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2,939,840

**SILICA-THICKENED GREASE CONTAINING
ALKYLENE CARBONATE DISPERSANT**

Ernest T. Fronczak, Crystal Lake, Ill., assignor to The Pure Oil Company, Chicago, Ill., a corporation of Ohio

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11 Claims. (Cl. 252—28)

This invention relates to new and useful improvements in greases and more particularly to greases which are formed by the thickening of oleaginous liquids with finely divided silica. In particular, this invention is directed to improved silica-base greases utilizing improved dispersion aids or dispersants which produce more homogeneous greases in greatly improved yield.

The classical definition of greases has embraced gelatinous, semi-solid, or solid lubricant materials produced by the combination of a liquid petroleum product with a soap. In recent years, however, there has been developed a number of greases which use non-soap or inorganic gelling agents. In *Manufacture and Application of Lubricating Greases* by Boner, Reinhold Publishing Corporation, 1954, there is one entire chapter and several other sections devoted to non-soap-thickened greases.

Non-soap-thickeners for preparation of greases which have received the most commercial attention have been finely-divided silica and chemically-modified bentonite clays, known as Bentones. Silica-thickened greases and Bentone greases are discussed at some considerable length in the aforementioned Boner text. These greases are similar only in that they are non-soap greases, and possess more differences than similarities. The silica-thickened greases and Bentone greases are each prepared utilizing standard grease making techniques in which the respective gelling agent is mixed with an oleaginous liquid, which may be a lubricating oil or a synthetic lubricant, with the aid of a dispersant material. The silica-thickened greases differ markedly from the Bentone greases in that the latter are very stable in the presence of water while the former disintegrate completely unless special waterproofing materials are used in the grease composition. These greases also differ markedly in the types of dispersants which can be used for effecting a dispersion of the gelling agent in the lubricant liquid. For example, butyl stearate and coconut oil are each reported (in Patent 2,662,058) to be dispersion aids for Bentone greases. These materials have been found to be useless as dispersants for silica-thickened greases. On the other hand, certain substituted carbamate salts, such as dimethylammoniumdimethylcarbamate, have been found to be useful as dispersants for silica-thickened greases and are not effective as dispersants for Bentone greases. It is seen, therefore, that the differences between the Bentone greases and the silica greases are so marked as to negate the probability of predicting useful compositions of one type of grease based upon known properties of the other types of greases.

It is therefore one object of this invention to provide an improved silica-thickened lubricating grease.

Another object of this invention is to provide a silica-thickened lubricating grease having an improved dispersion aid which produces a grease of higher yield.

A feature of this invention is the provision of an improved silica-thickened grease utilizing an alkylene carbonate, such as propylene carbonate, as a dispersion aid to produce an improved grease of higher yield.

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Other objects and features of this invention will become apparent from time to time throughout the specification and claims as hereinafter related.

This invention consists of a new and improved grease composition in which an oleaginous liquid is thickened with finely-divided silica particles which are dispersed with a small amount of an alkylene carbonate. When a grease is prepared in accordance with this invention using an alkylene carbonate as a dispersant for the silica gelling agent, a higher yield product is obtained than when prepared with other known dispersants.

In carrying out this invention, different greases were prepared for comparison using finely-divided silica as the gelling agent and using various dispersing agents, utilizing standard grease-making techniques.

Example 1

An NLGI #1 grease was prepared using finely-divided silica as the gelling agent for a mixture of bright stock and neutral oils. The silica gelling agent used for this grease is preferably a finely-divided amorphous silica, marketed under the proprietary name of Cab-O-Sil. This silica is more finely divided than other silica products and is in the form of an amorphous material which is produced by a high temperature vapor phase reaction of a siliceous material. The most common process for manufacturing this type of finely-divided silica is by the high-temperature, vapor-phase hydrolysis of silicon tetrachloride. This finely-divided silica can also be made by the high-temperature, vapor-phase thermal decomposition of silicon compounds such as silicon esters and also the high-temperature, vapor-phase hydrolysis of silicon compounds with superheated steam, e.g. the steam hydrolysis of silicon esters or silanes. The dispersing agent used for this grease was a polyalkylene glycol, marketed under the proprietary name of Ucon LB 550X, which performs a dual function of dispersing the silica and imparting water resistance to the grease. This polyalkylene glycol is oil-soluble material produced by the reaction of butanol and 1,2-propylene oxide, has an average molecular weight of about 1400 (acetyl value), and following physical characteristics:

45	Viscosity:	
	SUS @ 210° F.-----	91.9
	SUS @ 100° F.-----	550
	Viscosity index -----	140
	Pour point, ° F.-----	-30
50	Specific gravity @ 60° F.-----	1.003
	Flash point, ° F.-----	510
	Fire point, ° F.-----	580

In preparing this grease, a mixture of 81.09 parts by weight of 200 Vis. Neutral Oil, 9.01 parts by weight of 160 Vis. Bright Stock, and 0.9 part by weight of Ucon LB 550X (polypropylene-butylene glycol) were charged to a conventional grease-processing kettle, and thoroughly agitated and heated to 190°-205° F. The silica gelling agent (Cab-O-Sil, finely-divided amorphous silica) in the amount of 9.0 parts by weight was added to the hot mixture in the kettle with continued agitation. The mixture was then agitated further for about 90 minutes while the temperature was maintained at 190°-205° F. The grease mixture was then charged to a colloid mill with a rotor-stator clearance of 0.003 inch and was milled at a flow rate of 0.6-0.7 lb. per mintue. After milling, the grease was allowed to cool and subjected to the standard cone penetration test. The grease produced in this manner had a worked penetration of 330, which is in the range of a NLGI #1 grease.

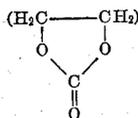
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Example II

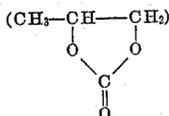
In another experiment, a grease was prepared according to this invention in the following manner. A mixture of 9.01 parts by weight of 160 Vis. Bright Stock, 81.09 parts by weight of 200. Vis. Neutral Oil, and 0.9 part by weight of propylene carbonate were charged to a conventional grease-processing kettle. The mixture was thoroughly agitated and heated to 190°–205° F. and 9 parts by weight of a silica gelling agent (Cab-O-Sil, finely-divided amorphous silica) was added to the hot mixture with continued agitation. After the addition of the gelling agent was completed, agitation of the mixture was continued for about 90 minutes at a temperature of 190°–205° F. The grease composition thus obtained was then charged to a colloid mill with a rotor-stator clearance of 0.003 inch and was milled at a flow rate of 0.6–0.7 lb. per minute. The grease was then cooled and tested for consistency using the standard cone penetration test. The grease obtained in this manner had a worked penetration of 251 and corresponded to an NLGI #3 grease. The grease which was thus produced was of much higher yield than the grease produced in the Example I. This grease had excellent high temperature lubricating properties but was deficient in resistance to water. However, the addition of any suitable water-proofing agent, such as high molecular weight polyalkylene glycols will produce desired water resistance in the grease. Other additives can be used, if desired, to impart E.P. properties to the grease or to protect against oxidation, corrosion, etc.

In formulating a grease composition according to this invention any oleaginous liquid, including lubricating oil fractions, synthetic lubricants (such as polyalkylene glycols, silicones and esters) or mixtures thereof, which have suitable viscosity characteristics, may be used as the base for the grease. Any finely-divided silica may be used as the gelling agent, although a finely-divided amorphous silica produced by high-temperature reduction, hydrolysis, or pyrolysis of silicon compounds such as silicon tetrachloride and silicon ester, is preferred. Finely-divided silica aerogels are also useful for this purpose. Any effective water-proofing agent, such as polyalkylene glycols, can be used to impart water resistance to the grease, and other additives may be used for their specific purposes if desired. As is disclosed in co-pending application, Serial No. 654,638, filed April 24, 1957, Ucon LB 550 X, the dispersant used in Example I, is a satisfactory water-proofing agent which is added to silica-thickened greases along with other dispersing agents to produce a water-resistant grease. As the dispersing agent, any alkylene carbonate having 2–18 carbon atoms per molecule may be used. Alkylene carbonates which are satisfactory dispersants include:

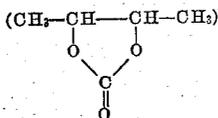
Ethylene carbonate



Propylene carbonate

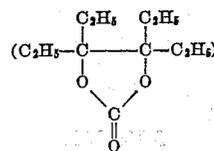


Butylene carbonate

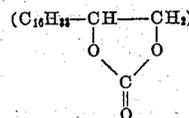


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Tetraethyl ethylene carbonate



And n-hexadecyl ethylene carbonate



In preparing greases according to this invention the silica gelling agent is added in an amount of 5–20% by weight of the total composition. The alkylene carbonate is added in the amount of 10–30% by weight of the silica used, or 0.5–6.0% by weight of the total composition. The water-proofing agent, which may be used if desired, is added in the amount of 0.5–5.0%. The balance of the composition is the oleaginous lubricating liquid used as the base thereof. In formulating greases in accordance with this invention it has been found that thicker greases can be prepared by increasing the amount of silica used, or by increasing the proportion of the alkylene carbonate used. In general, an increase in the alkylene carbonate-to-silica ratio will increase the thickness or consistency of the grease for any given silica concentration therein.

What is claimed is:

1. A lubricating grease composition comprising a major portion of an oleaginous lubricating liquid having incorporated therein sufficient finely-divided silica to produce a gel-like grease structure and a sufficient amount of a C₂–C₁₈ alkylene carbonate to effect a complete dispersion of the silica.

2. A grease as defined in claim 1 which includes a water-proofing agent for the silica.

3. A grease as defined in claim 1 in which the dispersant is a C₂–C₁₈ alkylene carbonate and is present in an amount of 10–30% of the silica.

4. A grease as defined in claim 3 in which the alkylene carbonate is propylene carbonate.

5. A lubricating grease comprising a major portion of a petroleum lubricating oil having incorporated therein sufficient finely-divided silica to produce a gel-like grease structure and sufficient amount of a C₂–C₁₈ alkylene carbonate to effect a complete dispersion of the silica.

6. A grease as defined in claim 5 which includes a water-proofing agent for the silica.

7. A grease as defined in claim 5 in which the alkylene carbonate is present in an amount of 10–30% of the silica.

8. A grease as defined in claim 5 in which the silica used is an amorphous silica of very fine particle size produced by a high-temperature, vapor-phase reaction of a siliceous material.

9. A grease as defined in claim 7 in which the alkylene carbonate is propylene carbonate.

10. A lubricating grease comprising a major portion of a petroleum lubricating oil having incorporated therein 5–20% of weight of total mixture of a gelling agent consisting of a very fine amorphous silica produced by a high-temperature, vapor-phase reaction of a siliceous material, and 0.5–6.0% by weight of the total mixture of a dispersant for the gelling agent consisting of propylene carbonate.

11. A lubricating grease as defined in claim 10 which includes 0.5–5.0% by weight of the total mixture of a polyalkylene glycol water-proofing agent for the dispersed silica.

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