

SUBSTITUTED THICKENER LUBRICATING GREASE

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2 SHEETS—SHEET 1

Figure-1

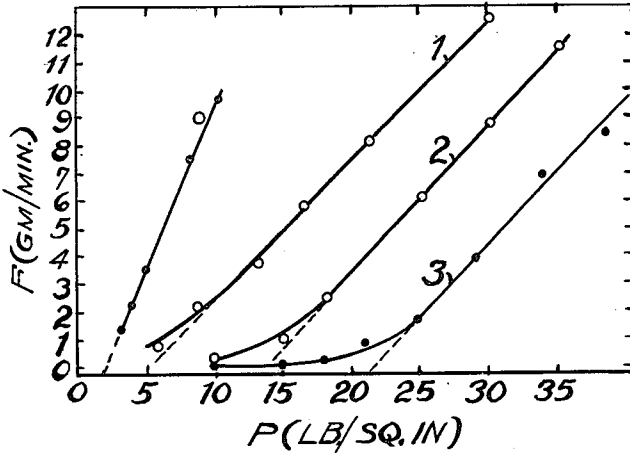


Figure-2

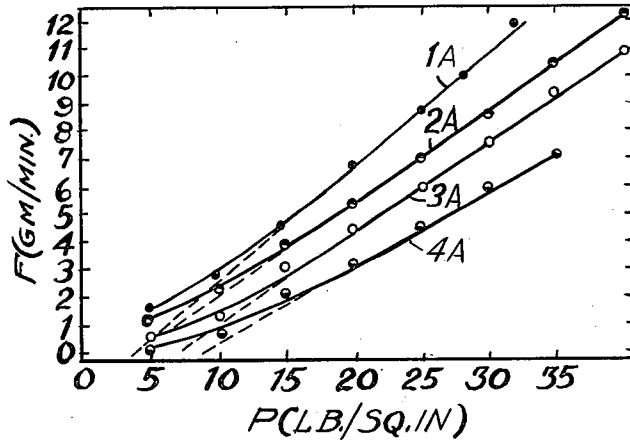
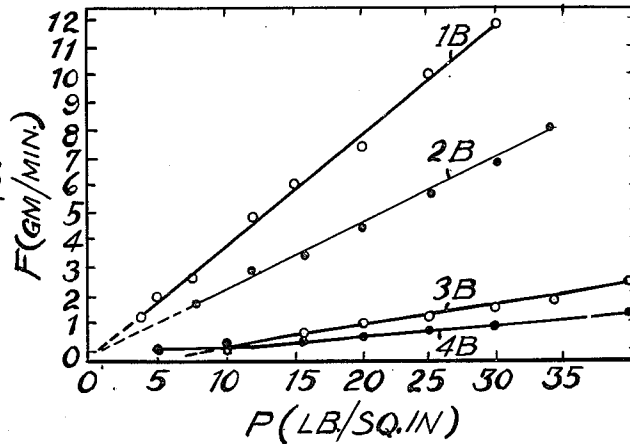


Figure-3



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Figure-4

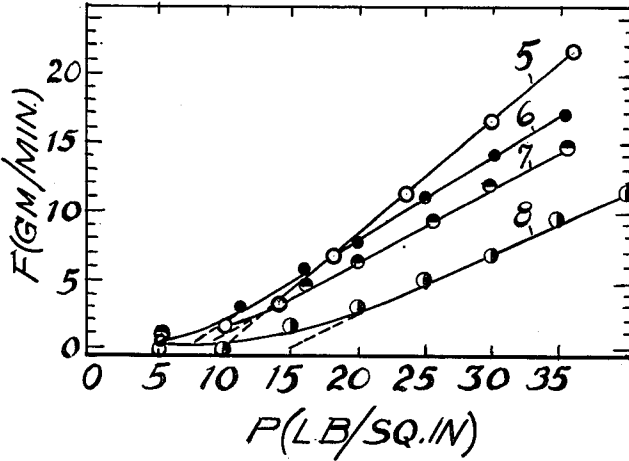
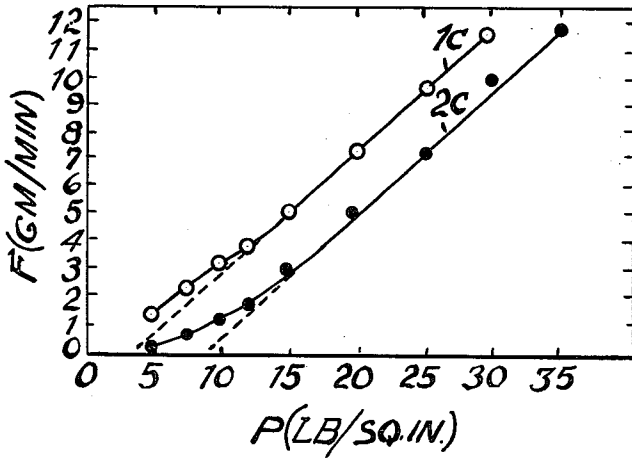


Figure-5



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UNITED STATES PATENT OFFICE

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SUBSTITUTED THICKENER LUBRICATING GREASE

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9 Claims. (Cl. 252-28)

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This invention relates to substituted thickener lubricating grease, and more particularly, to an improved lubricating grease wherein the thickening or grease structure forming material is of a new type, such as an aqueous gel, and preferably inorganic aqueous gel, origin.

In the prior art, lubricating greases have commonly been prepared by incorporating into mineral base lubricating oils of appropriate grade a gelling or thickening agent capable of setting up with lubricating oil into a grease-like structure. As a rule, the metal soaps of the fatty acids have been used almost universally as thickening agents. It has been suggested that various other materials than soaps, such as certain carbon blacks, bentonite, clay, aliphatic or aromatic sulfonates, and the like, might be substituted in whole or in part for the soap as a thickening agent. It has also been suggested that oils might be thickened to grease-like body by incorporating therein certain types of inorganic gels. The prior art has further suggested that the water of gelation in certain inorganic gels such as silica gel is preferably replaced without drying out the gel, since shrinkage results when such a gel is dried, making it extremely difficult to convert the gel into grease by substituting lubricant for the water. In other words, it is considered desirable, according to the prior art, to replace the original liquid continuous phase by another liquid which is more suitable for the intended use. This, it is suggested, may be accomplished by using a series of mutual solvents. For example, the prior art suggests that an aqueous silica gel may be used as an ingredient of a grease by first replacing the water by acetone, the acetone subsequently to be replaced with textile spirits, and the textile spirits finally replaced by a petroleum base lubricating oil. Such replacements may be accomplished by first extracting the liquid phase of the gel, i. e., water with one or more mutual solvents in series and then extracting the latter mutual solvent by contacting with an excess of the lubricant. The object of the present invention is to improve and extend the prior art methods and products by making possible, in some cases, a direct replacement of water with oil and also by stabilizing or otherwise improving various properties of the resulting product, such as its resistance to moisture, and the like.

During the course of experiments relating to this invention, it has been found that conventional colloidal suspensions of grease-forming particles, such as the soap particles used in the prior art, having a high ratio of length to breadth when recrystallized from oil. This means that they are long and slender, and relatively fibrous in character. This appears to be particularly true with the sodium and the calcium soaps that are widely used in greases. It has also been found that strong thixotropic properties are imparted to calcium soap-oil compositions by the addition of water in small quantities to such greases. As is well known in the prior art, it has been long considered necessary to add some water to the calcium soap greases to stabilize them.

While the exact bearing of the shape of the solid particles on the eventual viscosity of the colloidal composition is not known, it is believed that such shape is a factor of considerable importance in the formation of greases and grease-like compositions. For example, with respect to dispersions of particles approaching molecular sizes, Einstein's formula $n=2.5 C$ where n is specific viscosity and C is volume concentration, has been found to hold true for dilute solutions of molecules which are considered generally spherical in shape. This relationship changes to

$$n = \left(2.5 + \frac{f^2}{16} \right) C$$

where f is the ratio of length to width. Furthermore, where the particle is thought to be of disc-like shape, the formula applicable is

$$n = \left(\frac{4}{3} \frac{f}{\tan^{-1} f} \right) C$$

where f represents the ratio of diameter to thickness of the disc. The particles of colloidal size are of course much larger, but these formulae apply in a general way.

The above formulae suggest that particle shape is an important consideration when the purpose of the solid phase in a suspension is to change the flow characteristics of the liquid phase. It appears also that in some cases a small amount of water is a useful grease structure modifier. In conventional greases, it has been noted that there

is considerable difference in the penetration value of greases having similar soap content, depending upon the method by which the soap is introduced into the oil. This suggests that the fineness of subdivision as well as ultimate particle size of the solid phase is of considerable importance. Apparently the maximum thickening effect commonly occurs when the elemental particles of the solid are in the colloidal size range. Hence, it might be inferred from the preceding that the most desirable particle to be used as the solid phase in preparing lubricating grease may be compared to a flexible rod-like structure of essentially one dimensional character or an infinitely thin disc. The maximum thickening effect appears to be produced by thin elongated or flattened elements.

For complete control of grease structure the fiber length of the solid phase should be controlled as far as possible. The solid phase should also have a high melting point and as far as practicable it should be hydrophobic to give water resistance to the composition. At the same time it should attract and hold oil to its surface so as to prevent separation of the oil phase. Commonly available inorganic materials such as silica gel, for example, are deficient in the latter property and it is an object of the present invention to overcome such deficiency.

The present invention involves, as one aspect, the principle that solids having the general characteristics mentioned above may be prepared by two general methods to control particle size. The particles may be finely divided and subdivided, as by grinding or other means of comminution, or they may be prepared by building up to the desired size from the molecular sizes. It is frequently difficult, however, to obtain the particular range of sizes desired. For example, wood pulp when suspended in water may be ball-milled to a particle size approaching that of soap crystals in conventional soda base grease, but ball-milling of this material in oil is practically impossible. The transfer of a finely comminuted material from one medium to another also is attended with difficulties. As suggested above, in connection with silica gel, the removal of water from a suspension at pressures and temperatures below critical permits the solid phase to shrink badly due to the collapse of capillary spaces by the surface tension of the evaporating water. As a result, the solid becomes irreversibly coagulated and the whole desired effect of the comminution is lost. Hence, the procedure mentioned above of replacing the liquid phase without evaporation or drying appears to be essential in the use of many materials of the aqueous gel type or analogous materials as grease thickeners.

The thixotropic structure which appears to be necessary for satisfactory lubricating greases appears to result from the greater adhesional forces between discrete particles of the solid phase than between elements of the liquid and solid phase. These forces can be completely destroyed in many cases by contaminating the surfaces of the solid particles. In other cases these forces can be substantially increased by proper choice of contaminants. For example, water may be used for increasing such adhesion, whereas fatty acids added to titanium oxide produce the opposite effect. An aspect of the present invention is a means of controlling the forces mentioned, although the exact degree of control is not specifically determinable.

This property of thixotropy is dependent ap-

parently on the mutual dispersion of two separate phases into each other and is not simply a function of particle shape of the normally solid phase. Particle shape becomes of great importance, however, when it is desired to keep the total quantity or volume of solid low with substantial thickening effect. In other words, a high degree of mutual dispersion of liquid phase and the solid material is essential.

Hence particles having the greatest degree of aniso-dimensionality are desirable. Such substances as ball-milled sand, finely divided common salt, and the like possess good thixotropic properties but their particle shapes are such that very substantial quantities are required for a desired thickening effect. On the other hand, relatively long fibered materials such as ball-milled asbestos, wood pulp, and the like are quite satisfactory in thickening power, smaller relative quantities being required. Hence the criteria for suitable thickening agents depend in part upon their particle shape and in part upon the forces of attraction involved, although of course their relative affinities for oil, their chemical and abrasive properties, and the like, are also of great importance.

The inorganic gels such as silica gel, and analogous voluminous precipitates of inorganic nature appear to be particularly desirable. The finely divided precipitated materials such as silica gel, $\text{Fe}(\text{OH})_3$, $\text{Al}(\text{OH})_3$, etc., appear to have the desired particle shape, the requisite affinity for oil, and low abrasive properties. Organic materials such as cellulosic fibers and viscose, and metallo-organic materials such as the soaps are satisfactory. Many inorganic materials such as talc, bentonite, titanium oxide, carbon black, even graphite, and the like, have some utility, provided they can be processed so as to hold the oil in the grease structure and resist its displacement by non-lubricating fluids, especially by water. The latter is an important aspect of the present invention and is accomplished in many cases by providing a suitable coating for the solid phase at the dineric interface.

It is another object of the present invention to make possible the utilization of various inorganic or naturally occurring materials as the thickening or grease-forming ingredients of lubricating greases, so as to produce solid or semi-solid lubricants or greases having desirable properties for specific uses. Some of these products appear to be quite useful under certain unusual conditions, i. e., of temperature, etc., where conventional soap-thickened greases would not be usable.

A still further object of this invention is to improve methods for replacing the normal liquid phase such as water of gelation, or equivalent fluid, in inorganic gels and the like, with a lubricating material such as mineral lubricating oil to prepare a grease or grease-like material. In some cases, such replacement may be accomplished by a single step and in others by a plurality of successive steps, but the present invention contemplates the replacement by either one or more steps.

In one aspect of the present process, the liquid phase of an appropriate gel, preferably an inorganic gel such as silica aqua gels or a precipitate such as aluminum hydroxide is extracted continuously with acetone or some other volatile liquid which is mutually soluble in the liquid medium of the gel and in lubricating oil. This part of the process, which is analogous to that

described by Kistler in U. S. Patent No. 2,260,625, is continued until the original liquid phase, normally water, has been completely or very nearly completely displaced by the solvent. The resulting gel, containing a new liquid phase, is then dispersed in a proper amount of lubricating oil and the mutual solvent, which is preferably volatile, is then flashed off. In the present case, however, the processing preferably includes the provision of a water-repellent coating for the solid phase at the dineric interface. The resulting grease may be passed through a colloid mill to give it the final desired stable structure. By this method, greases may be successfully prepared from an inorganic gel like silica gel, or from other voluminous precipitates such as aluminum hydroxide, ferric hydroxide, or from certain other naturally occurring materials, such as wet finely divided mica, bentonite, cellulose, and asbestos. In general, the concentration of the solid phase in the final product will range between 5 and 20% by weight, based on the total composition.

An alternative method of substituting the continuous liquid phase in inorganic gels or voluminous precipitates consists in freezing the gel and subliming out the solvent at a very low pressure. This procedure obviates the shrinkage due to surface tension at the liquid interface, but for reasonable efficiency the solvent should have a high sublimation pressure and a low heat of sublimation. A similar result may be obtained by removing the liquid phase at its critical temperature and pressure. These considerations limit the solvents which may be used. The following examples will more fully illustrate the invention and make clear its application to lubricating greases.

In the attached drawings there are shown graphically in Figures 1 to 5 the pressure vs. flow characteristics of some of the grease or grease-like compositions of the following examples.

Example 1

Using a silica aqua gel, a grease was produced by exchanging the water in the aqua gel with ordinary ethyl alcohol and then heating in an autoclave at a pressure higher than the critical pressure to a temperature above the critical temperature of the alcohol (about 245° C. at 63 atmospheres), pumping off the alcohol vapor and dispersing the aerogel so produced in mineral base lubricating oil. Stable greases can be prepared by this method, using as little as 1.7 to 3.7% by volume of the aerogel. The particular gels have a structure of silica fibers which apparently is something like that of felt and they have a very high specific surface of about 600 square meters per gram. Oil is added directly to the gel and the mixture is milled to produce the grease structure.

Example 2

Results similar to those of Example 1 may be obtained by heating silica aqua gel in an autoclave above the critical temperature for water. The water vapor is pumped off and this process results in a precipitation of silica from solution at the critical temperature of the water. A finely divided silica resulted which could be dispersed to thicken the oil, although a true gel does not result. Comparable greases might also be obtained without going through the aerogel state. Thus the aqua gel may be

extracted with acetone and the acetone replaced by mineral oil, flashing off the solvent to form a true gel grease. Alternatively, the aqua gel may be first extracted with acetone, the acetone followed by petroleum ether, and thereafter the mineral oil introduced to replace the petroleum ether. Thus, the process may be carried out in two, three, or even more steps of substitution.

The grease produced according to the above Examples 1 and 2 are substantially transparent, with strongly thixotropic properties. Their flow properties are shown graphically in Figure 1 of the drawings. The steepest of these curves indicated at 0 shows an unthickened oil of 300 S. S. U. at 100° F. viscosity and a viscosity index of 40 flowing through a .78 millimeter diameter orifice, under the pressures indicated. The second curve, indicated at 1, shows the flow characteristics of a grease based on the same oil but containing 5% by weight of the aerogel. The curves indicated at 2 and 3 show the results of greases using 7½% and 10% respectively of the same aerogel in same oil. Viscosity measurements were carried out at 25° C. It will be noted that the yield value or the thickening effect of the gel decreased with decreasing concentration of aerogel without great change in mobility.

In the case of conventional greases, sodium stearate base greases, for example, it is commonly desirable to use oils of low viscosity index. With silica gel thickened greases, such as those of Examples 1 and 2, the greases have no true melting point and are not affected by the viscosity index of the oil. Hence oils of high viscosity index may be used. This ordinarily is not true of soap thickened greases. Inspection of silica gel base grease with respect to abrasion indicates that the material is quite free of abrasive properties.

As previously suggested, natural silica gel greases are not entirely satisfactory for some purposes, especially where appreciable quantities of moisture are present, because the hydrophilic surface of the silica preferentially absorbs water which under moist or humid conditions will gradually displace the oil. Thereafter, on evaporation of the water the silica will shrink to a point where it becomes useless as an oil thickener. Hence it is usually quite necessary to increase the water repellency of greases of this character.

Such water-proofing may be accomplished to a reasonable degree by providing a water-proofing coating of the solid phase at the dineric interface. This may be done by a thermal, vapor phase polymerization of butadiene and also of styrene on the surface of the silica aerogel preferably before the oil is incorporated therein. For example, gaseous styrene or butadiene may be passed over the dry aerogel in a heated tube and this has been accomplished experimentally. The silica gel products obtained in both cases were rather greasy to the touch and when mixed with oil imparted good water repellent qualities to the grease. Tests indicated that this repellency continued when 20 grams of grease were added to 200 cc. of water at 70° C. and stirred mechanically for 15 minutes. Qualitatively, some thixotropy appeared to be lost in this mixing.

It is not always necessary to pretreat the inorganic gel. Monomeric styrene was polymerized directly on to the surface of the silica gel by adding 2% of styrene to the oil before incorporating the silica gel and thereafter placing the grease prepared therefrom in an oven at

100° C. for two days. The resulting product was considerably darker than the original oil but it had excellent water resistance without too much loss in thixotropy. Better results were obtained by adding ¼ to 2% of polystyrene dissolved in chloroform, directly to the grease, followed by flashing off the chloroform. The latter experiments seem to indicate that both monomeric and polymeric styrene are adsorbed by silica, although this is not absolutely certain.

A silicone type of water-proofing material may also be used, giving excellent water-proofing but lowering the thixotropic properties considerably. In general, a compressed monolayer of a water repellent is desirable on the silica surface to give protection without substantially interfering with oil adsorption.

Aluminum stearate also may be used as the water-proofing agent. This material may be added in small quantities, 0.25 to 1%, for example, based on the dry weight of the silica gel to give good water resistance, although it commonly results in some loss in thixotropy.

Figure 2 of the drawing shows the flow-pressure relationship of various greases, using an oil of 300 S. S. U. viscosity at 100° F. and a viscosity index of 40. In the case of Figure 2, commercial aerogel was used as thickening agent in quantities of 10%, based on the weight of the total composition. Curve 1A shows the flow properties of grease water-proofed with silicone type water-proofing agent; curves 2A and 3A show respectively results of using 0.5% and 0.125% of aluminum stearate in the same grease; curve 4A shows the flow properties of the untreated grease. For some purposes, greases of the general character described above may be prepared by using a small quantity of a hygroscopic agent of low volatility, such as glycerol, to prevent coagulation as the water is withdrawn from the gel. The quantity of glycerol required is of the order of about 1% to 2% by weight, based on the final grease product.

Example 3

Another inorganic material of thickening power approximately equal to that of silica is aluminum hydroxide. This material is somewhat deficient in water resistance, but not as objectionable in this respect as silica. It has been found more difficult to waterproof greases thickened with aluminum hydroxide than silica. Small quantities of stearic acid, for example 0.25% to 2.0%, based on the weight of the total composition, appear to improve water resistance of aluminum hydroxide greases, probably due to the formation of minute quantities of aluminum stearate at active centers, probably over the dineric surface of the solid phase. The other water - proofing agents, such as polymerized styrene mentioned above, appear also to be applicable to aluminum hydroxide base grease, also reaction products of polybasic acids and polyhydric alcohols.

Example 4

Ferric hydroxide may be used as a thickener in much the same manner as aluminum hydroxide except that a higher volume concentration is required, due apparently to the fact that ferric hydroxide exists in the form of small discs or plates rather than rods. When this material is used, it appears to be necessary to completely remove the water before flashing off the solvent since otherwise the whole system

undergoes a change in phase and the mass becomes liquid.

Example 5

Natural fibers of cellulose, collagen and asbestos, when reduced sufficiently in size by wet ball-milling followed by solvent exchange to remove the water and replace it with oil, produce greases of good appearance at low concentrations of thickener. Ball-milling over extended periods tends to shorten such fibers considerably which is objectionable. It is preferable to use other methods which will not unduly break up the fibers since the ratio of fiber length to diameter is of some importance as indicated above. Water resistant properties may be imparted in the same general manner as described above in connection with Examples 1 to 3.

Example 6

Viscose precipitated with sulfuric acid, washed and solvent exchanged as described above, produces a reasonably good grease with low cellulose concentration. The particles, however, tend to have too small a length to width ratio. Hence the grease is not of particularly good stability. A material of elongated fibrous structure in the solid phase is preferable.

Example 7

An alcogel of calcium acetate produced a good grease after oil was substituted by solvent exchange. The weight of dry gel constituted about 6% of the final product. The needle-shaped crystals had a tendency to grow, however, with the result that the grease lost consistency on stirring. The experiment shows, however, that the principle of solvent exchange is applicable to soap thickened greases.

Example 8

An aqua gel of sodium stearate was solvent exchanged without heating and incorporated into lubricating oil in a 5% concentration by weight. The mutual solvent employed was acetone. The resulting product was approximately equivalent in consistency to a high melting soda soap grease having a soap concentration of 15% by weight.

Various precipitates of voluminous type appear to have certain utility in the general process described above. Thus, certain volcanic or other colloidal clays, such as bentonite and related materials, have the property of swelling in water to produce a heavy paste of low solids concentration. Some of these materials are not satisfactory for making lubricating greases because they show shrinkage when attempts are made to exchange the solvent. Apparently a three-dimensional structure does not exist in greases produced from these disc-like or plate-like materials to the same extent as with rod-like structures and to that extent they are less satisfactory.

Certain materials, such as suzorite and vermiculite, two minerals of the mica type, gave fairly good greases after extensive wet ball-milling and solvent exchange. A variety of other materials which appear to have possibilities are lignin, graphite, talc, magnesium oxide, calcium carbonate, ethyl cellulose, sodium carboxy-methyl cellulose, Irish moss, silt and sodium algenate. The greases listed in the table are exemplary.

Table

Solid phase	Per Cent Conc. (volume)	Per Cent Conc. (weight)	Treatment
Commercial volclay	7.8	17	Swollen in water, then solvent exchanged.
Suzorite	11.2	29	Ball-milled, then solvent exchanged.
Vermiculite	8.2	23	Do.
Asbestos	9.5	23	Do.
Do	5.6	15	Ball-milled 16 days, then solvent exchanged.
Fe(OH) ₃	6.5	22	ppt. Then solvent exchanged.
Fe(OH) ₃	4.7	17	Do.
Al(OH) ₃	3.3	8.4	Do.
Al(OH) ₃	3.6	9.1	Do.
SiO ₂	1.7	4.9	aqua gel made, then solvent exchanged.
Commercial aerogel	1.8	5.0	oil added directly.
Do	2.7	7.5	
Do	3.7	10	
Na Stearate	5.0	5	aqua gel made, then solvent exchanged.
Ca Acetate	4.1	6.1	alcogel made, then solvent exchanged.
Wood pulp	4.6	7	ball-milled, then solvent exchanged.
Collagen	7	9	Do.
NaCl	29.7	50	ppt. in acetone, then dried at 105.

These greases were all made using an oil of 300 S. U. S. viscosity at 100° F. and 40 V. I.

Figure 3 of the attached drawings shows flow-pressure relationships for suspensions of carbon black and cuprene in a 300 S. U. S. viscosity oil (210° F.) having a viscosity index of 40. A large change in mobility with relatively small changes in thickening effect is shown by the convergency of the several graphs 1B to 4B in this figure. Figure 4 shows flow-pressure relationships of various greases, that of curve 5 being prepared with 15% asbestos ball-milled for 16 days. The grease of curve 6 contained 17% of ferric hydroxide, Fe(OH)₃. That of curve 7 contained 9% of the aerogel referred to in Examples 1 to 3 and that of curve 8 contained 9% of aluminum hydroxide (Al(OH)₃ precipitate.) A superior yield value or thickening effect of the aluminum hydroxide should be noted.

Figure 5 shows the flow-pressure relationship for silica aerogel base greases, 10% concentration, by dry weight of gel, based on the total composition in oil of 300 S. U. S. viscosity at 210° F. and viscosity index of 95. The upper curve 1C of this figure represents the grease treated with 1% of polystyrene whereas the lower curve 2C represents the untreated grease.

It will be apparent from the foregoing that the present invention contemplates broadly the use of various substitutes for soap as oil-thickening agents for greases wherein the lubricating oil is incorporated by substitution or by solvent displacement of the original liquid phase. The invention contemplates particularly the waterproofing of such materials at the dineric interface so that oil incorporated into the grease will not readily be separated. In general, it appears to be most desirable to use a material of relatively long fibrous or thin plate-like structure and in concentrations of 1 to 20% by dry weight, based on the finished lubricant, and in most cases this material should be waterproofed.

As a rule, the grease will contain 0.05 to about 7%, preferably 0.1 to 1% by weight, based on the total composition, of a waterproofing material such as silicone polymers, alkyd resins, aluminum stearate, hydrocarbon polymers such as polystyrene and the like. Specific compositions containing silica-type polymers and alkyd resins are disclosed and claimed in our copending applications, Serial Nos. 66,131 and 66,132, filed December 13, 1948.

By "grease" is meant a solid or semi-solid composition containing available lubricating oil for

efficient lubrication, thickened sufficiently to prevent substantial flow or loss from bearings, gears and the like. Such lubricant "greases" or grease-like compositions while intended for lubrication primarily, may be useful for other purposes. They may, if desired, contain various modifiers, such as oxidation inhibitors, tackiness agents, extreme pressure additives, surface active agents and the like, as will be understood by those skilled in the art. The mineral base lubricating oils, as recited in some of the claims, may include such modifiers. The invention, however, is not necessarily limited to mineral base oils, since other oils of the natural or synthetic ester type, such as the alkyl esters of dibasic aliphatic acids, e. g., di-2-ethyl hexyl sebacate, and the like, as well as the oily polyglycols, and the like, may be used in lieu of mineral base oil for many purposes. For some purposes, such may be superior, as for extreme low temperature lubrication, for example.

What is claimed is:

1. A lubricating composition consisting essentially of mineral base lubricating oil thickened to a grease-like consistency by 1 to 20% by weight, based on the total composition, of an inorganic oxygen containing gel voluminous precipitate having its normal liquid content replaced by said lubricating oil, and 0.5 to 2% of a polystyrene water-proofing agent covering said precipitate in a compressed substantially monolayer coating to inhibit moisture absorption by said precipitate.
2. A composition consisting essentially of mineral base lubricating oil thickened to a grease-like consistency by 1 to 20% by dry weight, based on the total composition, of an inorganic gel of the group which consists of silica gel, aluminum hydroxide and ferric hydroxide having its normal water content substantially entirely replaced by said lubricating oil, and 0.05 to 2% of a polystyrene waterproofing agent covering said gel in a compressed substantially monolayer coating to inhibit reabsorption of moisture by said gel.
3. Composition according to claim 2 wherein said gel is silica gel.
4. Composition according to claim 2 wherein said gel is silica aerogel.
5. Composition according to claim 1 wherein said precipitate is a silica gel.
6. Composition according to claim 1 wherein said precipitate is an alumina hydroxide gel.
7. Composition according to claim 1 wherein said precipitate is a ferric hydroxide gel.
8. A lubricating grease composition consisting

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essentially of mineral base lubricating oil thickened to a grease-like consistency with 1 to 20% by dry weight, based on the total composition, of finely divided silica gel having its normal water content replaced by said oil, said gel being water-proofed by adding 0.05 to 2% by weight, based on the total composition, of a polystyrene waterproofing agent covering said gel in a compressed substantially monolayer coating.

9. A lubricating composition consisting essentially of mineral base lubricating oil thickened to a grease-like consistency by 7½ to 10% by dry weight, based on the total composition, of silica gel, and 0.1 to 1% of a polystyrene waterproofing

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agent adsorbed to said gel in a compressed thin layer.

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REFERENCES CITED

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

UNITED STATES PATENTS

Number	Name	Date
2,194,683	Badollet	Mar. 26, 1940
2,251,093	Williams	July 29, 1941
2,260,625	Kistler	Oct. 28, 1941