, mineral oil, lime and water as in Example 1, except that the lime was added in a water-in-oil emulsion.

The latter was made by stirring the lime into an oil solution of calcium petroleum sulfonate and then adding to the lime paste, the same amount of water as in Example 1. 29 pounds of a 30% solution of calcium petroleum sulfonates in oil were used to prepare the lime emulsion.

Though the saponification was started with the steam completely shut off, the mixture was observed to quickly form a smooth, homogeneous paste. There was no lumping of the mixture and no water separation whatever. Sampling of the batch at definite intervals of time showed that saponification was more complete and proceeded at much faster rate at the same temperature than in the case of Example 1.

EXAMPLE 3

A similar grease, using a lime emulsion was made with a mixture of tallow fat, instead of tallow fatty acids, using the same percentage of components as in Example 2.

Results were identical as in Example 2.

EXAMPLE 4

A similar grease as in Example 3 was made, using same percentages of raw material, except that a lime slurry was used instead of a lime emulsion. Formation of lumps and separation of water was observed as in Example 1. Also the properties of the finished grease were the same as of the grease prepared in Example 1.

A comparison of the ASTM penetration of the grease made in Examples 1, 2, 3 and 4 is shown in Table I below; a comparison of the water-resistance made in boiling water is shown in Table II below, and Table III shows a comparison of the rates of saponification by showing the percentages of completion of saponification next to the indicated time.

Table I

Examples	1	2	3	4
ASTM Penetration 77° F.	240	180	200	340
After six months	300	180	200	380

Table II

WATER PROOFNESS TEST

	Minutes
Example 1	5
Example 2	over 25
Example 3	over 25
Example 4	5

Table III

SAPONIFICATION RATE

Example	1	2	3	4
	Percent	*Percent*	*Percent*	*Percent*
After ½ hr.	60	82	80	58
After 1 hr.	64	85	83	60
After 2 hrs.	71	96	98	70
After 3 hrs.	84	(1)	(1)	84
After 4 hrs.	85	(1)	(1)	85
After 5 hrs.	86	(1)	(1)	86
After 6 hrs.	86	(1)	(1)	86

[1] Grease finished.

The finished greases of Examples 1 and 4 contained mechanically entrapped water in spite of prolonged heating, which was easily detected by slightly pressing the grease. This free water is highly objectionable to the grease since it causes softening of the grease on standing and affects its water-proofness. It is even more objectionable when considered from the point of corrosion of metal surfaces to be lubricated by the grease because each droplet of water forms a weak spot in the lubrication film, which form foci of corrosion of the metal.

EXAMPLE 5

A lithium grease was formed with 17.7% tallow fatty acid, 3.5% hydrofol fatty acid, 3.2% lithium hydroxide and 75.6% mineral oil by adding the fatty acids and lithium hydroxide slurry together with one-half of the mineral oil and heating the mixture to about 200° F. Saponification started but was not complete until the temperature was increased to about 300° F. and so maintained for about two hours. The balance of the oil was then added and the heating continued until a fluid gel had formed. To obtain this fluid gel formation the material had to be heated to about 450° F. and maintained at this temperature for an additional two hours. The finished grease had to be strained to remove the lumps.

EXAMPLE 6

A similar grease was prepared using the same percentages of fatty material, oil and lithium hydroxide, except that the latter was used in a water-in-oil emulsion having the composition as shown in Table IV. Saponification started even before heating, and was complete after cooking for about 15 minutes at about 230° F. The balance of the oil was then added and the heating continued to about 300° F. which resulted in a homogeneous fluid gel. The heat was then cut off and the finished grease cooled while stirring. No straining of the finished grease was necessary.

EXAMPLE 7

Barium grease and strontium grease were prepared in the same manner as the grease prepared in Example 6, using the same oil and fatty materials as were used in Example 6, except that the emulsions used contained the saponifying materials as shown in Table IV below. The greases obtained were of similar quality as those obtained in Example 6. No lumps were formed and no straining was necessary.

Table IV

For Lithium Grease—Lithium Emulsion	Li	For Barium Grease—Barium Emulsion	For Strontium Grease—Strontium Emulsion
	Percent	*Percent*	*Percent*
Lithium Petronate	24		
Barium Petronate		15	
Strontium Petronate			16
$LiOH.H_2O$	22		
$Ba(OH)_2 8H_2O$		35	
$Sr(OH)_2 8H_2O$			31
Oil	24	15	16
Water	30	35	37
	100	100	100

I claim:

1. In the method for the production of lubricating grease by saponification, the improvement which comprises intimately contacting a mineral lubricating oil, containing a solution of at least one member of the group consisting of fats and acids thereof, with an emulsion, said emulsion being a water-in-oil emulsion containing a petroleum mahogany sulfonate dissolved in a mineral oil, water in an amount to constitute the discontinuous phase of the water-in-oil emulsion and having an insoluble grease forming metal hydroxide dispersed throughout in amount sufficient to form a soap with said group member.

2. Improvement according to claim 1 in which said emulsion contains about 17 to 25% oil, about 30 to 60% water, and about 3 to 6% petroleum mahogany sulfonate.

3. Improvement according to claim 2 in which said hydroxide and said petroleum mahogany sulfonate are of the same metal.

4. A metal soap grease, having any water present in the internal phase, produced by the saponification of at least one member of the group consisting of fats and acids thereof dissolved in a mineral lubricating oil with a grease forming metal hydroxide dispersed throughout a water-in-oil emulsion containing a petroleum mahogany sulfonate.

5. An emulsion for the saponification of at least one member of the group consisting of a fat and an acid thereof dissolved in a mineral lubricating oil, which comprises a water-in-oil emulsion, containing a petroleum mahogany sulfonate dissolved in a mineral oil, water in an amount to constitute the discontinuous phase of the water-in-oil emulsion, and an insoluble grease forming metal hydroxide dispersed throughout in amount sufficient for the saponification.

6. An emulsion according to claim 5 in which said petroleum mahogany sulfonate is present in amount from about 3 to 6%, said oil being present in amount from about 17 to 25%, and said water being present in amount from about 30 to 60%.

7. An emulsion according to claim 6 in which said petroleum mahogany sulfonate and said hydroxide are of the same metal.

8. An emulsion according to claim 7 in which said metal is at least one member of the group consisting of calcium, lithium, barium and strontium.

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References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
2,111,907	Zimmer	Mar. 22, 1938
2,265,791	Zimmer	Dec. 9, 1941
2,343,737	Beerbower	Mar. 7, 1944
2,383,904	Zimmer	Aug. 28, 1945
2,394,907	Gallay et al.	Feb. 12, 1946
2,398,075	Brunstrum	Apr. 9, 1946
2,425,174	Carmichael	Aug. 5, 1947
2,485,861	Campbell	Oct. 25, 1949
2,535,101	Sproule et al.	Dec. 26, 1950