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CORROSION RESISTANT GREASE COMPOSITIONS

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This application is a continuation-in-part of my co-pending application Serial No. 227,531, filed October 1, 1962.

This invention relates to lubricating grease compositions suitable for use over a wide range of operating conditions. In one of its aspects, the invention relates to lubricating grease compositions containing certain materials which exhibit corrosion or rust-inhibiting characteristics when incorporated in these greases. Still more particularly, in this aspect, the invention relates to lubricating grease compositions exhibiting the aforementioned corrosion inhibiting characteristics and to a method for their preparation.

Grease compositions, in general, comprise a lubricating vehicle, thickened to a grease consistency with gelling or grease-forming agents, which may comprise metallic salts and soaps of organic acids of varying molecular weights present in various compositions thereof. These grease compositions, depending upon the environment in which they are employed, may be continuously or intermittently subjected to humid or wet atmospheric conditions, where moisture, even when present in relatively minor amounts, results in the corrosion of the metallic surface being lubricated. In an attempt to minimize or to avoid these corrosion phenomena, various means have been investigated which include the incorporation of corrosion or rust inhibiting materials into the grease compositions themselves.

Among the corrosion inhibiting materials that have been suggested for incorporation into grease compositions, are the water soluble nitrites or chromates, which are well known for their corrosion inhibiting properties. In particular, they comprise the alkali metal nitrites or chromates, and may also include the amine and ammonium nitrites and chromates. Of particular importance is sodium nitrite, which has been employed in various grease compositions in amounts varying from about 0.1 to about 1 percent by weight of the total soap. This material is generally employed in the form of an aqueous solution, although it has also been utilized in substantially anhydrous greases, as well. It has been found, however, that in order to make this material commercially feasible for use as a corrosion inhibitor in greases, it is necessary that the size of the dispersed sodium nitrite crystals be maintained below approximately 5 microns, in order to minimize abrasion of the metallic surfaces being lubricated by the grease. In this respect, it has been found that in practicing the method of the prior art in which sodium nitrite solutions are added to the grease during processing, and followed by the removal of water by dehydration, there results the formation of sodium nitrite crystals which are considerably larger in size than 5 microns. To overcome this undesirable tendency of large sodium nitrite crystal-formation, various methods have been resorted to, which have included high temperature spray-drying of sodium nitrite oil and water emulsions, or ball milling of dry sodium nitrite dispersed in oil or in grease, in order to maintain the sodium nitrite crystals at a small size. These methods have not, however, proven to be completely satisfactory. Thus, prior to the present invention, no effective and relatively simple method has been proposed for the incorporation of sodium nitrite, as a corrosion inhibiting agent, into grease compositions,

in which these crystals are present in relatively small sizes.

It is, therefore, an object of the present invention to provide an improved grease composition.

Another object of the invention is to provide grease compositions having improved corrosion-inhibiting properties, and wherein metallic surfaces being lubricated with grease containing them are not significantly abraded.

A still further object of the invention is to provide improved grease compositions having the aforementioned characteristics, and a method for their preparation.

Other objects and advantages inherent in the invention will become apparent to those skilled in the art from the following disclosure.

It has now been found that greases containing the improved corrosion-inhibiting compositions hereinafter discussed have overcome the disadvantages heretofore encountered in commercial applications, resulting in obtaining a grease of improved corrosion-inhibiting properties, and in which the corrosion-inhibiting agent itself does not impose any significant degree of abrasion upon the surfaces being lubricated. In general, the improved corrosion-inhibitor of the present invention comprises an alkali metal nitrite, a lubricating vehicle, and a thickening or gelling agent, and which, when prepared in accordance with the improved method of the invention, results in obtaining corrosion-inhibiting compositions in which the alkali metal nitrite crystals are present in a relatively small average size (i.e., below approximately 5 microns), so that the desirable corrosion inhibiting properties are attained by reason of their presence, and yet are too small in size to cause any significant degree of abrasion upon the metallic surfaces which are lubricated with greases containing them.

Insofar as the components comprising the corrosion-inhibitor are concerned, the alkali metal nitrites that may be employed include sodium nitrite, potassium nitrite and lithium nitrite. Of these sodium nitrite is preferred. The alkali metal nitrite is employed in the inhibitor composition in an amount from about 5 to about 60 percent by weight, preferably in an amount from about 10 to about 50 percent by weight, and comprises from about 0.05 percent to about 2 percent by weight, of the total grease composition.

The lubricating vehicle employed in the inhibitor composition may comprise any of the conventional hydrocarbon oils of lubricating viscosity and may include mineral or synthetic lubricating oils, aliphatic phosphates, esters and diesters, silicates, siloxanes and oxalkyl ethers and esters. Mineral lubricating oils, which are preferred as the lubricating vehicle, may be of any suitable lubricating viscosity ranging from about 45 SSU at 100° F., to about 2000 SSU at 100° F. and preferably from about 50 to about 250 SSU at 210° F. These oils may have viscosity indexes, varying from below 0 to about 100 or higher. Viscosity indexes from about 70 to about 95 are preferred. The average molecular weights of these oils range from about 250 to about 800. This lubricating vehicle is employed in the inhibitor in an amount sufficient to constitute the balance of the inhibitor composition, after accounting for the desired specific quantities of alkali metal nitrate, and thickening agents, in a given instance.

The thickening or gelling agent employed in the inhibitor composition comprises an essentially hydrophobic clay. This thickening agent may be prepared from a clay which is initially hydrophilic in character, but which has been converted into a hydrophobic condition by the introduction of long chain hydrocarbon radicals into the surface of the clay particles, prior to its use as a component of the inhibitor composition, as, for example, by being subjected to a preliminary treatment with organic cationic surface active agents, such as onium compounds. This method of conversion, being well known to those skilled

in the art, is believed to require no further discussion, and does not form a part of the present invention.

More specifically, the clays which are useful as starting materials in forming the thickening agents to be employed in the inhibitor composition, may comprise the naturally occurring chemically unmodified clays. These clays are crystalline complex silicates, the exact composition of which is not subject to precise description, since they vary widely from one natural source to another. These clays may be described as complex inorganic silicates such as aluminum silicates, magnesium silicates, barium silicates, and the like, containing, in addition to the silicate lattice, varying amounts of cation-exchangeable groups generally regarded as metallic oxide radicals. Hydrophilic clays which are particularly useful for conversion to the thickening agents of the present corrosion-inhibitor include montmorillonite clays, e.g. bentonite, attapulgite, hectorite, illite, saponite, sepiolite, biotite, vermiculite, zeolite clays, and the like. The thickening agent is employed in the inhibitor composition in an amount from about 0.5 percent to about 30 percent by weight, and preferably from about 3 percent to about 15 percent by weight, and comprises from about .02 percent to about 2.0 percent of the total grease composition, by weight.

The preparation of the improved inhibitor composition, in accordance with the process of the present invention, comprises, in general, preparing a mixture in the form of a dispersion of the lubricating vehicle and the thickening agent; preparing an aqueous mixture of the alkali metal nitrite and water; combining the two mixtures, forming an emulsion; and then susbtantially dehydrating the resulting combined mixture to produce the desired inhibitor composition, in which form it is ready for incorporation into specific lubricating grease compositions.

In preparing the mixture or dispersion of the lubricating vehicle and the thickening agent, an open grease kettle is preferably employed, into which the required quantities of lubricating vehicle and thickening agent are charged. The aqueous mixture of the alkali metal nitrite and water, which is also charged to the open grease kettle, is prepared by first dissolving the alkali metal nitrite in water, in such quantities as to obtain a mixture of desired concentration of these components. The combined mixture thus formed is heated to a temperature below about 212° F., and preferably to a temperature between about 175° F. and about 205° F. to insure a uniform dispersion of water and oil components. The above mentioned combined mixtures are then homogenized at elevated pressure, and the kettle is maintained at a temperature not higher than about 212° F. for a sufficient time to permit thorough admixture of the components. Thereafter, in order to effect dehydration of the combined material, the kettle temperature is gradually increased to a temperature from about 290° F., to about 300° F.

The following examples will serve to illustrate the preparation of typical improved corrosion-inhibitor compositions of the present invention, and their incorporation and performance in grease compositions.

Example 1

A dispersion of finely divided sodium nitrite in Bentone, a hydrophobic clay comprising a montmorillonite clay exchanged with an amine, as the thickening agent, was prepared employing a mineral oil as the liquid vehicle. These components were employed, in producing the desired dispersion as described below, in the following quantities.

	Percent by Weight	Pounds
Bentone	4.39	1.80
Mineral Oil, 75" at 100° F	73.41	30.14
Sodium nitrite	22.20	9.13

In preparing the above dispersion, an open kettle was charged with 30.14 pounds of the mineral oil. To the oil were then added 1.8 pounds of commercial Bentone, while recycling through a Manton-Gaulin homogenizer at a pressure of about 3000 p.s.i. 9.13 pounds of sodium nitrite were next separately dissolved in 8 pounds of water. Solution of the sodium nitrite was effected by heating this mixture to a temperature of 180° F. This aqueous solution of sodium nitrite was then added to the Bentone dispersion in the mineral oil in the kettle, and the combined mixture in the kettle was homogenized, employing a Manton-Gaulin homogenizer, at a pressure of about 3000 to about 3500 p.s.i. The kettle was heated and the temperature was controlled at approximately 205° F. for a period of 30 minutes, while homogenizing was taking place. After this time, the kettle temperature was gradually increased to a temperature of approximately 290° F., at which point the material was found to be substantially dry. Examination of the crystal size of the dispersed sodium nitrite particles in the thus prepared composition, showed an average particle size of less than 3 microns.

Example 2

A dispersion of finely divided sodium nitrite in a Bentone-thickened grease, as the thickening agent was prepared employing a mineral oil as the lubricating vehicle. These components were employed, in producing the desired dispersion as described below, in the following quantities.

	Percent by Weight	Pounds
Bentone-thickened grease	38.90	15.97
Mineral oil, 75" at 100° F	38.90	15.97
Sodium nitrite	22.20	9.13

In preparing the above dispersion, an open kettle was charged with 15.97 pounds of the Bentone-thickening grease, and 15.97 of the mineral oil. This mixture was recycled through a Manton-Gaulin homogenizer, operating at a pressure of about 3000 to about 3500 p.s.i. until a homogeneous gel had formed. This gel was heated to a temperature of about 180° F. and at this temperature 9.13 pounds of sodium nitrite dissolved in 8 pounds of water was added. This mixture was next dehydrated while homogenizing by heating to a temperature of approximately 300° F. After dehydration had taken place, examination of the crystal size of the dispersed sodium nitrite particles in the thus prepared composition, showed an average particle size of less than one micron.

Grease compositions, containing the above described corrosion inhibitors of the present invention, when subjected to a bearing test, conducted in accordance with the specifications of ASTM D1743–60T or by the CRCL–41 test, reveal a rating of No. 1, indicating the absence of any corrosive effect upon the bearing surfaces being treated. Thus, as indicated above, the improved corrosion-inhibitor compositions of the present invention may be incorporated in many grease compositions and have a wide variety of industrial applications.

As previously indicated it is necessary that the thickening agent of the novel inhibitor composition comprises an essentially hydrophobic clay and that this clay can be prepared from a clay which is initially hydrophilic in character. The use of hydrophilic clays, on the other hand, does not result in obtaining the improved inhibitor compositions of the present invention in which the nitrite particle size is below 5 microns. To illustrate this criticality, an attempt was made to employ commercially available bentonite, a hydrophilic clay, as the thickening agent, as shown in the following example.

Example 3

An attempt was made to form a dispersion of finely divided sodium nitrite in bentonite, as the thickening agent, and employing a mineral oil as the lubricating vehicle. These components were employed in the following approximate quantities.

	Percent by Weight	Grams
Bentonite	4.39	43.9
Mineral oil, 75″ at 100° F	73.41	734.1
Sodium nitrite	22.20	222.0

In preparing the above composition, a dispersion was attempted to be made by mixing 43.9 grams of bentonite with approximately 734.1 grams of the mineral oil, by passing the mixture through a homogenizer, in six tries. No gel was obtained. To 387.3 grams of this mixture a solution comprising approximately 111 grams of sodium nitrite in 100 grams of water was added. This mixture was stirred and then passed through the homogenizer for another six times. After homogenization had taken place, it was found that the bentonite had absorbed the aqueous solution of sodium nitrite, forming a paste or a gel. The mineral oil, however, had separated out to form a distinct layer.

It will also be understood that by following the procedure such as illustrated by the above Examples 1, 2 and 3 other alkali metal nitrites may be substituted for the sodium nitrite employed in the present corrosion-inhibitor composition. Similarly, other lubricating vehicles, dispersing and thickening agents, as disclosed above, may be also substituted in these compositions, in various combinations, if so desired. It will also be understood that any of the corrosion-inhibitor compositions prepared in accordance with the process of the present invention, may be incorporated in a wide variety of grease compositions, in addition to those described in the examples.

While preferred embodiments of the compositions of the present invention and the process for their preparation have been described for the purpose of illustration, it should be understood that various modifications and adaptations thereof, which will be obvious to those skilled in the art, may be made without departing from the spirit of the invention.

I claim:

1. A metal soap-thickened grease composition containing a corrosion inhibitor, said inhibitor comprising from about 5 to about 60 percent, by weight, of an alkali metal nitrite having an average particle size below about 5 microns; from about 0.5 to about 30 percent, by weight, of a thickening agent comprising an essentially hydrophobic clay; and an oil of lubricating viscosity as a vehicle.

2. A metal soap-thickened grease composition containing a corrosion inhibitor, said inhibitor comprising from about 10 to about 50 percent, by weight, of an alkali metal nitrite having an average particle size below about 5 microns; from about 3 to about 15 percent, by weight, of a thickening agent comprising an essentially hydrophobic clay, and an oil of lubricating viscosity as a vehicle.

3. A metal soap-thickened grease composition containing a corrosion inhibitor, said inhibitor comprising from about 5 to about 60 percent, by weight, of an alkali metal nitrite having an average particle size below about 5 microns; from about 0.5 to about 30 percent, by weight, of a thickening agent comprising an amine-exchanged montmorillonite clay, and an oil of lubricating viscosity as a vehicle.

4. A metal soap-thickened grease composition containing a corrosion inhibitor, said inhibitor comprising from about 10 to about 50 percent, by weight, of sodium nitrite having an average particle size below about 5 microns; from about 3 to about 15 percent, by weight, of a thickening agent comprising an amine-exchanged montmorillonite clay; and an oil of lubricating viscosity as a vehicle.

5. A metal soap-thickened grease composition containing a corrosion inhibitor, said inhibitor comprising, by weight, a major proportion of an alkali metal nitrite having an average particle size below about 5 microns; a minor proportion of a thickening agent comprising an essentially hydrophobic clay, and an oil of lubricating viscosity as a vehicle.

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DANIEL E. WYMAN, *Primary Examiner.*

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,227,651 January 4, 1966

Bertil A. Peterson

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 61, for "nitrate" read -- nitrite --; column 3, line 31, for "susbtantially" read -- substantially --.

Signed and sealed this 13th day of December 1966.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents

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