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PETROLEUM LUBRICATING JELLY

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This invention relates to improved petroleum lubricating jellies and, in particular, to thixotropic petroleum lubricating jellies having high dropping points, low running torques, and low oil separation characteristics.

In certain types of lubrication there are encountered conditions under which neither greases nor liquid oils are entirely satisfactory lubricants. For example, in the lubrication of ball bearings and other open bearing surfaces, particularly in relatively fine and intricate mechanisms such as cash registers and measuring instruments including airplane instruments, it is desirable that the lubricant be of such consistency that it will remain between the working surfaces and not be lost by dripping or leakage. At the same time, it is desirable that the lubricant be of relatively low viscosity under operating conditions, so as not to impede the operation of the mechanism. Liquid oil is not suitable because it will leak, while most greases are too viscous.

For this type of lubrication, petroleum jellies having thixotropic properties, that is, of jelly-like consistency when quiescent and liquid when agitated, have been recommended. These thixotropic petroleum jellies are mixtures of lubricating oils with waxes having melting points above about 120° F. and generally have a soap incorporated therein. These compositions have proved to be satisfactory lubricants under most conditions: however, they are subject to the disadvantage that they have relatively low dropping points and are rather unstable with respect to oil separation characteristics.

It is therefore an object achieved by this invention to provide thixotropic petroleum jellies having high dropping points and low oil separation characteristics.

This and other objects achieved by this invention will become apparent in the following detailed description thereof.

I have discovered that when a compound of a bentonite with an organic base is incorporated in thixotropic mixtures of wax and oil in such amount that the ratio of bentonite compound to wax is substantially less than 1:1, compositions result which while retaining their thixotropic properties have considerably higher dropping points and lower oil separation characteristics than similar soap-containing compositions. This

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combination of properties makes these compositions particularly valuable in certain types of lubrication such, for example, as in the lubrication of aircraft instruments and control bearings. Because of their thixotropic properties combined with high dropping point and low oil separation, these compositions are better retained in the bearings at elevated temperatures; and because of their relative softness at low temperatures, as indicated by their high penetration and low running torque at low temperatures, they function satisfactorily as lubricants over the broad temperature range to which such bearings are subjected.

In preparing the improved thixotropic jellies of my invention, wax of high melting point, advantageously wax substantially free from oil and having an ASTM wax melting point above about 120° F., is used. Ceresin wax such as is obtained from ozokerite having an ASTM wax melting point of about 160° F. has proved particularly satisfactory although other waxes may be used, such as beeswax, paraffin wax, petroleum ceresin, or refined rod wax, montan wax, shellac wax, or mixtures of these waxes with oil-free petroleum waxes having melting points above about 120° F. The amount of wax used may be varied widely, depending on the particular wax used and the properties desired in the lubricant. In general, the amount of wax used should be sufficient to provide an oil-wax mixture having a cloud point above about 110° F. In order to obtain this cloud point, at least about 4.0 per cent by weight of wax having a melting point above about 120° F. is usually required. Generally, it is not necessary to employ more than about 28 per cent by weight of wax.

The oil used in forming these thixotropic jellies may vary widely in properties. In general, substantially any oil properly designated as a lubricating oil may be used, depending upon its intended application. Thus, oils having viscosities as low as 50 SUS and as high as 4000 SUS at 100° F. may be used under appropriate circumstances. The viscosity index of the oil may vary from —50 to 125 or more. In general, however, I have found oils having viscosity indexes above about 90 are most advantageous. The percentage of oil in the jelly may be varied to regulate the temperature-consistency characteristics of the

lubricant, but generally should comprise about 70 to about 90 per cent by weight of the total composition.

The bentonite compounds employed in accordance with the invention are compounds composed of a montmorillonite mineral in which at least a part of the cation content of the mineral has been replaced by an organic base. Clays that swell at least to some extent on being contacted with water and contain as a primary constituent a mineral of the group known as montmorillonites are generally referred to as bentonites. Such clays, which contain exchangeable alkali metal atoms either naturally or after treatment, constitute the raw materials employed in making the bentonite-organic base compounds used in the compositions of this invention. So far as known, all naturally occurring montmorillonites contain some magnesium and certain of them, as exemplified by Hector clay, contain such a high percentage of magnesium that they largely have magnesium in place of the aluminum content characteristic of the more typical montmorillonites.

The bentonite-organic base compounds are preferably prepared as described in U. S. Patent No. 2,033,856, issued March 10, 1936, by bringing together the bentonite and the organic base in the presence of aqueous mineral acid to effect base exchange. The organic bases should preferably be titratable with mineral acids. Among these reactive bases are many alkaloids, and cyclic, aliphatic, and heterocyclic amines. The bentonite-organic base compounds used in preparing the lubricating compositions of this invention are preferably those prepared by bringing together a bentonite clay and such organic bases as aliphatic amines, their salts, and quaternary ammonium salts. Examples of such amines and salts are: decylamine, dodecylamine, tetradecylamine, hexadecylamine, octadecylamine, hexadecyl ammonium acetate, octadecyl ammonium acetate, dimethyldioctyl ammonium acetate, dimethyldidodecyl ammonium acetate, dimethyldodecyhexadecyl ammonium acetate, dimethyldicetyl ammonium acetate, dimethylhexadecyloctadecyl ammonium acetate, dimethyldioctadecyl ammonium acetate, and the corresponding chlorides and quaternary ammonium chlorides. The organic bases employed should be such as to impart substantial organophilic properties to the resulting compounds. The preferred bentonite compounds are prepared from quaternary ammonium compounds in which the N-substituents are aliphatic groups containing at least one alkyl group with a total of at least 10 to 12 carbon atoms. When aliphatic amines are used they preferably contain at least one alkyl group containing at least 10 to 12 carbon atoms.

The amount of bentonite compound used may vary over wide limits depending upon the particular oil with which the bentonite compound is to be blended and upon the properties desired in the final lubricating compositions. While as much as 15 per cent by weight of the total composition may comprise the bentonite compound, I prefer to use smaller amounts, that is, in the order of about 2 to 10 per cent by weight. It should be understood, however, that depending upon the consistency of the composition desired, less than 2 per cent or more than 10 per cent of the bentonite compound may be employed. In accordance with my invention, the bentonite compound is always used in amounts such that the weight ratio of the bentonite compound to wax in the finished jelly is not greater than 1:1. In most cases the ratio of the bentonite compound to wax is between about 1:1 and 1:15. When a bentonite compound is mentioned, it is understood, of course, that one or more of such compounds is intended.

In some instances, as when using short or single chain aliphatic amine bentonite compounds for example, dispersion of the organic bentonite compound in the oil can be facilitated by the use of one or more solvating agents. Suitable solvating agents are polar organic compounds such as organic acids, esters, alcohols, ethers, ketones, and aldehydes, especially low molecular weight compounds of these classes. Examples of suitable solvating agents are: ethyl acetate, acetic acid, acetone, methyl alcohol, ethyl alcohol, benzoyl chloride, butyl stearate, cocoanut oil, cyclohexanone, ethylene dichloride, ethyl ether, furfural, isoamyl acetate, methyl ethyl ketone, and nitrobenzene. In cases where the use of a solvating agent is desirable for effecting more rapid and more complete dispersion of the organic bentonite compound in the oil, ordinarily only a relatively small amount of such agent may be necessary. However, as much as about 50 per cent by weight based on the amount of the bentonite compound can be used.

In instances where the petroleum lubricating jelly is subjected to prolonged use under oxidizing conditions, I advantageously incorporate in the lubricating jelly a small amount of a diaryl amine oxidation inhibitor. As examples of some of the diaryl amines which may be used in the compositions of my invention may be mentioned diphenylamine, phenyl alpha naphthylamine, phenyl beta naphthylamine, alpha alpha, alpha beta and beta beta dinaphthylamines, and the like. Other diarylamines as well as their derivatives wherein one or more hydrogen atoms on one or both of the aromatic nuclei are replaced by a substituent group may be used. The substituting group may, for example, be one selected from the class of aryl, alkyl, amino, aryloxy and alkyloxy radicals, so long as the presence of the substituent does not render the diaryl amine insoluble in oil, or soluble in water or otherwise adversely affect the effectiveness of the diaryl amine. The amount of the diaryl amine employed will depend to a large extent upon the severity of the conditions to which the composition is subjected, as well as the particular diaryl amine used. For instance when the composition is subjected to prolonged use under oxidizing conditions, such as under extreme temperature and pressure, the diaryl amine requirement will be much greater than when relatively mild operating conditions are encountered. Generally, however, the amount of the diaryl amine employed is between about 0.1 and 1.5 per cent by weight based upon the weight of the total composition. In any case, an amount sufficient to substantially inhibit oxidational deterioration is employed.

In compounding these thixotropic lubricants, the wax and oil to which a diaryl amine has previously been added are customarily first melted together. The bentonite compound is then stirred into the mixture and the mixture is milled until a uniform composition is obtained.

The advantageous properties obtained by incorporating dimethyldicetyl ammonium bentonite in a thixotropic oil-wax mixture (Composition B), as compared with a thixotropic soap-type jelly

(Composition A), are illustrated in the following table.

	A	B
Composition (Parts by Weight):		
Oil (58 SUV at 100° F.)	78.0	78.0
Aluminum soap	4.0	
White Ceresin Wax	18.0	18.0
Dimethyldicetyl ammonium bentonite		4.0
Diphenylamine (added)	0.2	0.2
Consistency (A. S. T. M. D217-44T):		
Unworked	235	330
Worked	379	385
Percent Increase over Unworked	61.2	16 7
Dropping Point (A. S. T. M. D566-42), °F	199	>450
ABEC Machine:		
Final Running Torque—		
At 80° F	180	175
At 150° F	120	90
Percent Grease Leakage—At 80° F	31	24
Low Temperature Torque:		
204P Ball Bearing, Gm-Cm for 1st Revolution—		
At 0° F	712	961
At −40° F	815	1,090
Oil Separation, percent: 10 Grams in Nickel Filter Cone, 24 hrs. at 160° F	69	6

It is apparent from these results that a compound of a bentonite with an organic base has a very beneficial effect in increasing the dropping point and decreasing the oil separation of a thixotropic lubricant. It will be seen from a comparison of the results obtained that there is less change in consistency between the unworked and worked composition containing dimethyldicetyl ammonium bentonite than there is in the composition containing soap. It will further be seen that whereas the low temperature torque of the composition containing the bentonite compound is slightly greater than that of the soap-containing compound, the running torque of the bentonite compound is lower.

While this invention has been described with reference to specific details and examples of the production and properties of the compositions of my invention, it is to be understood that the invention is not intended to be limited to such details and examples except as recited hereinafter in the appended claims.

I claim:

1. A thixotropic petroleum lubricating jelly consisting essentially of a mixture of a major amount of a petroleum lubricating oil with a wax having a wax melting point above about 120° F., the amount of said wax being sufficient to provide an oil-wax mixture having a cloud point above about 110° F., and having incorporated therein an amount of a compound of a bentonite and an organic nitrogen base such that the ratio of bentonite compound to wax by weight is not greater than 1:1.

2. A thixotropic petroleum lubricating jelly consisting essentially of a mixture of a major amount of a petroleum lubricating oil with a wax having a wax melting point above about 120° F., the amount of said wax being sufficient to provide an oil-wax mixture having a cloud point above about 110° F., and having incorporated therein an amount of a compound of a bentonite and an aliphatic organic nitrogen base such that the ratio of bentonite compound to wax by weight is between about 1:1 and 1:15.

3. A thixotropic petroleum lubricating jelly consisting of about 70 to about 90 per cent by weight of a petroleum lubricating oil, at least about 4.0 per cent by weight of a wax having a melting point above about 120° F., and between about 1 and about 15 per cent by weight of a compound of a bentonite and an aliphatic organic nitrogen base such that the ratio of the bentonite compound to wax by weight is not greater than 1:1.

4. A thixotropic petroleum lubricating jelly consisting of about 70 to about 90 per cent by weight of a petroleum lubricating oil, about 4.0 to about 28 per cent by weight of a wax having a melting point above about 120° F., and between about 2.0 and 10.0 per cent by weight of a compound of a bentonite and an aliphatic organic nitrogen base such that the ratio of the bentonite compound to wax by weight is between about 1:1 and 1:15.

5. A thixotropic petroleum lubricating jelly consisting of about 70 to about 90 per cent by weight of a petroleum lubricating oil, about 4 to about 28.0 per cent by weight of ceresin wax, about 2 to about 10 per cent by weight of dimethyldicetyl ammonium bentonite such that the ratio of the bentonite compound to wax by weight is between about 1:1 and 1:15, and between about 0.1 and about 1.5 per cent by weight of a diaryl amine.

6. A thixotropic petroleum lubricating jelly consisting of about 78 per cent by weight of a petroleum lubricating oil, about 18 per cent by weight of ceresin wax, about 4 per cent by weight of dimethyldicetyl ammonium bentonite and about 0.2 per cent by weight of diphenyl amine.

7. A thixotropic petroleum lubricating jelly consisting of about 70 to about 90 per cent by weight of a petroleum lubricating oil, about 4.0 to about 28 per cent by weight of a wax having a melting point above about 120° F., and between about 2.0 and 10.0 per cent by weight of dimethyldicetyl ammonium bentonite such that the ratio of the bentonite compound to wax by weight is between about 1:1 and 1:15.

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