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### 2,820,006

# PROCESS FOR THE PREPARATION OF GREASE COMPOSITIONS

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This invention relates to a novel process for the preparation of thickened lubricating oil compositions, especially lubricating greases, which are gelled with inorganic colloids.

Lubricating greases are mixtures of a liquid oleaginous medium containing a sufficient amount of a colloidal gel to impart a semi-solid structure thereto. The normal gelling agents use din such greases are soaps, but a new type of lubricating grease, particularly useful at elevated temperatures, has recently been developed, wherein the gelling agent is an inorganic colloid. Several processes have been described for the production of these inorganic gel greases, the most effective of which are the so-called "aerogel process," the "alcogel process," and the "directtransfer process." In the aerogel process, the hydrogel is dehydrated by solvent displacement with a miscible low boiling liquid, such as acetone. The resulting organogel then is heated under pressure to a temperature above the critical temperature of the liquid which is then flashed off, leaving the highly expanded aerogel. The latter is then dispersed in oil to form a grease. The alcogel process involves displacing all of the water from an inorganic hydrogel by an organic solvent, such as an alcohol, mixing the resulting organogel with a lubricating oil, removing the organic solvent, and, if necessary, milling the product to produce a grease structure. The alcogel process enables the formation of gels without any substantial decrease in the gel skeleton. Generally, a water-proofing agent is added to some stage of the process. In the directtransfer process, an inorganic hydrogel is incorporated directly into a lubricating oil in the presence of a surfaceactive agent, and the water removed from the resulting mixture, for example, by evaporation. In this case also it may be necessary to mill the product to produce a grease

The aerogel process is expensive in equipment requirements, while the alcogel process requires excessive amounts of alcohol for complete dehydration. While the direct transfer process is an advance over the aerogel and alcogel processes from a processing point of view, it suffers from the disadvantage that the products tend to lose their mechanical stability on prolonged use at relatively high temperatures. Thus, at 150° C., greases made by the direct transfer process lose their mechanical stability after only about 24 to 48 hours' use.

Now, in accordance with this invention, a process has been developed, which, while retaining many of the advantages of the direct transfer process, results in lubricating greases having the property of retaining their mechanical stability at high temperatures. The lubricating greases made by the process of the present invention are able to retain their mechanical stability for longer periods and at higher temperatures than similar greases made by the direct transfer process.

According to the present invention, thickened lubricating oils and lubricating greases are prepared by a proc-

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ess which comprises mixing a hydrogel of an inorganic colloid with a lubricating oil, a hydrophobic surface-active agent, and such a quantity of a mutual solvent for the lubricating oil and water that a homogeneous liquid phase is obtained when the mixture is heated, if necessary under pressure, subjecting the mixture to such conditions of temperature and pressure that said single homogeneous liquid phase is obtained, and distilling off the water, and, if desired the mutual solvent, while maintaining said liq-10 uid phase homogeneous. The essential feature of this process is the step of ensuring that the three liquids present in the mixture are in the form of a single liquid phase while the water is removed. Surprisingly, this step is essential to produce products with the desired high mechanical 15 stability at high temperatures. Thus, it has already been proposed to make inorganic gel greases by mixing a hydrogel and alcohol with a lubricating oil and removing the water by azeotropic distillation, and finally distilling any remaining alcohol; but, if such a process is carried out without taking steps to ensure that the liquids are in a single liquid phase during the distillation of the water, the resulting products are no more mechanically stable at high temperatures than if they were made by the ordinary direct transfer process.

One result obtained by the use of the above process which has not been elucidated at the present time, comprises the unexpected and substantial increase in the surface area of the gels so prepared. It has been found that the gels prepared by the subject "uniphase" process have a surface area nearly double that of gels prepared by the ordinary direct-transfer process. In spite of this, and still more unexpected, is the fact that the grease can be made hydrophobic by the use of substantially smaller ratios of hydrophobic surface-active agents than are required for greases prepared by the ordinary direct-transfer process or the ordinary solvent-transfer (alcogel) process. Another unexpected feature comprises the improved corrosion characteristics of these greases as compared with similar compositions prepared by earlier proc-While the corrosion characteristics of microgel greases can be, in general, improved by the presence of magnesia, sodium nitrite, or other substances, it has been found unnecessary to resort to the use of these additives in the present instance.

The process of the present invent involves the following steps:

(A) Preparation of the hydrogel of the inorganic colloid and breaking it up into a size suitable for handling and washing to remove salts formed during the preparation, if desired, followed by partial removal of water. The present invention is not concerned with this step which may be carried out by any of the known methods, some of which have been mentioned above.

(B) Mixing the hydrogel with the mutual solvent, the lubricating oil, and a surface-active agent.

(C) Distilling off the water and, if desired, the mutual solvent while maintaining the liquid ingredients as a single phase.

(D) Generally, milling or shearing of the resulting product to impart a grease structure.

As regards step (B) it is preferred to mix the hydrogel with the mutual solvent, and, if necessary, the surface active agent before adding the lubricating oil. The amount of mutual solvent to be added will vary over a considerable range depending on the water content of the hydrogel, the proportion of inorganic colloid desired in the finished grease and the mutual solvebility of the mutual solvent, lubricating oil and water. If the latter is not known from the appropriate miscibility curves it can easily be determined on a laboratory scale by examining mixtures of the mutual solvent, lubricating oil and water.

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For example, if a grease is to be made using isopropyl alcohol as the mutual solvent and di-(2-ethylhexyl)sebacate as the lubricating oil, a series of water solutions in isopropyl alcohol can be made up and these mixed with the sebacate in say a weight ratio of 1 part of alcohol solution to 2 parts of sebacate. Each of the mixtures can then be heated to boiling with continuous stirring and the changes in the phase systems observed. If a clear uniphase system is obtained on boiling, that mixture is suitable for application in the present process. Thus with 10 the ingredients mentioned above, it has been found that provided the water content of the water-isopropyl alcohol solution is not more than 20% by weight uniphase solutions are obtained on boiling the mixture and are therefore suitable for use in the present invention. In making the above test it may in some cases be necessary to adjust the pressure in order to determine the optimum conditions for mutual solubility at the boiling point in which case, of course, the optimum pressure found will be employed in applying that mixture to the grease manufacture proper.

It is preferred to mix the mutual solvent with the hydrogel in stages, stirring well between each addition to allow diffusion equilibrium to be reached. If necessary, after each of the stages supernatant liquid can be decanted in order to reduce the total bulk of liquid to be handled in the process. When the necessary quantity of mutual solvent has been incorporated into the hydrogel, the lubricating oil is stirred in with the surface-active agent, if one is being used. Also at this stage any other ingredients of the grease, such as an antioxidant, may be added. The mixing with the lubricating oil may conveniently be carried out in the vessel in which the distillation is to be effected.

As regards step (C), this may be carried out in any suitable distillation apparatus as long as it is provided with means for the application of heat, maintenance of the desired pressure, introduction of the various components and the removal and condensation of water and the mutual solvent vapors. Ordinary distillation stills connected to water condensers may be readily adapted for this purpose. This distillation may be continued until all of the water and mutual solvent has been removed or it may be stopped when the water content of the mixture is at a suitable low figure. Thus, quantities of up to 1.0% by weight of water (when the grease may be regarded as substantially anhydrous) may be left in some lubricating greases prepared by the process of this invention.

Following the distillation step, it is generally desirable to subject the dehydrated composition to a milling or 50 shearing action in order to produce a satisfactory lubricating grease structure. The mixture left as a residue from the distillation is generally in the form of a slurry having the consistency of a heavy oil. This may be subjected to shearing action by forcing it through small orifices under 55 high pressure. Such orifices preferably have diameters of 0.01 to 0.025 inch and are 3 to 12 inches long while the pressure is generally 5000 to 12,000 lbs. per square inch. However, an alternative piece of equipment, which may be employed, is a homogenizer such as that available on the market bearing the trademark "Manton Gaulin" homogenizer. The use of the latter type of apparatus in conjunction with the described orifice tubes substantially reduces the amount of recycling necessary to obtain a lubricating grease with the desired minimum penetration. Moreover, the amount of gelling agents required to produce a lubricating grease of a given penetration is reduced by approximately 15% when using the Manton Gaulin homogenizer as compared with the orifice-pump method. It is preferable to mill or shear the mixture at a temperature of 20° to 55° C. In addition to the above, other homogenizers or paint mills may be utilized. The above conditions apply particularly to greases gelled with amorphous colloids. Clay greases usually require homogenizing but ordinarily do not need milling.

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The process of the present invention may be carried out with a wide variety of ingredients. The oleaginous media to be used are substantially water-immiscible oleaginous materials, which may be of synthetic or natural origin or mixtures thereof. The preferred type of lubricating oil is a mineral oil having a viscosity of at least that of an SAE-10 lubricating oil, but for high temperature purposes heavier oils are desirable. A particularly suitable type of mineral lubricating oil for use in high temperature greases is that known as "bright stock." Other oleaginous media may be present either as the sole lubricating oil or in admixture with a mineral lubricating oil. Especially useful oils for this purpose are the esters of phosphorus acids, such as trioctyl phosphate, and di-(2-ethylhexyl) hexanephosphonate. Other suitable lubricating oils are the esters of aliphatic dicarboxylic acids wherein the alcohol radical contains an aliphatic hydrocarbon chain having between 4 and 12 carbon atoms and the dicarboxylic acid radical contains from 6 to 12 carbon 20 atoms. Suitable examples of such esters are di(2-ethylhexyl) sebacate and di(isooctyl) adipate. Polymeric oxyalkylene compounds, such as polypropylene oxide and its copolymers with ethylene oxide, and polymers of glycols, such as polymers of trimethylene glycol, are also suitable, as are the high boiling liquid organic polymers containing silicon, such as the liquid polymethylphenyl siloxanes and polymethyl siloxanes.

Inorganic colloids suitable for use in this invention must be capable of existing in hydrogel form. They can broadly be divided into two main classes, namely, crystalline materials and those having amorphous structures. The crystalline materials are principally the synthetic or natural clays. Preferably they are those clays known as "swelling clays" and of these the montmorillonites are especially useful. Two specific clays which are especially useful are Wyoming bentonite and hectorite. The latter material, possibly due to its high magnesium content, has been found to produce lubricating greases having exceptionally satisfactory anticorrosion characteristics. The amorphous inorganic colloids include various metallic oxides and hydroxides, e. g., silica aluminum hydroxide, magnesium hydroxide; sulphides such as zinc sulphide; silicates, sulphates and chlorides or mixtures of such substances. Silica is especially useful due to its low cost and ready availability. Alkaline earth metal hydroxides, e. g., calcium hydroxide, are also useful, especially when mixed with silica.

It is preferred that the hydrogel used as a starting material in the process of the present invention contains between about 0.5% and about 10% by weight of the inorganic colloid. The best results are obtained, however, when the hydrogel contains between about 1% and about 5% by weight of the inorganic colloid. Using silica as a specific example of the materials contemplated, such a hydrogel may be produced by addition of an acid to a sodium silicate solution or, conversely, by the addition of sodium silicate solution to an acid. For example, a commercial sodium silicate solution may be acidified with dilute sulphuric acid to form a hydrosol, which in a short time converts to a hydrogel containing appreciable amounts of sodium sulphate. This is normally washed by decantation and usually requires two or more changes of water to eliminate the substantial amounts of sodium sulphate present. However, filtration methods may be resorted to. or the hydrosol may be contacted with ion exchange resins to remove the salt. It will be understood that all of the hydrogels used in the present process have been washed substantially free of water soluble inorganic salts (e. g. sodium chloride or sulfate), and that the other ingredients of the greases are substantially salt free as well.

The mutual solvent for the lubricating oil and water may be any liquid capable of yielding a uniphase liquid system with the lubricating oil and water under some suitable conditions of proportions, temperature and pressure.

These conditions will clearly vary with the nature of the

particular ingredients employed and only illustrative examples of such conditions need be set down in this specification. However, the proportions, temperature and pressure necessary for any particular set of ingredients can be determined by preliminary test or by study of the appropriate miscibility curves.

The degree of miscibility which the mutual solvent has with the lubricating oil and water is preferably sufficiently high as to avoid the necessity of using a large proportion of the mutual solvent to reach the desired uniphase condi- 10 tions. If the mutual solvent used is of the type which does not form an azeotropic mixture with the water, it will be advisable to use one having a boiling point higher than that of water. If however, the mutual solvent is of the type which does form an azeotropic mixture with the 15 water then one having a boiling point below that of water can be used. A mutual solvent which does not possess a boiling point higher than water and does not form an azeotropic mixture with the water may be used in some cases but it necessitates the use of a large proportion thereof in order to ensure the removal of substantially all of the water. Unless the mutual solvent is to be left in the finished lubricating grease it should have a boiling point substantially lower than that of the lubricating oil. It is sometimes convenient to use a mutual solvent which is only slightly miscible with water at low temperatures, since then the distillate comprising water and the mutual solvent will be in the form of two phases, thus facilitating solvent recovery.

Examples of mutual solvents which may be used in the 30 process of the invention are alcohols, such as ethyl alcohol, the propyl alcohols, the butyl alcohols, the amyl alcohols, diacetone alcohol, cyclohexanol and methylcyclohexanol; ketones, such as diethyl ketone, methyl ethyl ketone, methyl isobutyl ketone, di-isobutyl ketone 35 and dihexyl ketone; ethers, such as the di-isopropyl, methyl tertiary butyl, methyl tertiary amyl, dihexyl, dioctyl and diphenyl ethers and dioxane; esters, such as ethyl acetate, methyl propionate, ethyl isobutyrate and ethyl acetoacetate; diols, such as propylene, butylene and hexylene 40 glycols, diglycols, such as dipropylene and dibutylene glycols; glycol monoalkyl ethers, such as the mono-methyl, -ethyl, -propyl and -butyl ethers of ethylene or propylene glycol, glycerine mono- or di-alkyl ethers, such as monoor di-propyl glycerine; phenol, the cresols, amines and heterocyclic bases, such as the alkanolamines, pyridine and morpholine. Mixtures of such mutual solvents also may be employed. Preferably the mutual solvents have from 2 to 12 carbon atoms per molecule.

In the process of the present inveniton it is preferred 50 to incorporate an oil-soluble surface-active agent when mixing the hydrogel, lubricating oil and mutual solvent prior to the distillation step. The said surface-active agent can be incorporated in any one or more of the hydrogel, lubricating oil or mutual solvent prior to mixing them 55 or it can be added separately. In many cases the presence of a surface-active agent, particularly a cationic surfaceactive agent, is necessary to maintain the inorganic colloid in the form of a hydrogel. For example, when making a clay hydrogel, the surface active agent used for this purpose serves also to stabilize the finished lubricating grease against the deleterious effects of water.

Surface-active agents used in the process of the present invention may be cationic, anionic or non-ionic in character. Preferably they are cationic materials. Generally in order that it may be sufficiently oil-soluble, the surfaceactive agent should contain at least 12 carbon atoms per molecule and preferably should contain at least one hydrocarbon radical per molecule having at least 12 carbon 70 atoms. Suitable cationic materials include the "onium" compounds, such as ammonium, phosphonium, sulphonium and arsonium compounds. The ammonium compounds include salts of aliphatic, alicyclic, aromatic and heterocyclic amines including primary, secondary and 75 volume being that of the kilogram to the litre.

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tertiary amines and polyamines, as well as quarternary ammonium compounds. Other suitable ammonium salts are those of inorganic acids, such as octadecyl heptadecyl ammonium bromide of tridodecyl ammonium chloride, and those of organic acids, such as heptadecyl ammonium acetate. Illustrative examples of other classes of suitable onium compounds are triphenyl alkyl phosphonium or stibonium halides and dialkyl or diaryl phosphonium or sulphonium halides.

Other suitable cationic materials are the aliphatic longchain amines having 12 or more carbon atoms, such as octadecylamine, and the long-chain polyamines. Amidoamines such as the monoamides of diethylene triamine and triethylene tetramine, are particularly effective. Complex substances may be employed, such as the partial amides formed from higher fatty acids or rosin acids with aliphatic polyamino-hydroxy compounds, such as the condensation products of halohydrins, such as epichlorhydrin, with ammonia or a primary or secondary amine. Preferred products are the condensation products of epichlorhydrin and ammonia, converted to partial amides having between 1/4 and 3/4 of their amino groups in the amide form, by reaction with higher fatty acids or rosin type acids, such as stearic acid or the acids derived from tall oil or the animal or vegetable fats and oils. Similar useful products may be obtained by condensation of acrolein and ammonia followed by partial amide formation.

In addition to the cationic surface-active agents, anionic materials may be employed, such as fatty acids and hydroxy-substituted fatty acids having at least 12, and preferably, at least 16 carbon atoms, per molecule, such as stearic acid and its homologues and 12-hydroxystearic acid. The acids may be used as such or in the form of their soaps, preferably polyvalent metal soaps and, still more preferably, amphoteric metal soaps. Preferred soaps are aluminum 12-hydroxystearate, lead 12-hydroxystearate and calcium naphthenate. The above and other suitable hydrophobic surface-active agents are disclosed in the following U. S. patents: Stross 2,554,222; Stross, 2,584,-085; Stross, 2,573,650; Abrams, 2,599,683; Peterson, 2,623,852; Stross, 2,625,508; Stross, 2,623,853; Jordan, 2,531,440; Sirianni, 2,583,603, 2,583,605.

In order to provide the finished lubricating grease with a satisfactory resistance to water, the surface-active agent should generally be present in an amount of at least 25% by weight of the inorganic colloid. The proportion of surface-active agent employed is dependent upon the particular colloid employed, the particular surface-active agent employed, and the degree of water resistance required. Generally, the surface-active agent is used in an amount up to about 100% by the weight of the colloid.

If the surface-active agent is added to the hydrogel before the latter is mixed with the lubricating oil and mutual solvent it is useful to heat the hydrogel and surface-active agent together at a temperature above 60° C. for a period of at least 30 minutes. This results in a hydrogel wherein the surface-active agent is evenly distributed on the surface of the gel particles, which leads to floccu-60 lation of the gel particles and facilitates sedimentation. The water that separates is removed from the hydrogel before incorporation thereof in the lubricating oil and mutual solvent, thereby shortening the period necessary for the removal of water by distillation.

While the chief importance of the present invention lies in its application to the production of lubricating greases, it is also applicable to the production of lubricating oil compositions thickened, but not gelled, with inorganic colloids. Specific examples of such compositions are the oil base drilling muds containing inorganic gels.

The following examples illustrate the invention, the parts referred to being parts by weight except where otherwise stated, the relation of parts by weight to parts by

6000 parts by volume of an aqueous sodium silicate solution containing 750 parts of SiO<sub>2</sub> were added to 10,200 parts by volume of 0.86 N sulphuric acid with agitation. In about two minutes the mixture set to a soft gel having a pH 6.0 and containing 5% by weight of SiO<sub>2</sub>. The gel was washed free of salts by repeated slurrying with water and filtration. When the gel was salt free, isopropyl alcohol was added in stages, with stirring between each addition so that diffusion equilibrium is reached prior to decantation of supernatant liquid, until the 89.2% by weight of the water originally present in the gel had been replaced by isopropyl alcohol.

The lubricating oil employed was di(2-ethylhexyl)- 15 sebacate. In this was dissolved 2% by weight of an oilsoluble surface active agent made by condensing epichlorhydrin with aqueous ammonia and converting the product into a partial amide by reaction with an amount of commercial stearic acid equivalent to one third of its basicity. There was also dissolved in the lubricating oil 0.1% of phenyl-beta-naphthylamine to act as antioxidant. This doped lubricating oil was mixed with the silica gel-waterisopropyl alcohol mixture, in the proportion of 95.3 parts of the oil for each 4.6 parts of silica in the silica gel-waterisopropyl alcohol mixture. It had previously been determined that the water, isopropyl alcohol and oil in the resulting mixture gave a uniphase system at room temperature and remained uniphase on boiling.

The mixture of hydrogel, isopropyl alcohol and doped lubricating oil prepared as above was then subjected to distillation until the isopropyl alcohol was removed, when the water content was just under 0.1% by weight. a pressure of about 10,000 lbs. per square inch.

The product was an excellent lubricating grease having a silica content of 4.6% by weight and a water content of 0.07% by weight. It was extremely stable at high tem-  $_{40}$ peratures, retaining its consistency after being subjected to 150° C. for 120 hours.

## Example II

A silica hydrogel was prepared and washed as in Example I. 94.4% by weight of the water in the gel was replaced by isopropyl alcohol in the manner described in Example I.

The lubricating oil employed was medicinal paraffin containing the same additives as described in Example I in the proportion stated therein. This oil was mixed with the silica gel-water-isopropyl alcohol mixture, in the proportion of 95.8 parts of the oil for each 4.2 parts of silica in the said mixture. It had previously been determined that the water, isopropyl alcohol and oil in the resulting mixture gave a uniphase system at about 90° C. and remained uniphase on boiling.

The mixture of hydrogel, isopropyl alcohol and doped lubricating oil prepared as above was then subjected to distillation until all the water and isopropyl alcohol had been removed and the residual mixture was sheared as described in Example I.

The product was an excellent lubricating grease having 65 a silica content of 4.2% by weight and substantially no water. It was extremely stable at high temperatures, retaining its consistency after being subjected to 150° C. for 120 hours.

Grease compositions containing the same proportions 70 of the same ingredients described above but prepared by the direct-transfer process retained their original consistency at 150° C. for only about 24 hours after which the latter greases commenced to become progressively softer

Two greases were prepared, one by the process described in Example I, using 45% based on the weight of the silicia of a 1/3 amide formed between stearic acid and tetraethylene pentamine as the hydrophobic surface-active agent. The second grease contained the same ingredients and proportions but was prepared by the direct-transfer process. It was found that the grease prepared by the process of the present invention provided substantially complete protection against corrosion of metallic parts lubricated therewith as compared with only partial protection provided by the direct-transfer grease.

We claim as our invention:

1. In the process for the formation of grease compositions, wherein a hydrogel of an inorganic grease-forming colloid is mixed with a water immiscible lubricating oil of the group consisting of hydrocarbon oils and aliphatic esters of dicarboxylic acids and a hydrophobing proportion between 25% and 100%, based on the colloid, of a hydrophobic cationic surface-active agent, water is removed therefrom and the remaining substantially anhydrous composition is homogenized, whereby a grease composition is formed, the improvement which comprises admixing with said oil and hydrogel a sufficient amount of a lower mono-hydric alcohol mutual solvent with the oil and water so as to maintain a single liquid phase at least at the temperature of water removal and until substantially all of the water and mutual solvent have been removed, the initial weight ratio of water to solvent being between about 5.6:94.4 and about 20:100.

2. In the process for the formation of grease compositions wherein a silica hydrogel is mixed with a water immiscible aliphatic ester of a dicarboxylic acid and beresidual mixture was sheared by being forced three times at tween about 25% and about 100% by weight based on the through a capillary 6" long and 0.02" in diameter under silica of a hydrophobic cationic surface-active agent water silica of a hydrophobic cationic surface-active agent, water is removed therefrom and the remaining substantially anhydrous composition is homogenized, whereby a grease composition is formed, the improvement which comprises admixing with said diester and hydrogel a sufficient amount of a lower monohydric alcohol mutual solvent for the diester and water so as to maintain a single liquid phase at least at the boiling point of water and until substantially all of the alcohol and water have been removed, the initial weight ratio of water to solvent being between about 5.6:94.4 and about 20:100.

3. In the process for the formation of grease compositions wherein a water-insoluble metal oxide hydrogel is mixed with a water-immiscible lubricating oil of the group consisting of hydrocarbon oils and aliphatic esters of dicarboxylic acids and between 25% and 100% by weight based on the oxide of a hydrophobic cationic surface active agent, water is removed therefrom and the remaining substantially anhydrous composition is homogenized, whereby a grease composition is formed, the improvement which comprises admixing with said oil and hydrogel a sufficient amount of an alcohol, said alcohol having 2-12 carbon atoms per molecule and being a mutual solvent for the oil and water so as to maintain a single liquid phase at least at the boiling point of water and until substantially all of the alcohol and water have been removed, the initial weight ratio of water to alcohol being between about 5.6:94.4 and about 20:100.

4. In the process for the formation of grease compositions wherein a silica hydrogel is mixed with a water immiscible mineral oil and between about 25% and about 100% by weight based on the silica of a hydrophobic cationic surface-active agent, water is removed therefrom and the remaining substantially anhydrous composition is homogenized, whereby a grease composition is formed, the improvement which comprises admixing with said mineral oil and hydrogel a sufficient amount of a lower monohydric alcohol mutual solvent for the mineral oil and water so as to maintain a single liquid phase at least at the 75 boiling point of water and until substantially all of the

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alcohol and water have been removed, the initial weight ratio of water to solvent being between about 5.6:94.4 and about 20:100.

5. A process according to claim 3 wherein the metal oxide is an amorphous colloid.

6. A process according to claim 3 wherein the metal oxide is silica.

7. A process according to claim 3 wherein the water immiscible lubricating oil is an aliphatic ester of a dicarboxylic acid.

8. A process according to claim 3 wherein the mutual solvent is a monohydric aliphatic alcohol having between 2 and 12 carbon atoms per molecule.

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