Patented Dec. 30, 1952 2,623,853

UNITED STATES PATENT OFFICE

2,623,853

GREASE COMPOSITIONS

Fred H. Stross, Berkeley, Calif., assignor to Shell Development Company, Emeryville, Calif., a corporation of Delaware

No Drawing. Application April 6, 1951, Serial No. 219,759

12 Claims. (Cl. 252—28)

1

This invention relates to lubricants and more particularly to the bodied types, including those lubricants generally embraced by the term "greases."

The production of bodied lubricants is customarily carried out by the gelling of lubricating oils with fatty acid soaps, as well as the soaps of sulfonic acids or the corresponding soaps of aliphatic dicarboxylic acids. The principal shortcomings of greases so prepared comprises their relatively high thermal coefficient of viscosity as well as their tendency to fluidize at relatively moderately high operating temperatures. Recent advances in this art have included the preparation of greases gelled with inorganic colloidal materials such as silica and other amorphous colloids. These greases have substantially higher resistance to thermal influences since their structure is relatively stable up to the boiling point of their lubricating oil component. They exhibit no phase changes such as those shown by ordinary soap greases.

A more recent type of grease somewhat similar to greases formed from amorphous inorganic colloids comprises those gelled with the so-called "onium-clays." In the preparation of this latter type of grease, clays are dispersed in a swelling medium, such as water, and treated with an onium compound so as to positively cause replacement of the alkali metal cations (forming part of the clay structure) with an onium radical. Under these conditions the gelling agent thereupon ceases to be an inorganic entity, since it is composed of both organic and inorganic portions. It has been stressed in the literature disclosing this type of grease that metallic ion replacement with the onium ions was essential for the production of such greases.

While these greases exhibit many of the desirable properties of grease compositions and are similar in their thermal responses to grease gelled with colloidal silica, several disadvantages appear to be inherent due to their chemical composition. In the first place, this latter type of grease emulsifies to a great extent in the presence of water. While the act of emulsification is by itself of no consequence, in effect this produces a relatively fluid composition which eventually runs out of bearings or other surfaces lubricated therewith. The most serious disadvantage of such greases, however, comprises their inability to prevent corrosion of bearing surfaces, when water is present, during use of the grease. An investigation of the fundamental reasons for this shortcoming has disclosed the fact that the onium ions, being chemically bound to the clay

2

nucleus, are not readily displaced from the clay surface; at the same time metallic surfaces, such as those found in the usual automotive bearings, are preferentially wet by water which in turn displaces oil therefrom. Hence, it will be seen that the metallic bearing surface is exposed to the corrosive influences of water due to the inability of the onium clay grease to afford it sufficient protection.

It is an object of the present invention to provide improved grease compositions. It is another object of this invention to provide novel grease compositions exhibiting minimum emulsification characteristics. It is a further object of this invention to provide greases capable of protecting metallic surfaces against corrosion. Another object of this invention comprises the production of greases gelled with clays having minimum emulsification characteristics and maximum corrosion protecting properties. Other objects will become apparent during the following discussion.

Now in accordance with this invention, it has been found that bodied lubricants may be produced which are gelled to grease consistency with natural or synthetic clays. Moreover, the clays employed in the present compositions are substantially in their original inorganic form, having absorbed on their surfaces cationic hydrophobic surface-active agents, said agents containing only trivalent nitrogen atoms.

The greases prepared according to the compositions of this invention are distinguished from those discussed hereinbefore in that the gelling agent is composed principally of inorganic colloidally dispersed clay, retaining substantially all of its original alkali metal cations. However, the retention of the original metallic cations is not a primary requisite for the production of the present greases, instead the primary difference in these clays comprises the fact that the hydrophobic surface-active agent is merely absorbed on the colloidal surfaces of the clay rather than being reacted with it as in the case of the "onium clays." It has been found, as the data will prove in the examples contained hereinafter, that greases prepared according to this invention are substantially superior to those produced with the known onium clays.

The compositions of this invention comprise the following ingredients in the approximate proportions:

(1) Colloidally dispersed clay: from about 3% to about 20% by weight of the total grease composition,

(2) Cationic surface-active agent: from about 25% to about 100% based on the weight of the clay,

(3) Organic lubricating liquid: substantially the balance of the composition.

The cationic hydrophobic surface-active agents useful in the compositions must be chosen so as to provide a minimum possibility for reaction with any portion of the clay molecules. Since onium clays are produced by reaction with onium compounds such as ammonium salts or by reaction of acidified clays (i. e., montmorillic acid) with free amines, these types of compounds or reactants are to be avoided. The reason for this avoidance will become apparent during the following discussion.

In order to prevent any substantial amount of reaction between the clay and absorbed amine, it appears to be necessary to employ cationic surface-active agents which contain substantially only nitrogen atoms in their trivalent state. This naturally excludes onium compounds from the class of materials to be absorbed on the colloidal clay surfaces. Likewise, in the preparation of the present compositions, it is necessary to employ clays which have not been acidified so as to remove the replaceable alkali metal cations. Hence, the present compositions are to be prepared by the use of natural or synthetic clays wherein the majority of the ion-exchange sites are satisfied by the presence of metallic cations. moreover, these "clay-salts" (that is, natural or synthetic clays) must be treated with trivalent nitrogen containing surface-active agents.

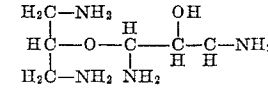
The cationic surface-active agents especially preferred for use in the present compositions comprise those which are substantially water insoluble or highly hydrophobic. This class, therefore, includes principally amines which term is understood to include hydrophobic amido-amines as well as hydrophobic amines. Several of the preferred classes of such substances are as follows: Primary, secondary, tertiary alkyl amines and other aliphatic amines, including dodecylamine, hexadecylamine, octadecylamine and mixtures thereof. These are normally regarded as the "fatty amines." Preferably, the amines should contain at least 10 carbon atoms and, still more preferably, at least 10 carbon atoms in a single aliphatic hydrocarbon chain. Other suitable amines useful in these compositions include aromatic amines such as naphthylamine, and heterocyclic amines such as pyridine and pyrimidine derivatives. An especially preferred type of heterocyclic amines comprises those included in the tetrahydropyrimidine nucleus and particularly those having at least 10 carbon atoms per molecule. Such materials include alkylated 1,2,3,4-tetrahydropyrimidines, 2,3,4,5-tetrahydropyrimidines and 3,4,5,6-tetrahydropyrimidines. A typical member of this species is 2-heptadecyl and 3,4,6-trimethyl, 3,4,5,6-tetrahydropyrimidine. Other suitable materials are the following: 2-dodecyl, 4,4,6-trimethyl, 3,4,5,6-tetrahydropyrimidine; 2,4-dimethyl, 2,4,6-trioctyl, 2,3,4,5-tetrahydropyrimidine; 6-heptadecyl, 1,2,3,4-tetrahydropyrimidine.

Another class of suitable hydrophobic surface-active agents may be generically described as hydroxyalkyl polyalkylene amines. These are preferably in the form of amido-amines in which at least 30% of the amino nitrogen atoms are in the amino form with relatively high molecular weight organic acids. Other materials include polyalkyl amines, aliphatic hydroxy amines and amido alkyl amines. Suitable polyalkylene polyamines include such substances as triethylene tetramine and tetraethylene pentamine.

Suitable hydrophobic derivatives are those in which a hydroxyalkyl group is attached to a nitrogen atom, such as hydroxyethyl ethylenediamine or those in which the hydroxy radical is a substituent attached to a hydrocarbon chain separating two nitrogen atoms. Such a material is: 2-hydroxy-1,3-diaminopropane.

An especially desirable type of additive for use in the present compositions comprises the complex mixtures of amines obtained by condensation of a monohaloepoxyalkane with ammonia or a low molecular weight aliphatic amine. The preferred type of material within this class includes the condensation products of epichlorohydrin and ammonia. Generally, these materials are prepared by heating the ammonia or amine with the mono-haloepoxyalkane at a temperature between about 20° C. and about 60° C. for a period between about 10 minutes and about 4 hours. Preferably, the ammonia or amine is present in a ratio of between about 4 and about 20 moles per mole of monohaloepoxyalkane.

Under these reaction conditions a mixture of derivatives is formed. Ordinarily, the reaction product comprises from 5% to 50% of 1,3-diamino 2-hydroxypropane with minor amounts each of dimeric and polymeric derivatives. The exact configuration of these dimers and polymers has not been determined. It is believed that they are for the most part linear in structure and may be linked through either the nitrogen atoms, or through hydroxyl groups to form either type dimers or polymers. The dimer is believed to have the following structure:



The above types of materials may be utilized without amide formation if they are hydrophobic in nature. However, in many cases, it is preferable to cause amide formation to take place with high molecular weight organic acids in order to produce amido amines having especially suitable properties for use in these compositions. Preferably, the acids employed are higher fatty acids having at least 12 carbon atoms and generally are derived from mixtures of naturally occurring materials such as animal and vegetable fats and oils. Similar results are obtained by partial amide formation with still more complicated mixtures, such as the crude mixture of acids known as "tall oil." The most effective type of such mixture comprises those mixtures having a preponderance of fatty acids containing from 14 to 20 carbon atoms and preferably from 16 to 18 carbon atoms.

The partial amides especially suitable for use in this invention may be obtained by heating the amines, such as the condensation product of epichlorohydrin and ammonia, to a temperature between about 150° and 225° C. for a period between about 15 minutes and 2 hours. Under these circumstances, the amides so formed comprise from about one-third to about two-thirds of the nitrogen atoms present in the amine molecule.

Cationic surface-active agents, such as those described above, are to be absorbed on the surfaces of colloidal clay in such a way that reaction with the clay surface is held to a minimum. Clays either of natural or synthetic derivation, especially suitable for use in the present com-

positions are those exhibiting substantial swelling properties in water. These swelling properties appear to be allied to the base exchange capacity of the clay although the latter capacity is not utilized in the formation of present compositions. However, instead of defining the preferred class of clays in terms of a vague degree of swelling capacity, it is believed preferable to define the clays to be used as those exhibiting an exchange capacity of at least 25 (based on the milli-equivalents of exchangeable base capacity per 100 grams of clay) and preferably greater than about 60. The montmorillonites are especially suitable and have a base exchange capacity of between about 60 and 100. The clays particularly contemplated in these compositions include montmorillonites bearing cations such as sodium, potassium, lithium. These include bentonites such as those of the Wyoming bentonite type. Magnesium montmorillonites (especially hectorite) and saponite are highly efficient in these compositions. These clays are characterized by an unbalanced crystal lattice and are believed to have negative charges which are normally neutralized by inorganic cations. As found in nature they exist as metallic salts of the weak clay-acid with bases such as the alkali or alkaline earth metal hydroxides. The exact composition of clays is not subject to precise description since they vary widely from one natural deposit to another. As far as present knowledge permits, they may be described as complex inorganic silicates such as aluminum silicate, magnesium silicate, barium silicate and the like.

While the natural clays provide a cheap and large source of inorganic gelling agents, they possess the disadvantages of containing abrasive materials which must be separated therefrom and of varying to a substantial degree from one natural deposit to another. The abrasive substances, referred to as gangue, may be separated by immersing the clay in water and allowing the non-colloidal particles to settle out.

The use of natural materials may be avoided by the preparation of synthetic clays or by the manufacture of synthetic zeolites. Synthetic clays are typically prepared by coprecipitation of silica and magnesia, drying the coprecipitated gel, mixing the resulting aerogel with an alkali metal base compound such as potassium or sodium hydroxide and heating the mixture for a period of ½ to 4 days at a temperature of from 150° C. to 400° C. under pressures of 200 to 1,000 pounds per square inch.

The clay greases comprise as their major ingredient an organic lubricating liquid which is preferably mineral lubricating oil and particularly petroleum lubricating oil but which may be any one of, or a mixture of, other oleaginous substances. These may be utilized in admixture with mineral lubricating oils, such as:

I. Synthetic lubricants produced by the Fischer-Tropsch, Synthol, Synthine and related processes, e. g.:
   - A. Polymerization of olefins such as ethylene, butylene, and the like, and their mixtures in presence of a Friedel-Crafts or other type condensation catalyst under elevated temperatures and pressures.
   - B. Polymerization of unsaturated hydrocarbons in presence of a catalyst and then condensing said polymerized product with an aromatic hydrocarbon such as xylol, benzol, naphthalene, etc.
   - C. Oxidation of polymerized olefins of lubricating range as noted under A and B.
   - D. Process of converting natural gas to carbon monoxide and hydrogen, followed by catalytic reaction under elevated temperature and pressure to produce hydrocarbons of lubricating range (Synthol process).

II. Synthetic lubricating products produced by the Bergius process, e. g., by:
   - A. Hydrogenation of coal, peat, and related carbonaceous materials under pressure and elevated temperature in presence of a catalyst.
   - B. Hydrogenation of asphalts, petroleum residues and the like.

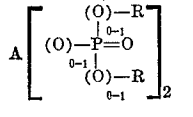
III. Synthetic lubricants produced by the Voltolization process, e. g., by:
   - A. Voltolization of fatty materials such as fatty oils.
   - B. Voltolization of mixtures of fatty oils and petroleum hydrocarbons.
   - C. Voltolization of unsaturated hydrocarbons, their mixtures, and the like.

IV. Organic synthetic lubricants:
   - A. Alkyl esters of organic acids, e. g.:
     - Alkyl lactates
     - Alkyl oxalates
     - Alkyl sebacates (2-ethylhexyl sebacate)
     - Alkyl adipates
     - Alkyl phthalates (dioctyl phthalate)
     - Alkyl ricinoleates (ethyl ricinoleate)
     - Alkyl benzoates
   - B. Alkyl, alkylaryl esters of inorganic acids, such as the phosphorus esters.

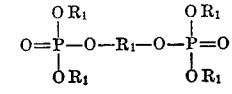
This particularly desirable class of oleaginous bases for the present compositions comprises organic phosphorus esters including phosphates, phosphonates, phosphinates, as well as the corresponding oxides. Typical species include:

Tricresyl phosphate
Trioctyl phosphate
Tributyl phosphate
Bis(3,5,5-trimethylhexyl) 2,4,4-trimethylpentene phosphonate
Tris(3,5,5-trimethylhexyl) phosphate
N-heptenyl bis(3-butylpentane) phosphinate
Bis(3,5,5 - trimethylhexane) octane phosphine oxide

Another highly desirable type of phosphorus lubricants includes the diphosphorus compounds including the four classes referred to above. Preferably, the diphosphorus compounds have a configuration as follows:



wherein A is an organic radical preferably aliphatic or aromatic hydrocarbon radical or alternatively an oxahydrocarbon radical or the corresponding sulfur, selenium or tellurium containing hydrocarbon radicals preferably saturated aliphatic hydrocarbon radicals having from 4 to 12 carbon atoms. The above configuration contemplates diphosphates, diphosphonates, diphosphinates and diphosphine oxides. A particularly desirable configuration comprises those diphosphates having the following configuration:



wherein each R_1 is an aliphatic hydrocarbon radical having from 2 to 6 carbon atoms. It has been found that lubricants of this particular configuration possess unexpectedly extreme low temperature operating characteristics. Species of such lubricants include:

1,4-butanediol bis(dibutyl phosphate)
1,3-propanediol bis(diamyl phosphate)

V. Synthetic lubricants made by polymerization of alkylene oxides and glycols at elevated temperatures in the presence of catalysts such as iodine, hydriodic acid, etc.:
- A. Polymers of alkylene glycol:
  - Trimethylene glycol
  - Propylene glycol
  - Tetramethylene glycol
  - Hexamethylene glycol
  - Pentamethylene glycol
- B. Copolymers of:
  - Trimethylene glycol and triethylene glycol
  - Trimethylene glycol and hexamethylene glycol
  - Trimethylene glycol and diethylene glycol
- C. Copolymers prepared from certain epoxides at elevated temperatures and in presence of KOH or BF_3-ether catalyst, e. g.:
  - Ethylene oxide and propylene oxide
  - Isobutylene oxide and propylene oxide
  - Epichlorohydrin and propylene oxide
- D. Sulfur containing polymers obtained by treating allyl alcohol, divinyl ether, diallyl ether, diallyl sulfide, dimethallyl ether, glycols, with H_2S in presence of a catalyst such as toluene sulfonic acid, peroxides, ultraviolet light, e. g.:
  - Dihydroxy diethyl sulfide
  - Dihydroxy dipropyl sulfide
  - Trimethylene glycol and dihydroxy dipropyl sulfide
  - Trimethylene glycol and dihydroxy diethyl sulfide

VI. Polymers obtained from oxygen-containing heterocyclic compounds, e. g., polymerization of tetrahydrofuran in the presence of a catalyst.

VII. Silicon polymers, e. g.:
- Polyalkyl siloxane and silicate polymers
- Alkylaryl siloxane and silicate polymers
- Dimethyl siloxane and silicate polymers, etc.

It has been found that the compositions of the present invention exhibit emulsification characteristics and provide greatly increased corrosion protection for bearings lubricated therewith. While the reasons for each of these phenomena are still to a large measure obscure, the following tentative theories may be advanced. However, it will be understood that applicant's invention is not predicated upon these theories but instead depends upon the exact constitution of these compositions rather than upon the theoretical reasons for their superiority.

Onium clay greases appear to swell in lubricating oils to a somewhat greater degree than do inorganic clays bearing absorbed cationic surface-active agents. While, at first, this may appear to be an economic advantage, the technical results are definitely detrimental. To obtain a grease of a given penetration or consistency, it is necessary to use a somewhat larger ratio of inorganic clay and absorbed amine than of onium clays. However, it has been found that greases gelled to a given consistency emulsify to a greater extent if the amount of solids content is relatively low. Hence, if gelling materials having a somewhat reduced capacity are employed (such as those of the present invention) it is possible to prepare greases having a somewhat higher solids content and, therefore, a correspondingly lower emulsifying tendency.

With respect to the corrosion characteristics of the present compositions, it is believed that the physically absorbed surface-active agents are less strongly bound to the colloidal clay particles than are the onium radicals, which are chemically bound in the onium clays. Hence, when such compositions are used for the lubrication of bearings or other metallic surfaces and water contaminates the lubricant, the surface-active material is more readily desorbed from the clay surface and, hence, is available for absorption on the metallic surface. Once the metallic surface has been effectively coated with at least a molecular layer of the cationic hydrophobic active agent, the surface then becomes water repellant and is effectively prevented from immediate contact with water. Moreover, since it is water repellant, it is also oleophilic and attracts a film of the lubricating oil thus enabling optimum lubricating operation.

The exact proportion of surface-active material to be used in the present compositions will vary with the surface area of the colloidal clay particles. Ordinarily, the colloidal particles suitable for the gelation of lubricating oils have surface areas varying from about 40 to 300 square meters per gram. It is preferred that the surface-active material be present in an amount to provide a mono-molecular layer on at least about 75% and, preferably at least 85% of this surface area. In terms of a weight relationship this usually amounts to a surface active agent weight ratio of between ¼ and 1 part for each part of colloidal clay, and optimum amounts are between about 40% and about 65% based on the weight of the clay.

The present compositions are preferably prepared by dispersing the clay in water to form a dilute hydrosol, allowing sufficient time (or utilizing centrifugal force) to separate the gangue, and subsequently adding the hydrophobic surface-active agent. As this material is absorbed on the particles of the clay, gelation results and the material may be readily filtered to produce a hydrogel of the clay bearing absorbed cationic material. This hydrogel is then mixed with the lubricating oil and preferably briefly milled to produce a fine dispersion of the hydrogel in the oil. The next step preferably comprises removal of water, under conditions of vacuum and/or high temperature. When the system has become dehydrated, so that less than about 1% of water remains, the composition resembles a slurry which is then subjected to milling or other shearing action until a grease structure is obtained.

The above technique for the preparation of greases is usually termed the "direct transfer process," since the water-wet gel is directly transferred into the lubricating oil without an intermediate drying period. However, if preferred, the clay bearing absorbed surface-active agent may be dried and subsequently incorporated in oil. Another process comprises the preparation of a clay hydrosol and incorporation thereof in a lubricating oil having dispersed therein the hydrophobic surface-active agent. Under these conditions, the clay hydrogel is formed in the

presence of the lubricating oil and is subsequently dehydrated under conditions similar to those described above.

Greases prepared according to the compositions of this invention have been compared with those produced by the onium clay technique. The examples which follow describe various greases prepared by both methods and the properties of these compositions:

Example I

An aqueous suspension of a magnesium montmorillonite containing approximately 2% of the latter is blended with octadecylamine to produce a hydrogel which is filtered to remove as much water as possible. The hydrogel is then mixed with a medium viscosity lubricating oil and dehydrated by heating to a temperature of 153° C. at atmospheric pressure for about 6 hours. The resulting dehydrated composition is passed through a paint mill for 5 times with small amounts of oil being added. The resulting grease will contain 5.5% solids, ⅓ of which is octadecylamine. The grease so prepared can be tested for its corrosion characteristics by operation in an automobile water bearing assembly. The conditions for this test comprise the following:

A tapered roller bearing assembly is operated at 1750 R. P. M. for 2.5 hours at 65–85° C. under a thrust load of 530 lbs. At 30-minute intervals 2 cc. of 0.05% NaCl solution is injected into the assembly. After this operation is completed, the assembly is examined for corrosion. The results of the tests are expressed in terms of arbitrary ratings from 0 to 10 wherein 0 denotes complete rusting of the bearings and 10 expresses perfect operation with substantially no rusting. Under these conditions the bearing lubricated with the above grease was given a rating of 8.

If a grease is prepared from the same clay by conversion to an onium clay using dimethyl dialkyl ammonium chloride (wherein the alkyl radicals comprise approximately 30% hexadecyl and 70% octadecyl), the resulting grease when tested in the same type of dynamic corrosion bearing will be given a rating of about 5.

Example II

A grease similar to that described above can be prepared by using a Wyoming bentonite having absorbed thereon 50% by weight of an amino amide prepared as follows:

A condensation product was prepared by reacting 10 moles of ammonia with 1 mole of epichlorohydrin at a temperature of about 40° C. for a period of 30 minutes. The reaction mixture was then heated with sodium hydroxide to convert all the chloride present in the reaction mixture to sodium chloride which was then separated from the product by centrifuging. The reaction mixture was then heated to a temperature of about 110° C. to remove the volatile constituents and to produce the condensation product. A typical product had a molecular weight of 105 and contained 43.1% carbon, 9.5% hydrogen and 21.2% nitrogen, the balance being oxygen and a small amount of impurities and had an equivalent weight of about 38 as a base. The condensation product was then heated at a temperature of 175–200° C. for a period of 1 hour with a mixture of tallow oil acids. These tallow oil acids are a mixture comprising primarily stearic, palmitic and oleic acids. Sufficient acid was employed to neutralize about ⅓ of the amino groups present in the condensation product. The resulting grease, when tested in the bearing corrosion test will be given a rating of 10.

A similar grease prepared from the same clay converted to an onium clay with dimethyl dioctadecylammonium chloride results in a formation of a grease having a rating of 4 in the above-described bearing corrosion test.

Example III

When a second magnesium montmorillonite (hectorite) is used in the formation of a grease having absorbed thereon the epichlorohydrin-ammonia condensation product partial amide described in the foregoing example, the bearing corrosion test rating is 10.

After completion of the test, the bearing is disassembled and left overnight in the presence of the water added during the test. After approximately 12 hours of exposure under these conditions, a rating of 10 will still be given to the bearing. A grease prepared from the same hectorite converted to an onium clay by the use of dimethyl dioctadecylammonium chloride has a rating of 4 in the bearing corrosion test. After standing overnight in the presence of the water added during the test, the bearing will be completly rusted and is given a rating of 0.

Example IV

A fourth clay, namely, Kinney clay (resembling a Wyoming bentonite) is utilized for a grease formation using the epichlorohydrin-ammonia condensation product described in Example II. The resulting grease, when tested in the bearing corrosion apparatus, is given the rating of 9. After standing overnight under static conditions, it can be given a rating of 8.

Example V

Many greases prepared with inorganic gelling agents progressively become degraded in their corrosion characteristics upon storage. It has been determined, however, that greases made according to the compositions of the present invention are stable to storage up to periods of at least 9 months. A grease was prepared substantially according to Example III above, utilizing hectorite having absorbed thereon 50% by weight of the epichlorohydrin-ammonia condensation product described in Example II. This grease was tested in the bearing corrosion apparatus periodically over periods up to 9 months without any change from the original rating of 10.

Example VI

A grease is prepared utilizing Wyoming bentonite having absorbed thereon approximately 60% by weight of an octadecylamine. When this grease is tested in the above-described bearing corrosion test, a rating of between about 7 and 10 will be obtained.

Example VII

If greases are prepared from hectorite, having absorbed thereon 70% of 2-heptadecyl, 4,4,6-trimethyl, 3,4,5,6-tetrahydropyrimidine, the resulting greases will be found to have high ratings, e. g., about 8 in the bearing corrosion test.

This is a continuation-in-part of application Serial Number 782,694, filed October 28, 1947, which issued May 22, 1951, as United States Patent 2,554,222.

I claim as my invention:

1. A bodied lubricant comprising a petroleum lubricating oil having colloidally dispersed there-

in a Wyoming bentonite, the colloidal particles of said bentonite having absorbed thereon from about 25% to about 100%, based on the weight of the bentonite, of a tall oil partial amide of a condensation product of epichlorohydrin and ammonia.

2. A bodied lubricant comprising a petroleum lubricating oil having dispersed therein a gelling amount of a magnesium bentonite, said bentonite having absorbed on the surfaces thereof, from about 25% to about 100%, based on the weight of the bentonite, of an aliphatic hydrophobic hydroxyamine.

3. A bodied lubricant comprising a petroleum lubricating oil having colloidally dispersed therein a gelling amount for hectorite, the particles thereof having absorbed thereon from about 25% to 100%, based on the weight of hectorite, of an aliphatic hydrophobic hydroxyamine.

4. A bodied lubricant comprising a petroleum lubricating oil having dispersed therein a gelling amount of colloidal clay particles, said clay having a base exchange capacity greater than about 60 milliequivalents per 100 grams and bearing absorbed thereon from about 25% to about 100% by weight of said clay of an aliphatic cationic hydrophobic surface-active agent, said agent containing nitrogen substantially only in the trivalent state.

5. A grease composition comprising a petroleum lubricating oil having colloidally dispersed therein a gelling amount of clay having a base exchange capacity greater than about 60, the colloidal particles of said clay having absorbed thereon from about 40% to about 65%, based on the weight of the clay, of an aliphatic cationic hydrophobic surface-active agent, said agent containing nitrogen substantially all of which is in the trivalent state.

6. A grease composition comprising a mineral lubricating oil having colloidally dispersed therein a gelling amount of a bentonite having absorbed on its surfaces from about 25% to about 100% by weight thereof of a cationic hydrophobic surface-active agent containing substantially only trivalent nitrogen, said agent having at least one aliphatic hydrocarbon chain of at least 10 carbon atoms.

7. A grease composition comprising a mineral lubricating oil having colloidally dispersed therein a bentonite, said bentonite having absorbed on the surfaces thereof from about 25% to about 100% by weight of said bentonite of an aliphatic hydrophobic hydroxyamine.

8. A bodied lubricant comprising a mineral lubricating oil having colloidally dispersed therein a gelling amount of a clay having a base exchange capacity greater than about 60, the colloidal particles of said clay having absorbed thereon from about 25% to about 100% by weight thereof of a hydrophobic aliphatic amine.

9. A grease composition comprising a mineral lubricating oil having colloidally dispersed therein a gelling amount of a bentonite, the colloidal particles of said bentonite having absorbed thereon a hydrophobic aliphatic amido amine.

10. A grease composition comprising a lubricating oil having colloidally dispersed therein a gelling amount of a bentonite, the colloidal particles thereof having absorbed thereon from about 25% to about 100%, based on the weight of said bentonite, of a hydrophobic aliphatic amido amine.

11. A bodied lubricant comprising a lubricating oil having colloidally dispersed therein a gelling amount of a clay having a base exchange capacity greater than about 60, the colloidal particles thereof having absorbed thereon a hydrophobic aliphatic cationic surface-active agent, the nitrogen atoms of said agent being substantially only in the trivalent state.

12. A grease composition comprising an organic lubricating liquid having colloidally dispersed therein a grease-forming amount of a clay having a base exchange capacity of at least 25, the colloidal particles of said clay having absorbed on the surfaces thereof from about 25% to about 100%, based on the weight of the clay, of a cationic hydrophobic surface-active agent, the nitrogen atoms of the said agent being substantially only in the trivalent state.

FRED H. STROSS.

REFERENCES CITED

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

UNITED STATES PATENTS

Number	Name	Date
2,531,440	Jordan	Nov. 28, 1950
2,554,222	Stross	May 22, 1951