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HIGH TEMPERATURE COMPLEX GREASE MANUFACTURING PROCESSES

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This invention relates to high temperature lubricating grease compositions and to a process for their preparation. Particularly, the invention relates to a process for the preparation of complex lubricating greases wherein the soap system consists of a combination of the salt of a low molecular weight acid and the soap of a high molecular weight acid wherein the mol ratio of the salt to the soap is above about 2.1:1. The improved process of this invention comprises the steps of preforming a salt-soap complex lubricating grease containing a salt-soap ratio of between about 10:1 to 50:1, adding a calculated amount of this salt-soap complex grease to a simple grease composition, heating the mixture to a temperature substantially below that necessary to complex the salt and soap and homogenizing the mixture to obtain a finished grease composition containing from 1 to 15 mols of salt per mol of soap.

In the preparation of high temperature lubricating grease compositions of the type contemplated herein, it has been found necessary to use temperatures in the order of 450 to 500° F. in order to form a true complex of the low molecular weight acid salt and the high molecular weight acid soap. The instant invention relates to the preparation of these general type lubricating grease compositions wherein a salt-soap complex grease is preformed that contains a ratio of salt to soap of about 10:1 to 50:1, and admixing this preformed complex with a major proportion of a simple soap grease in a second step. The incorporation of the two greases may be accomplished at temperatures below about 350° F., representing a tremendous saving in heat and time of manufacture and also eliminating the necessity of subjecting the lubricating oil to high temperatures.

The invention will be more clearly described by reference to the following illustrative examples:

EXAMPLE I

a. Preparation of high acetate soap concentrate—

Formulation:	Percent weight
Glacial acetic acid	25.20
Hydrogenated fish oil acids	2.00
Hydrogenated castor oil	2.00
Hydrated lime	17.40
Phenyl alpha-naphthylamine	1.00
Di-octyl azelate	52.40

Preparation

Charged all ingredients to a fire heated kettle with the exception of the acetic acid and the phenyl alpha naphthylamine. Warmed to 135° F., then added the acetic acid and continued heating to 500° F. Cooled grease to 300° F., added phenyl alpha naphthylamine, cooled to 250° F. and homogenized by passage through a Gaulin homogenizer.

Properties:

Appearance	Excellent uniform product.
Penetrations 77° F. mm./10—	
Unworked	174.
Worked 60 strokes	208.
Dropping point, ° F.	None.

b. Preparation of finished grease—

Formulation:	Weight percent
12-hydroxystearic acid	6.56
Hydrated lime	.96
Complex concentrate from part (*a*)	24.00
Phenyl alpha-naphthylamine	.80
Di-isooctyl azelate	67.68

Preparation

Water slurry of lime, fatty acid, and sufficient ester to make a 20% soap concentrate are charged to kettle and cooked until dehydrated with a maximum kettle temperature of 265° F. The complex concentrate (*a*) and remainder of oil added with the inhibitor and the temperature raised briefly to 330° F., followed by slow cooling. At 200° F. 1.0% H_2O added and the grease is homogenized after brief stirring. The mol ratio of low molecular weight acid to high molecular weight acid in the complex soap was 3.8.

Inspections:	After storage for 37 days	
Penetration, mm./10—		
Unworked	242	236
Worked 60 strokes	278	280
Unworked (after 48 hours)	235	---
Dropping point, ° F.	500+	---
Appearance	Smooth, homogeneous.	

EXAMPLE II

a. Preparation of high acetate soap concentrate

MOL RATIO ACETIC ACID TO FATTY ACIDS 40:1

Formulation:	Percent weight
Glacial acetic acid	14.20
Fish oil acids, hydrogenated	0.85
Hydrogenated castor oil	0.85
Hydrated lime	9.70
Phenyl alpha naphthylamine	0.50
Mineral oil	79.90

The materials other than acetic acid and phenyl alpha naphthylamine were charged to a fire heated grease kettle and warmed to 130° F. The acetic acid was charged and the mass heated to 500° F. The grease was cooled to 250° F., the phenyl alpha naphthylamine added and further cooled to 200° F. where it is homogenized if desired.

Properties:

Appearance	Soft semi-fluid.
Dropping point, ° F	500+.
Stability	Separates oil on standing unless homogenized.

b. Preparation of simple calcium soap grease—

Formulation:	Weight percent
Animal fat	9.50
Hydrated lime	1.35
Water	0.95
Mineral oil	88.52

Properties:

Appearance	Excellent, smooth, uniform product.
Penetrations 77° F. mm./10—	
Unworked	300.
Worked 60 strokes	310.
Dropping point, ° F	200 (water is evaporated and soap and oil separates).

c. Preparation of finished grease:

20% produce of *a* 80% product of *b*

The grease of *b* as finished or at 300° F. before the addition of water may be employed. In either case, 20% of the grease of *a* is added before heating to 300° F. or at 300° F., held at this temperature while agitating for approximately 3 hours, cooled rapidly to 200° F. and homogenized and packaged.

Properties:

Appearance	Excellent smooth grease.
Dropping point, ° F	450.
Penetration 77° F. mm./10—	
Unworked	315.
Worker 60 strokes	340.
Worked 75,000 strokes	384.
Wheel bearing test	Pass.
Leakage	Small amount of grease in retainer cup.

EXAMPLE III

a. The simple calcium soap grease preparation of Example II*b* was repeated.

b. Preparation of high acetate complex grease—

Formulation:	Percent weight
Glacial acetic acid	25.20
Hydrogenated fish oil acids	2.00
Hydrogenated castor oil	2.00
Hydrated lime	17.40
Phenyl alpha-naphthylamine	2.00
Mineral oil	51.40

Preparation

All ingredients were charged to a fire heated kettle and heated to 500° F., and then cooled to 200° F. Phenyl alpha naphthylamine was added at 250° F. during the cooling cycle.

Penetration 77° F. mm./10:	
Unworked	175.
Worked 60 strokes	200.
Appearance	Somewhat grainy.

c. Finished grease composition—	Percent
Grease of Example II*b*	85.00
Grease of *b* above	15.00
or	
Animal fat	8.08
Hydrogenated fish oil acids	.30
Hydrogenated castor oil	.30
Acetic acid	3.78
Hydrated lime	3.76
Phenyl alpha-naphthylamine	0.30
Mineral oil	83.48

Preparation

The animal fat, mineral oil and hydrated lime were charged to a steam kettle and heated to 300° F. That temperature was maintained until the fat was completely saponified. Then there was added the grease described in Example III*b*. Mixing was continued at 300° F. for 2 hours. The mix was then cooled to 200° F. and the product was homogenized.

Properties:

Appearance	Excellent smooth uniform grease.
Total saponifiable	12.46%.
Mol ratio acetic acid to high molecular weight acids	1.8:1.
Penetrations 77° F. mm./10—	
Unworked	300.
Worked 60 strokes	310.
Worked 10,000 strokes	360.
Dropping point ° F	485.

EXAMPLE IV

	Percent
Grease of Example II*b* (simple calcium soap grease)	45.00
Grease of Example III*b* (complex calcium soap grease)	12.50
Mineral oil	42.50
or	
Animal fat	4.28
Hydrogenated fish oil acids	.50
Acetic acid	3.15
Hydrated lime	2.91
Phenyl alpha-naphthylamine	0.25
Mineral oil	88.41

Preparation.—Similar to Example IIIc

The mol ratio of acetic acid to high molecular weight acid was 2.8:1.

Properties:

Appearance	Excellent.
Total saponifiable	8.43.
Penetrations 77° F. mm./10—	
Unworked	323.
Worked 60 strokes	340.
Worked 10,000 strokes	362.
Dropping point, ° F	500.
Wheel bearing test	Pass, excellent, no leakage and no fluidization.

(Added 0.25% phenyl alpha naphthylamine inhibitor to grease before running following tests.)

Norma Hoffman oxidation:	
Hours to 5 p. s. i. drop in oxygen pressure	140
Lubrication life—10,000 R. P. M.—	
250° F., hours	1128

As is shown by the examples above, the lubricating oil used may be either a naturally occurring mineral lubricating oil or a synthetic lubricating oil. It will be understood that the lubricating oil viscosity will preferably be within a range of from about 35 SUS to about 150 SUS at 210° F. In addition to diester synthetic lubricants illustrated by Example I, other forms of synthetic lubricating oils such as complex esters prepared by the coreaction of alcohols, dibasic acids and glycols, formals, polymerized olefins, ether alcohols, ether esters, ester ethers and the like may be used.

The complex grease compositions resulting from the instant inventive process preferably contain at least 1 mol of the low molecular weight acid per mol of the high molecular weight acid. The upper limit of the salt-soap ratio will be in the vicinity of 15 although it may be desirable for some purposes to have a ratio even higher than this.

It is generally desired that the finished grease composition contain between about 5% and 30% by weight of total salt soap complex. From 8 to 20 weight percent is preferred.

Although in the examples cited above, calcium is the alkaline earth metal used, and although calcium is contemplated in the preferred embodiment of this invention, it will be understood that other alkaline earth metals such as barium, magnesium and strontium may be used

as mixed bases. It has been found, however, that the alkali metal complexes yield grease compositions of undesirable properties when the mol ratio of low molecular weight acid to high molecular weight acid is in excess of about 2:1. Although it is preferred that both the salt and the soap be of the same metal, it is not necessary that this be so. For example, the calcium soap of the fatty acid may be used in conjunction with a barium salt of a low molecular weight acid.

The high molecular weight acids that are used in forming the grease compositions of this invention may be any of the commonly known grease making acids having from 12 to 30 carbon atoms, preferably from 18 to 22 carbon atoms, per molecule. These acids include stearic acid, hydroxy stearic acid, dihydroxy stearic acid, arachidic acid, hydrogenated fish oil acids, tallow acids, oleic acids, ricinoleic acids, etc.

The low molecular weight acid operable for forming the greases of this invention is preferably acetic acid. However, other saturated or unsaturated aliphatic mono- or poly-carboxylic acids having about 1 to 6 carbon atoms per molecule are operable. A partial list of operable low molecular weight acids includes formic acid, acetic acid, propionic acid, butyric acid, furoic acid, acrylic acid, adipic acid, including their hydroxy derivatives such as lactic acid, etc.

To summarize briefly, this invention relates to an improved process for the preparation of complex salt-soap thickened lubricating grease compositions. The process involves two major steps wherein there is first formed a salt-soap complex lubricating grease which contains a high ratio of low molecular weight acid to high molecular weight acid, preferably a mol ratio of about 10:1 to 50:1. This complex is then admixed with a simple alkaline earth metal soap grease in a second step in proportions such that the finished composition contains a mol ratio of low molecular weight acid to high molecular weight acid of between about 1:1 and 15:1. The second step may be carried out at temperatures substantially below those necessary to form the salt soap complex, that is, substantially below about 350° F. The resulting grease compositions have all the desirable properties of similar prior art greases but have the advantage of subjecting the major portion of the lubricating oil to substantially lower temperatures than was hitherto thought possible.

What is claimed is:

1. A process for the preparation of a high temperature lubricating grease composition wherein a major proportion of the lubricating oil in the final composition has not been subjected to high temperatures above about 350° F. which comprises the steps of forming at a temperature in the range of 450° to 500° F., a complex-thickened grease from a lubricating oil, an alkaline earth metal soap of a grease-making acid having in the range of 12 to 30 carbon atoms per molecule, and an alkaline earth metal salt of a monocarboxylic acid having in the range of 1 to 6 carbon atoms per molecule, with the mole ratio of salt to soap being in the range of 10:1 to 50:1, admixing at a temperature in the range of 300° to 350° F. a minor amount of said complex-thickened grease with a major amount of simple soap grease formed from a lubricating oil and an alkaline earth metal soap of a grease-making acid having in the range of 12 to 30 carbon atoms per molecule, in proportion such that the mole ratio of salt to soap in the finished formulation is in the range of 1:1 to 15:1 and the total salt-soap content thereof is in the range of 5 to 30% by weight, and homogenizing the admixture to obtain the finished composition.

2. The process of claim 1 wherein said alkaline earth metal salt is calcium acetate.

3. A process for the preparation of a high temperature lubricating grease composition wherein a major proportion of the lubricating oil in the final composition has not been subjected to high temperatures above about 350° F. which comprises the steps of forming at a temperature in the range of 450° to 500° F. a complex-thickened grease from a lubricating oil, a calcium soap of a grease-making acid having in the range of 12 to 30 carbon atoms per molecule and calcium acetate, the mole ratio of acetate to acid being in the range of 10:1 to 50:1, admixing at a temperature in the range of 300° to 350° F. a minor amount of said complex-thickened grease with a major amount of simple soap grease formed from a lubricating oil and a calcium soap of a grease-making acid having in the range of 12 to 30 carbon atoms per molecule, in proportion such that the mole ratio of acetate to soap in the finished formulation is in the range of 1:1 to 15:1 and the total acetate-soap content thereof is in the range of 5 to 30% by weight, and homogenizing the admixture to obtain the finished composition.

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