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POLYVALENT METAL MONO AND DICARBOXYLIC ACID SOAP THICKENED LUBRICATING OIL

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2 Claims. (Cl. 252—35)

This invention relates to greases prepared with soaps comprising polycarboxylic acids.

In copending application Serial No. 133,957, filed December 19, 1949, which issued as U. S. Patent No. 2,699,428, new soaps are described which are prepared by co-reacting a hydroxide with fatty acid material which includes a small proportion of polycarboxylic acid or acids. The co-forming of such soaps produces materials which are "soluble" in lubricating oils whereas normally the reaction product of a carboxylic acid with a metal to form a soap is not soluble.

This application is a continuation-in-part of our copending application Serial No. 133,956, filed December 19, 1949, now abandoned.

The greases produced from soaps of this new form are remarkably better than greases heretofore known.

Present greases are deficient in a number of respects. Of these the most important is the lack of ability of the greases to withstand working. While an oil does not wear out under use, greases hitherto known did. Standard working tests are known for greases, and a grease which stood up for 5,000 strokes of the testing machine without breakdown was considered a good grease. By means of the present invention greases may be produced which show no substantial breakdown or deterioration between 5,000 strokes and 100,000 strokes.

Another defect of present greases is that they do not set well at high temperatures. Thus, on a warm day it may be impossible to produce a satisfactory grease, and even after production of a grease at a low temperature, it tends to bleed or break down at a higher temperature. The greases of the present invention, produced with the soaps described, will wet at temperatures markedly above those heretofore possible, are stable at higher temperatures, and will set more quickly.

Another defect of former greases has been the inability to procure a very stiff grease without also making it grainy. The new improved greases may be made stiffer than before without spoiling the grease structure and generally with less soap than was heretofore required.

The greases of the present invention embody primarily aluminum soaps, but soaps of other metals are also useful, as for example sodium, lithium, calcium, barium, magnesium, zinc and lead.

The fatty acids which may be employed are normally between 8 and 22 carbon atom molecules. Normally these are referred to as "stearic acids" in commerce, but actually they may include much or all of other acids such as palmitic, myristic, 12-hydroxystearic, 9,10-dihydroxystearic, oleic, lauric, behenic and ricinoleic. The proportion of these acids does affect the soap and the ultimate grease. Therefore, grease formations with any particular soap is to some extent a matter of test and adjustment. One of the advantages of these soaps, however, is that they work substantially equally well with oils from various origins, whereas previously greases had to be carefully compounded with oils from selected sources.

The soap employed need not be completely reacted. For example, aluminum may form mono, di or tri salts, and normally the di salt is that employed. The other salts may likewise be used.

A preferred carboxylic acid is a dibasic acid, and the preferred dibasic is sebacic. The dimer of linoleic acid, known commercially as Emery M461–R is likewise particularly good. Adipic, pimelic and suberic acid have likewise produced very satisfactory greases.

The amount of polycarboxylic acid used may vary from 0.5% to 25% of the total acid by weight. Normally the ratio will be from 5–20% polycarboxylic acid and from 80–95% by weight of monocarboxylic acid.

As set forth in said copending application, the chemical structure of the soaps is not specifically known. It is assumed, however, that cross linkages are produced within the molecules so that highly linked structures of three dimensional characteristics are produced which are soluble in oils, the solubility presumably depending upon the preponderance of two dimensional chains in the molecules. Mere mixtures of separately formed polycarboxylic acid soaps and monocarboxylic acid soaps do not produce solubility in the polycarboxylic acid portions thereof.

The following examples illustrate the preparation of suitable greases:

Example 1

A mixture of 84 parts of hydrated lime with 1,000 parts of 300 S. U. V. @ 100 oil was added to 640 parts of a 40:40:20 of stearic, oleic and sebacic acids. This mixture was then heated to 250° F. until saponification was complete. The mixture was then cut back to a 10.0% soap content with more oil. The grease was then run through a homogenizer. The worked penetration of the grease was 260–270.

A similar grease made without the dicarboxylic acid gave a penetration of 370 at the same soap content.

Example 2

95 parts of hydrogenated herring acids and 5 parts of sebacic acid were saponified with 22 parts of caustic soda in aqueous solution. Aluminum sulphate was added in excess until the solution had a pH of 5. The solid precipitate was filtered, washed, dried and ground. (Aluminum Soap "A.") 6% of this product was dissolved in a Mid-Continent oil (300 S. S. U. at 100° F.) at 300° F. to form a stiff gel. For comparative purposes a similar sample was made using 100 grams hydrogenated herring acid and no sebacic acid. (Aluminum Soap "B.")

Comparative penetration results of greases (at 77° F.) containing 6% of Soaps "A" and "B" are shown in the following table:

	Soap "A"	Soap "B"
Unworked	177	210
60 Strokes	284	350
5,000 Strokes	305	430

Example 3

One part of lithium hydroxide and 6 parts of water were stirred together at 180° F. and then caused to react with 6.5 parts of 12-hydroxy stearate and 0.5 part of sebacic acid in 15 parts of a 100 S. S. U. at 100° F. oil. At the end of the reaction the temperature was raised to 300° F. to eliminate the water from the mixture. Seventeen parts of oil were then added and the temperature was raised to 380° F. for 2 hours. The temperature was decreased to 200° F. by adding enough oil to give a lithium soap content of 12% and

the entire composition was pumped through a 60 mesh screen. The worked penetration of the resultant mixture was 305. A similar run without the sebacic acid gave a grease that was too thin to take penetrations on.

Example 4

An aluminum soap was prepared in the same manner as described in Example 2, except that 90 grams of hydrogenated herring acid and 10 grams of sebacic acid were used (Aluminum Soap "C"). 6% of this material dissolved in the oil described in Example 2 by heating to 300° F. and then cooled to form a gel gave penetrations as follows:

Unworked	Worked	5,000
146	230	260

Example 5

A stearate was prepared in exactly the same manner as Example 2, using 95 grams of hydrogenated herring acid and 5 grams of Emery's 461R dimer acid (chemically the dimer of linoleic acid) (Aluminum Soap "D"). When 6% of this material was dissolved in the Mid-Continent oil as described previously, penetrations obtained were as follows:

Unworked	60 Strokes	5,000 Strokes
175	285	335

Example 6

A soap made and treated as Example 2 except that raw materials were 90 grams of hydrogenated herring acid and 10 grams of Emery's 461R dimer acid (Aluminum Soap "E"). A grease made as in Example 2 containing 6% of this soap gave penetrations as follows:

Unworked	60 Strokes	5,000 Strokes
150	250	280

Example 7

In order to check the effect of higher concentration of dicarboxylic acids a soap was made up using 85 grams of hydrogenated herring acid and 15 grams of Emery's 461R dimer acid. A grease made as in Example 2 containing 6% of this soap gave the following penetrations:

Unworked	60 Strokes	5,000 Strokes
165	255	290

Aluminum stearate greases prepared by dissolving six parts of aluminum stearate with 94 parts of oil using the material described in Example 6 gave excellent results with all the different types of oils tried. Results with the various oils are shown in the following table.

In every instance it will be noted that the penetration of yield of the grease was superior for the soap prepared in Example 6 and in addition the change in penetration on working (delta value) was smaller in every instance than for soaps not containing the dicarboxylic acid.

As further evidence of the efficiency of the stearate prepared in Example 6, greases were made up using a a lower percentage of stearate. The following tables show that products with contents as low as 3½% of aluminum stearate make greases of exceptionally good worked stability:

	Penetrations	
	60 Stroke	5,000 Stroke
Midcontinent Stock, 300 SSU @ 100° F.:		
4.0% Aluminum Soap "E"	335	365
4.5% Aluminum Soap "E"	310	345
5.0% Aluminum Soap "E"	290	320
5.5% Aluminum Soap "E"	270	300
6.0% Aluminum Soap "E"	250	280
Gulf Coastal Stock, 65 SSU @ 210° F.:		
3.5% Aluminum Soap "E"	335	365
4.5% Aluminum Soap "E"	295	325
5.0% Aluminum Soap "E"	270	300
6.0% Aluminum Soap "E"	230	260

In order to determine the effect of oxidation resistance upon the finished grease, greases made up from the stearate described in Example 2 were subjected to test in a Norma Hoffman bomb. Oxygen pressure drop after 100 hours at 210° F. was three pounds or less for all greases made with Pennsylvania base structure. This showed that the stearates have no effect upon the oxidation resistance of the finished grease.

The foregoing detailed description is given for clearness of understanding only, and no unnecessary limitations should be understood therefrom as modifications will be obvious to those skilled in the art.

We claim:

1. A grease comprising a lubricating oil and a grease thickening amount of a polyvalent metal soap, said soap comprising soap molecules co-formed by reaction of polyvalent metal ions with mixed mono and polycarboxylic acids, said polycarboxylic acid in the soap is sebacic acid, the proportion of sebacic acid being from about 0.5% to 25% of the total carboxylic acid by weight, and the monocarboxylic acid being of the class consisting of the fatty acids containing from 8 to 22 carbon atoms.

2. A grease as set forth in claim 1 in which the soap is an aluminum soap and the metal ions are aluminum ions.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

November 4, 1958

Patent No. 2,859,179 John H. Lux et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 6, after "dibasic" insert -- acid --; column 4, line 7, after "using" strike out "a".

Signed and sealed this 24th day of March 1959.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
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