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ALUMINUM SOAP GREASES

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1 Claim. (Cl. 252—37)

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This invention pertains to aluminum soap greases and methods for their preparation. More particularly, it relates to an improved aluminum soap base lubricating grease suitable for use on the chassis of automotive vehicles and the like, which is modified to improve its texture and which is adapted to be produced by modern methods involving continuous processing with rapid cooling. This invention pertains further to an improved process for producing an aluminum soap grease having desirable characteristics of stability and consistency notwithstanding the use of rapid cooling techniques.

It has been known in the prior art that aluminum soap base greases may be improved in certain respects by the incorporation therein of various modifying ingredients. Thus in the patent to Zimmer and Morway, No. 2,365,037, there is disclosed an aluminum soap base grease in which is incorporated a small amount of aluminum naphthenate or aluminum oleate with a small quantity of a free acid, such as oleic acid or stearic acid. Also, in the patent to Sproule and Zimmer, No. 2,394,567, there is described a process for producing an aluminum soap grease which comprises the use of a phenolic compound as a modifier or stabilizer. This patent specifically mentions the use of tertiary octyl phenol and points out that by the use of the phenolic modifier aluminum soap greases may be rapidly cooled and still have a desirable consistency and gel structure, a difficulty which had previously been encountered in the continuous or rapid production of aluminum soap greases.

In the production of aluminum soap greases, for example greases containing aluminum stearate, the continuous and semi-continuous methods have been used in the past with only limited success. It has been found that without grease structure modifiers, such as those referred to above, grease produced by the use of continuous and semi-continuous apparatus with quick cooling apparatus sets up in hard crusts resulting in graininess and considerable syneresis. The use of an alkylated phenolic modifier as described in the patent to Sproule and Zimmer, No. 2,394,567, mentioned above, has resulted in some improvement in the grease but it has been found that there is a breakdown in consistency when greases produced by the continuous method are dispensed in high pressure grease guns and the like or otherwise subjected to substantial shearing stresses. Even with the alkylated phenol modifier, when aluminum soap greases are subjected to high shearing stresses, the grease structure is temporarily broken down, the composition reverting to a very soft fluid or semi-fluid mass which recovers part of this lost consistency in standing (delayed thixotropy) but not sufficiently fast to prevent the grease from running out of bearings or dripping onto the floors and parts of machinery where it is not desired. Such partial breakdown in structure obviously is objectionable to those who handle the lubricant.

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On the other hand, greases produced by the general process described in the Zimmer and Morway patent, No. 2,365,037, may in some instances lack the uniformity in consistency and structure which is desired, being subject to the deficiencies more fully pointed out in the Sproule and Zimmer patent referred to above.

We have found that by the addition of a substantially saturated acid in a quantity of approximately 10 to 20% of the aluminum soap, the amount of the alkylated phenol modifier hitherto required may be considerably reduced and also the amount of aluminum soap required to obtain a given grease consistency or hardness may be reduced appreciably. The structure and stability of the lubricant are improved even when the quantities of these other materials are appreciably reduced. Apparently the addition of a saturated fatty acid overcomes the tendency of aluminum soap greases to break down unduly under shearing stresses. Greases produced in this manner have been found to be exceptionally satisfactory for automotive chassis lubrication and related uses. The excellent qualities of adhesiveness to metal and rapid recovery of body after working make the aluminum soap greases, such as aluminum stearate greases, especially desirable for this purpose.

Prior to the present invention, a grease comprising, for example petroleum base mineral oil with a suitable quantity such as 7.5% by weight of aluminum stearate and a small amount of modifier such as 1.5% of an alkylated phenol, for example, iso-octyl phenol, appeared to be a very satisfactory lubricant unless and until subjected to high shear stresses such as are in-

volved in modern high pressure grease guns and the like. In view of the structural breakdown mentioned, and also because grease packages for the trade had to be allowed to stand for some 96 hours or longer before the grease would revert to a final solid grease structure ready for final inspection, further improvements in stability have been needed and are among the main objects of this invention.

A grease having good shear stability, produced by continuous methods involving rapid cooling, was compounded comprising 6.0% aluminum stearate of commercial grade and 1.2% of hydrogenated fish oil acids, which are substantially saturated acids having between 12 and 22 carbon atoms per molecule. As a modifier, 0.56% iso-octyl phenol was added, the soap and modifier being mixed into about 92% of a low cold test mineral oil having a viscosity of 900 to 1000 S. S. U. at 100° F. with addition of a small amount, about 0.25% of a tackiness agent, such as an oil solution of polybutene.

All of the above materials were charged to a grease kettle and the temperature raised to about 300° F. while mixing. At this temperature a fluid homogeneous mass was formed. The grease was cooling progresses. It prevents the grease from forming soft cores in shipping containers, a difficulty that formerly arose with frequency in aluminum soap greases produced by the rapid cooling process. Also the grease does not require a long standing time for reversion to a final solid grease structure. What is even more important, it shows no signs of syneresis or oil separation in containers over longer periods of time, even when shallow wells are dug into the grease to enhance the tendency toward oil separation. However, the most important effect of the added acid is to prevent structure breakdown of the grease under shearing stress. The following table shows the various characteristics of greases containing different amounts of aluminum soap (aluminum stearate), the iso-octyl phenol structure modifier and added fatty acid. All of the greases listed were prepared to an original worked penetration of 320 to 340 mm./10. It should be noted that the quantity of aluminum stearate used was considerably less in the examples where the fatty acid was employed. The optimum composition covered by this table appears to be that consisting of 6% aluminum stearate, 0.56% iso-octyl phenol and 1.2% fatty acid.

TABLE I

	Grease without added acid and with a large amount of modifier		Grease with varying amounts of added acid and small amount of modifier		
Formulation					
Aluminum Stearate—Per cent	8.50	7.00	6.00	6.00	6.00
Iso-Octyl Phenol—do	1.70	1.40	0.56	0.56	0.56
Tackiness Agent (Polybutene in oil)—do	0.25	0.25	0.25	0.25	0.25
Mineral Oil (900/1000 Vis./100° F.)—do	89.55	91.35	91.99	92.28	93.19
Saturated Acid of 12–22 carbon atoms—do			1.20	0.60	
Properties					
ASTM Penetration (unworked) 77° F	218	196	205	220	226
ASTM Penetration Worked 60 Strokes	332	343	320	318	307
ASTM Penetration Worked 150 Strokes	345	354	325	322	305
ASTM Penetration Worked 300 Strokes	344	354	331	321	317
ASTM Penetration Worked 600 Strokes	347	370	343	328	314
ASTM Penetration Worked 1200 Strokes	376	395	348	343	332
ASTM Penetration Worked 1800 Strokes	380	395	352	347	325
Penetration after passage through a standard commercial air operated grease gun	379	371	338	341	340
Appearance in container after reversion to solid grease	Excellent	Good	Excellent	Excellent	Syneresis
Syneresis after three months of normal bulk plant storage in 100 lb. containers	Large	Large	None	None	Large

next pumped into and through a scraped-surface water-jacketed cooler, so as to be cooled to an exit temperature of about 85° to 95° F. The grease emerging from the cooler was ready for direct packaging and was ready for final inspection in one-half to two hours after packaging.

Stearic acid may be used as the saturated fatty acid or various hydrogenated vegetable or fish oil acids which are commercially available may be employed. The optimum quantity of the free or excess fatty acid to be used appears to be in the range of about 10 to 20%, based on the quantity of aluminum soap employed. The aluminum soap is preferably aluminum stearate although other substantially saturated aluminum soaps having between 12 and 22 carbon atoms may be used if desired. The added or excess acid appears to have a synergistic effect on the grease modifier (iso-octyl phenol, as in the example given above). For this reason a considerably lesser quantity of the modifier can be employed than in the case where no free acid is used.

The use of the added acid in combination with smaller amounts of the modifier modifies the grease structure sufficiently to keep the composition substantially in a fluid state while the rapid

It has been found that the quantity of added fatty acid required varies somewhat with the source of the aluminum stearate, apparently because some aluminum stearates of commercial grade include appreciable quantities of free fatty acids. However, when purchasing aluminum stearate this free acid is held to a minimum by specification for economical reasons. Thus, where the aluminum soap already contains some free fatty acid, the effects of the addition of further acid will vary, depending on the original acid content of the soap. To determine the effect of the variations of different aluminum stearates, various batches of lubricating compositions were prepared by both rapid cooling and conventional pan cooling methods. The mineral oil used was a blend of lubricating oils having a viscosity after blending of 940 S. S. U at 100° F. Soap contents of the finished greases were adjusted to give products having worked penetrations between 320 and 340. All of the greases prepared by rapid cooling softened excessively when subjected to shear in a grease worker or in a standard grease gun. Table II shows the comparative structure stability characteristics of three greases prepared from commercial alumi-

num stearates by the continuous cooler and the pan methods, respectively.

TABLE II

Structure stability of aluminum stearate pressure lubricant

Aluminum Soap Source	A		B		C	
Manufacturing Process	Cooler	Pan	Cooler	Pan	Cooler	Pan
Formulation						
Aluminum Stearate, percent	7.00	6.00	8.50	9.00	8.00	6.50
Iso-octyl Phenol, do	1.40	------	1.70	------	1.60	------
Tackiness Agent, do	0.25	0.25	0.25	0.25	0.25	0.25
Mineral Oil, do	91.35	93.75	89.55	90.75	90.15	93.25
ASTM Penetrations						
Unworked	196	161	218	177	241	211
After 60 Strokes	332	320	332	339	326	335
After 1800 Strokes	395	338	380	352	382	360
After Passage Through Gun [1]	371	347	379	362	378	388
One Hour After Passage Through Gun [1]	327	324	330	345	360	348

[1] High pressure grease gun with needle fitting.

The data in Table II show that an aluminum stearate pressure type lubricant possessing adequate structure stability under shear could not be prepared by rapid cooling methods without a change in formula, it being noted that no fatty acids were added. The formulation was improved to an unexpected degree by the addition of stearic acid, or of a hydrogenated fish oil acid which is approximately equivalent to stearic acid. The quantity of acid employed in the latter series of tests was about 10% of the soap content and the iso-octyl phenol content, which had been 20% of the soap content in the experiments covered by Table II, was reduced to 10%. A typical product containing 7.0% of aluminum stearate, 0.7% hydrogenated fish oil acid and a mineral oil base stock of 940 S. S. U. viscosity at 100° F. with 0.6 to 0.7% of the modifier was quite satisfactory. It showed a worked penetration of 331 and a penetration of 328 after passage through a standard commercial grease gun. Other greases prepared from aluminum stearate obtained from some commercial sources showed slightly poorer stability than others even when the added acid content was increased to 20% of the soap content. These data representing greases containing varying quantities of aluminum stearate from various sources, are indicated below in Table III.

TABLE III

Aluminum Soap Source	A	B		C
Manufacturing Process	Cooler	Cooler	Pan	Cooler
Formulation				
Aluminum Stearate, percent	6.00	6.00	9.75	7.00
Hydrogenated Fish Oil Acid, do	1.20	1.20	------	0.70
Iso-octyl Phenol, do	0.60	0.60	------	0.70
Tackiness Agent, do	0.25	0.25	0.25	0.25
Mineral Lubricating Oil, do	91.95	91.95	90.00	91.35
ASTM Penetrations				
Unworked	190	212	------	200
After 60 Strokes	320	320	332	331
After 1,800 Strokes	352	348	360	345
After Passage Through Grease Gun	338	348	352	328
Appearance	Good	Good	Good	Good

The aluminum stearate grease obtained from commercial source C listed in the table above had particularly good structure stability when prepared on a laboratory scale by rapid cooling. It appears to have excellent structure stability even under conditions of high shear and to give good performance in commercial grease dispensing equipment.

The quantities of fatty acid to be added and the alkylated phenol modifier appear to be somewhat critical. The amounts used may be varied somewhat, depending upon the characteristics of the aluminum soaps with which they are used. Under some conditions both additives may be used in quantities as little as 0.1 to as much as 2.0% by weight, based on the total grease composition. The fatty acid may be from 1 to 3 times the quantity of the alkyl phenol. Generally speaking, the quantities employed will be between 0.25 and 2.0% for the fatty acid and 0.25 and 1.25% for the alkylated phenol modifier. For greases containing soaps of good commercial quality the range may often be even narrower. Based on the amount of soap used in the grease, the added fatty acid may be from about 10 to 20% by weight. A good over-all formula for the finished lubricant appears to be 5 to 9% by weight of aluminum soap of saturated or substantially saturated fatty acid, for example aluminum stearate, 89 to 94% of mineral lubricating oil of appropriate grade, i. e. of 35 to 1000 S. S. U. viscosity at 210° F. and 0.25 to 1.25% of a substantially saturated fatty acid, with 0.25 to 1.25% of modifier. The added acid is preferably stearic acid, although the other saturated fatty acids having between 12 and 22 carbon atoms, and mixtures thereof, may be employed. The phenol type modifier is preferably an alkylated phenol, particularly iso-octyl phenol, but other phenols such as resorcinol may be used, as described in application Serial No. 638,431 by Beerbower and Zimmer, now Patent No. 2,449,580. A tackiness agent also is usually desirable, though not indispensable. It is commonly used in quantities of 0.1 to 0.5% and other conventional additives such as corrosion inhibitors, oxidation inhibitors, extreme pressure agents, and the like, may be employed, as will be understood by those skilled in the art.

The following table gives still further data which were obtained on various samples of grease prepared from aluminum soaps obtained from various sources. It will be noted that good balance is required between the aluminum stearate or other soap, the fatty acid, and the alkylated phenol. Where the quantity of soap is reduced, the added acid needs to be increased somewhat. These data pertain to the rapid cooling process which has been found more difficult to apply to the production of satisfactory aluminum soap greases than to some of the other types of greases under commercial manufacture.

TABLE IV

Characteristics of aluminum stearate chassis lubricants prepared by rapid cooling

Soap Brand	A			B			C		
Formulation									
Aluminum Stearate, percent	6.00	6.00	6.00	6.00	6.00	6.00	7.00	7.00	7.00
Added Fatty Acid, do	1.20	0.60		1.20	0.60	0.60	0.70	0.70	0.35
Iso-octyl Phenol, do	0.60	0.56	0.56	0.60	0.60	0.30	0.70	0.35	0.70
Tackiness Agent, do	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Mineral Lubricating Oil, do	91.95	91.59	93.19	91.95	91.55	92.85	91.35	91.95	91.95
ASTM Pentration									
Unworked	190	220	226	212	219	279	200	289	197
After 60 Strokes	320	320	307	320	331	338	331	322	320
After 1,800 Strokes	352	342	325	348	358	347	345	340	333
After Balcrank Grease Gun	338	357	340	348	377	362	328	330	335
Appearance	Good	[1] Poor	[1] Poor	Good	[1] Poor	[1] Poor	Good	[1] Poor	[1] Poor

[1] Bleeding.

What is claimed is:

A lubricating grease composition consisting essentially of 91 to 93% by weight of mineral lubricating oil, 6 to 7% aluminum stearate, 0.6 to 1.2% of hydrogenated fish oil acids which are substantially saturated fatty acid having 12 to 22 carbon atoms per molecule, and 0.6 to 0.7% iso-octyl phenol.

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REFERENCES CITED

The following references are of record in the file of this patent:

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2,365,037	Zimmer et al.	Dec. 12, 1944
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2,394,567	Sproule et al.	Feb. 12, 1946
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