

[54] LUBRICATING COMPOSITION

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[57] ABSTRACT

Lubricating composition comprising a hydrocarbon oil
and polyethylene having an average molecular weight
within the range of from over 1 million to about 5 mil-
lion, the composition being of various forms, namely a
fluid-like gel, a jelly-like gel and a rigid gel, depending
on the amount of polyethylene present in the composi-
tion.

10 Claims, No Drawings

LUBRICATING COMPOSITION

This is a continuation of application Ser. No. 771,273, filed Oct. 28, 1968, now U.S. Pat. No. 3,547,819, which is a continuation-in-part of abandoned application Ser. No. 668,692, filed Sept. 18, 1967, which is a continuation of abandoned application Ser. No. 486,214, filed Sept. 9, 1965, which in turn is a continuation-in-part of abandoned application Ser. No. 363,294, filed Apr. 28, 1964.

In a related application, namely Ser. No. 771,348, filed on Oct. 28, 1968, and now U.S. Pat. No. 3,541,011, there is described and claimed lubricating compositions containing a lubricant, polyethylene having an average molecular weight within the range of from over 1,000,000 to about 5,000,000 and, in addition, a polyethylene having a molecular weight of 1,000,000 or less.

There is a great demand and a long-felt need in every industry for a lubricating product which, once used as a lubricating medium in an object, is lubricant-releasing and imparts a long-lasting self-lubricating quality to the object. For example, self-lubricating bearings are extremely useful in the automotive industry, the aviation industry, and almost anywhere bearings are used. If such lubricating product has a mechanical strength sufficient to be a part of the structure of the object for which it is a lubricant, then, not only the self-lubricating quality of the object achieved, but the object also can be produced easily, economically and without the usual problems of manufacturing. Accordingly, an object of this invention is to fulfill the long-felt need for the lubricating product by providing a lubricating composition which is simple, economical and easy to use.

Another object of this invention is to provide a lubricating composition the viscosity of which may be varied within a wide range.

Another object of this invention is to provide a lubricating composition comprising a lubricant and a high molecular weight polyethylene.

Another object of this invention is to provide a lubricating composition which has sufficient mechanical strength to form all or a portion of the structure of an object.

Another object of the present invention is to provide a lubricating material for an object to impart a long-lasting self-lubricating quality to the object.

Another object of this invention is to provide a lubricating composition that can be poured and set up in situ. These and other objects and advantages of this invention will become apparent and best understood from the following description and appended claims.

The term "self-lubricating" as used herein in connection with an object, such as a bearing, indicates that a continuous or periodical supply of a lubricant from an external source to the object is unnecessary. The percentages of different materials used herein are all based on the total weight of the composition of this invention. The terms "molecular weight" as used herein denote average molecular weight. The terms "high molecular weight" as used herein denote a molecular weight of from over 1.0×10^6 to about 5.0×10^6 . The terms "lower molecular weight" as used herein denote a molecular weight of 1,000,000 or less, for instance, between about 1,000 and 100,000.

Briefly, it has been discovered that a gel composition comprising a polyethylene having a high molecular

weight, that is a molecular weight within the range of from over 1,000,000 to about 5,000,000, and a lubricant, such as a hydrocarbon oil, is a long-lasting lubricating medium. The higher the molecular weight of the high molecular weight polyethylene and/or the higher the amount of high molecular weight polyethylene in the composition, the firmer and more viscous is the gel composition. Thus, depending upon the specific molecular weight of the high molecular weight polyethylene and the specific amount thereof in the composition, the physical characteristics of the composition vary and range from a thin, weak gel which behaves as a liquid to a firm, tough, rigid gel which looks like and functions as a solid. The intermediate composition between the weak gel and the rigid gel can be characterized as a jelly-like gel.

In storage, the composition of this invention has the capability of retaining the lubricant for as long as desired. Upon use as a lubricating medium for an object, such as a solid lubricant for a bearing, the composition is continuously and gradually lubricant-releasing for the life of the object and imparts a long-lasting, self-lubricating quality to the object. The firm, rigid gel can be, if desired, produced and adapted to function as a part of the structure of the object for which it is a lubricating medium. For instance, the lubricating composition may form the socket portion of a ball joint comprising a ball and a socket and, at the same time, can lubricate the surface of the ball.

The amount of the high molecular weight polyethylene employed in the gel composition depends upon the desired viscosity, toughness, and strength of the composition. As stated hereinabove, the higher the amount of the high molecular weight polyethylene in the composition, the more viscous and stronger is the gel. For instance, the gel behaves as a liquid when up to about 1 percent of the high molecular weight polyethylene is used. The mere presence of the high molecular weight polyethylene in the gel has an influence on its properties. However, it is desirable that the liquid gel composition contain at least about 0.5 percent of the high molecular weight polyethylene in order to realize fully the benefits gained by adding it to the oil. The gel behaves as a gelatinous jelly-like composition when from greater than 1 percent to about 5 percent of the high molecular weight polyethylene is used. The gel is rigid and functions as a solid when from over 5 percent to about 90 percent or higher of the high molecular weight polyethylene is used to form the gel. It has been found that the structural strength of the rigid gel composition is excellent when the amount of high molecular weight polyethylene is within the range of about 25 percent to about 85 percent. Consequently, in applications where structural strength of the composition is important, it is preferred that the composition contain about 25–85 percent of high molecular weight polyethylene. In applications where structural strength of the rigid gel composition is not of primary importance, amounts of high molecular weight polyethylene within the range of about 8 percent to about 40 percent can be used advantageously.

Surprisingly, it has been found that the inclusion of a lower molecular weight polyethylene, that is one having a molecular weight of 1,000,000 or less, in the gel composition of this invention modifies the lubricant-

retaining and lubricant-releasing characteristics of the gel. (As mentioned hereinabove, lubricating compositions containing the lower molecular weight polyethylene in combination with a lubricant and a higher molecular weight polyethylene are described and claimed in U.S. Pat. No. 3,541,011 issued Nov. 17, 1970.) Such modification in the lubricating quality of the gel is especially advantageous when the composition is a firm, rigid gel and is to be used as a part of the structure of a self-lubricating object, for instance, a bearing. More specifically, the presence of the lower molecular weight polyethylene causes a slow down in the rate at which the lubricant is dispensed from the composition. By varying the amount of lower molecular weight polyethylene in the composition, there can be made compositions which have different lubricant dispensing rates for particular applications.

The molecular weight of the lower molecular weight polyethylene employed in a gel composition is preferably much lower than that of the high molecular weight polyethylene, for example, between about 1,000 to about 100,000.

Most preferably, the molecular weight of the lower molecular weight polyethylene is within the range of about 20,000 to about 50,000. Compositions containing polyethylene within this molecular weight range have been found to have very desirable oil-releasing characteristics for the various applications in which the composition can be used.

The amount of the lower molecular weight polyethylene to be used to produce the thin, weak gel can vary; however, the preferred amount is within the range of about 0.25 percent to about 1 percent. In a liquid gel composition which contains a lower molecular weight polyethylene, the amount of high molecular weight polyethylene can be as low as 0.25 percent. It is also preferred that the total polyethylenes in the liquid gel composition be not more than about 1 percent. The amount of the lower molecular weight polyethylene in the jelly-like composition and the rigid gel composition can vary; however, it is preferred to use, respectively, up to about 10 percent, and up to about 40 percent of the lower molecular weight polyethylene. Most preferably the jelly-like composition should contain about 1 percent to about 5 percent of the lower molecular weight polyethylene. Most preferably, the rigid gel should contain about 1 percent to about 15 percent of lower molecular weight polyethylene.

The amount of the lubricant to produce the thin, weak gel, the jelly-like gel, and the rigid gel of this invention can vary over a wide range. However, it is preferred to employ, respectively, about 99 percent or higher, from about 95 percent to less than 99 percent, and from about 10 percent to less than 95 percent of the lubricant.

The unique properties of the composition of this invention allow it to be prepared in a variety of ways, each of which has its particular advantages. In general, the process of producing the composition of this invention is simple and comprises admixing the high molecular weight polyethylene, preferably in powdered or flake form, with a lubricant, for example, a mineral oil, heating the mixture, with or without stirring or agitation, to a sufficient temperature high enough to cause melting of the polymer component thereof and for a

time sufficient to obtain a homogeneous mixture of polyethylene and lubricant. Upon cooling, the homogeneous mixture or mass forms the gel composition of this invention. The viscosity and strength of the composition depend, as described hereinabove, on the molecular weight of the high molecular weight polyethylene employed and the amount thereof.

The temperatures employed to heat a mixture of a high molecular weight polyethylene and a lubricant in the method of this invention are sufficiently high to melt and cause mixing of the components of the mixture. In general, the heating temperatures employed range between about 240°F. and 450°F.; however, the temperatures should not be high enough to cause thermal decomposition. When the mixture begins to form into a homogeneous mass, it will be observed that for a composition which contains about 5 percent or more high molecular weight polyethylene, there is obtained a thick cohesive, but flowable, composition. On the other hand, when the high molecular weight polyethylene comprises less than about 5 percent of the composition, there is obtained a thin fluid mixture at the temperature of homogeneous mass formation.

After the components have blended into a homogeneous composition within the temperature range of about 240°F. to 450°F., the composition should then be allowed to cool. When preparing the jelly-like and rigid gels, it will be observed that as the homogeneous mass is cooled, it will begin to take on the appearance of its final form at a temperature generally within the range of about 200°F. to about 300°F. This is the temperature transition range where the homogeneous mass begins to change from a flowable state to a solid jelly-like or rigid state.

During the heating of a mixture of high molecular weight polyethylene and lubricant, it is preferred to agitate the mixture to cause better mixing so that the polyethylene does not settle out of the mixture. Any conventional method of producing the mixing, such as in an ordinary extruder or sigma mixer is satisfactory. A conventional laboratory propeller-type mixer can be used conveniently when preparing the fluid-like gels.

Another important advantage that is realized when the polyethylene/lubricant mixture is agitated or mixed as it is being heated is that the overall time for preparing the gel composition can be shortened. In this connection it is noted that the time for preparing the composition will depend upon a number of variables, including the overall amount of the composition that is being prepared and the manner in which the components are mixed and heated during the preparation. By way of example, it can take from about 5 to 10 minutes to prepare about 1,000 grams of the composition by hand mixing. Longer mixing and heating times may be required to prepare larger batches of the composition. On the other hand, large amounts of the composition can be prepared in less time if an extruder is used as, the heating and agitating device. If the polymer/lubricant components are not agitated during the heating stage, it generally takes about 45 to about 60 minutes before the components blend into a homogeneous mass.

There are applications, however, where that characteristic of the composition of this invention which enables it to be prepared without stirring during the heat-

ing stage of preparation is extremely important. For example, in many applications wherein it is expedient that the rigid gel composition be made by molding it in situ to form a self lubricating object, for example by encapsulating multiple bearings in a housing, it is very difficult, if not impossible to stir and agitate the mixture due to the size or shape of that mold. Whereas heretofore known polymer/lubricant compositions which require stirring and agitation for preparation would hardly be suitable for such applications, the composition of this invention can be used very suitably and to good advantage. Thus, at the expense of a somewhat longer preparation time, the unique properties of the composition of this invention which allow it to be prepared by simply heating without stirring enable it to be used in applications where the use of known polymer/lubricant compositions would be attended by difficult problems. As will be shown in connection with the examples reported below, attempts to prepare lubricating compositions containing oil and a polyethylene of molecular weight lower than about one million, by simply heating a mixture of the oil and polyethylene have shown that the preparation of a homogeneous composition was attained only after heating the mixture for a much longer period of time than that needed to prepare the composition of this invention.

Another and preferred method of producing the rigid, tough gel composition of this invention comprises first preparing a homogeneous fluid-like, thin, weak gel composition of the high molecular weight polyethylene, in accordance with the method of this invention described hereinabove, and then mixing additional high molecular weight polyethylene with the weak gel composition to obtain a liquid dispersion mixture thereof. (This dispersion mixture is stable over a relatively long period of time, that is the polyethylene does not tend to settle out of the liquid dispersion mixture.) This dispersion mixture is heated with or without stirring to a temperature sufficient to melt the mixture, preferably between about 240°F. and 450°F. The heated mixture forms the desired rigid composition upon cooling to a temperature sufficient to solidify it. This usually occurs at a temperature between about 200°F. and 300°F. The amount of the high molecular weight polyethylene dispersed in the weak gel composition should be sufficient to produce the desired rigidity and toughness for the final gel product. In any event, the total amount of high molecular weight polyethylene in the rigid gel should be the same as that described hereinabove for a rigid gel composition of this invention.

An advantage that is realized by preparing the rigid gel composition of this invention according to the preferred method set forth immediately above is that the above described stable dispersion mixture can be shipped in liquid form by a formulator to a manufacturer or processor who can then convert the liquid dispersion mixture, by heating and cooling as described, into a rigid self-lubricating object of any desired shape. It is noted that if an amount of high molecular polyethylene in excess of about 5 percent is combined with a lubricant and dispersed therein, it will be found that the polyethylene tends to settle out in a relatively short period of time, that is, it is not a stable

dispersion as is the stable dispersion described above. As a consequence, one is confronted with handling problems when this type of dispersion is shipped from the formulator to the manufacturer or processor.

The rigid, tough gel composition of this invention can be easily adapted to be a part of the structure of a self-lubricating object, such as an anti-friction bearing. In this manner, not only the rigid gel composition is a structural part of the object, such as the retainer and/or seal, but also it is continuously and gradually lubricant-releasing for the life of the object and imparts a long-lasting, self-lubricating quality to the object. For instance, to adapt the rigid composition to be a part of the structure of a bearing, a dispersion mixture of a weak gel composition of this invention and a high molecular weight polyethylene is prepared as described above and used to fill the space between the inner and outer rings of the bearing, i.e., the space in which the rolling elements of a bearing are located. The bearing which is filled with the dispersion mixture can be heated to a temperature between 240°F. and 450°F. without stirring. The heated dispersion mixture in the bearing is then cooled to solidify the mixture, thereby forming a rigid, strong portion of the bearing. The rigid gel in the bearing tends to eliminate the need for a retainer and/or seal for the rolling elements and the oil which exudes from the gel lubricates the bearing. Such bearing can be used in any conventional manner, such as to support the shaft in an electric motor.

The composition has the capability of being poured and molded in situ for uses such as electrical insulation for cables and as an encapsulating material for electronic components. The product of this invention has high voltage breakdown and self-healing characteristics and with certain additives, such as boron, it is a shielding material for nuclear reactors. The composition of this invention can be used in other applications depending upon the physical properties and characteristics of the composition. For instance, the weak gel composition which behaves like a liquid is useful in the lubrication of gear trains or as a motor oil which substantially reduces the wear and tear of a motor. The gelatinous, jelly-like composition is useful in the lubrication of porous or non-porous sleeve, ball, and roller bearings. Present methods for lubricating porous and non-porous sleeve bearings involve the use of a reservoir which holds a felt or fibrous material which is impregnated with oil which is fed by capillary action to the bearings. The compositions of the present invention can be substituted for the oil impregnated felt or fibrous materials and will function to lubricate the bearings in a manner such that the disadvantages inherent in the use of felt and fibrous materials are avoided.

The rigid gel composition not only can be used in forming the self-lubricating bearing described above, but it can also be used in producing different articles, such as the socket of a ball joint. To form the socket by previous methods required high precision which makes those methods expensive. It has been found that an economical and easy method of producing the socket comprises filling a container with the dispersion mixture of the weak gel composition and a high molecular weight polyethylene, heating the dispersion mixture in the container between about 240°F. and 450°F. to ob-

tain a homogeneous mixture thereof, inserting the ball into the container so that the homogeneous mixture covers the surface of the ball or any desired portion thereof, and cooling the homogeneous mixture to sufficient temperature to solidify the mixture, thereby forming a rigid and strong socket around the ball to produce the ball joint which is easily removable from the container. The ball joint thus produced is self-lubricating for the life thereof and has a strength sufficient to be used as the socket of the ball joint for any conventional application thereof.

Another extremely important property of the composition of this invention is that the rigid gel composition is capable of being molded by reciprocating screw injection machines of the types which are used for molding thermoset resins. As is well-known for certain applications, these types of molding machines have inherent advantages over other types of molding machines. Whereas it is impractical, if not impossible, to mold high molecular weight polyethylene in the aforementioned reciprocating screw injection machines, the rigid gel composition of this invention have properties which enable the composition to be molded very conveniently into a variety of shapes by said machines.

It should be understood that various additives normally used in lubricants can be added to the composition of this invention. Other additives, such as pigments and dyes, could also be used. However, we have found that the addition of a small amount of a suitable finely divided filler to the rigid composition of this invention, minimizes any possible dimensional instability, such as shrinkage, of the composition. The finely divided fillers should have a particle size within the range of about -50 to about -325 U.S. mesh and should have a relatively low coefficient of friction and be nonabrasive for the material or article which is being lubricated. Exemplary of such fillers are molybdenum disulfide; nylon powder, the preparation of which is described in U.S. Pat. Nos. 2,639,278 and 3,022,542; Lexan which is the trademark of General Electric Company for a polyaryl carbonate; and Delrin which is the trademark of DuPont de Nemours and Company for a polyacetal. The amount of the finely divided filler can vary over a wide range, and should be sufficient to produce the desired result; however, it is preferred to use between about 1 percent to about 20 percent of the filler.

As mentioned above, the high molecular weight polyethylene component of this invention has a molecular weight within the range of from over 1×10^6 to about 5.0×10^6 . Although the molecular weight range of the polyethylene is from over 1.0×10^6 to about 5.0×10^6 , a molecular weight range of from about 1.5×10^6 to about 3.5×10^6 is preferred.

Examples of high molecular weight polyethylenes are: a polyethylene produced by Allied Chemical Co. under the trade designation AC-3X, which has a particle size of about 40 mesh and a molecular weight of about 3×10^6 ; a polyethylene produced by Allied Chemical Co. under the trade designation AC-1221, which has a melt index of 0.00, particle size of less than 100 mesh screen, and an average molecular weight of from about 1.5×10^6 to about 2.5×10^6 ; and a polyethylene produced by Hercules Powder Company under the tradename Hi-Fax 1900, which has an

average molecular weight of over 2×10^6 and a melt index of 0.01.

The melt index of the high molecular weight polyethylene is between 0.00 and less than about 0.1 as measured by ASTM D1238-62T.

The high molecular weight polyethylenes can be produced by any conventional method. One method of producing the high molecular weight polyethylenes is by a process using Ziegler catalysts as described in a book entitled "Polyethylene" by R. A. V. Raff and J. B. Allison, 1956, page 78.

Examples of the lower molecular weight polyethylenes are: a polyethylene produced by Allied Chemical Company under the trade designation of AC-6A, which has an average molecular weight of 2000, specific gravity of 0.92, a viscosity of 180 c.p.s. at 284°F., and a melting point of 219°-226°F.; a polyethylene produced by Allied Chemical Company under the trade designation of AC-615, which has a molecular weight of 5,000, specific gravity of 0.92, a viscosity of 4000 c.p.s. at 284°F., and a melting point of 224°-232°F.; a polyethylene produced by Allied Chemical Company under the trade designation of AC-617, which has a molecular weight of 1500, a specific gravity of 0.91, a viscosity of 100 c.p.s. at 284°F., and a melting point of 210°-217°F.; a polyethylene produced by Eastman Chemical Company under the tradename Tenite, which has a melt index of 7, a specific gravity of 0.92, and a molecular weight of 23,000; and a polyethylene produced by U.S.I. Chemical Company under the tradename of Microthene (MN-718), which has a melt index of 22, a specific gravity of 0.915, and an average particle size of less than 20 microns. The lower molecular weight polyethylenes can be produced by different conventional methods which are well known in the art.

The lubricant employed in the practice of this invention can be any hydrocarbon oil which has a lubricating viscosity. Excellent results have been obtained when the oil utilized is a mineral oil; and mineral oils are the preferred oil component. The mineral oil may be a solvent-extracted or a solvent-refined oil. Exemplary of the hydrocarbon mineral oils are: Harmony oil No. 44 (SAE No. 10) produced by Gulf Refining Co., which is a pure hydrocarbon oil containing an antifoam additive, rust and oxidation inhibitors, and has a viscosity of 150.2 SUS at 100°F.; Insulating oil No. 10-C produced by General Electric Corporation; Sunoco 280 (SAE No. 20) produced by Sun Oil Co., which has a viscosity of 250-280 SUS at 100°F. and a specific gravity of 0.87 - 0.88; Gulf SAE 10 which is a regular motor oil and is produced by Gulf Oil Co.; Security No. 71 (SAE No. 40) produced by Gulf Oil Co., which is a pure hydrocarbon oil containing no additives and has a viscosity of 610 SUS at 100°F.; Security No. 53 (SAE No. 20) produced by Gulf Oil Co., which is a pure hydrocarbon oil containing no additives and has a viscosity of 305 SUS at 100°F.; Kendall oil 10W which is a regular motor oil; Shell oil No. 66602, bearing infusion oil, produced by Shell Oil Co.; Harmony oil No. 41 (SAE less than No. 20) produced by Gulf Oil Co., which is a pure hydrocarbon oil containing additives, antifoam, rust and oxidation inhibitors, and has a viscosity of 105 SUS at 100°F.; and Esso's Ultragear oil, SAE No. 90.

Although the hydrocarbon mineral oils are by far the preferred lubricant component, other oils, including vegetable and animal oils, of lubricating viscosity can be also used. In addition, various of the synthetic oils comprising hydrocarbon groups can be utilized.

The high molecular weight polyethylene component of the composition of this invention is insoluble in such typical organic solvents as benzene, toluene and decalin. Its solubility properties—or more aptly its insolubility properties—are thus quite different from the solubility properties of substantially lower molecular polyethylenes which are soluble in one or more of the aforementioned organic solvents and which are known to form solutions—as opposed to gels—when combined with oil. Solubility tests performed with polyethylenes of molecular weight within the range of about 1.5–2.5 million (Allied Chemical Co. AC—1221) have shown that it is insoluble at room temperature in benzene, toluene and decalin and also that it is insoluble in these organic solvents at their boiling point temperatures which are 176°F., 230°F., and 377°F., respectively.

During the solubility testing, it was observed that the above described high molecular weight polyethylene swelled in benzene at 176°F.; and that in toluene at 230°F. and in decalin at 377°F., there were obtained gelatinous mixtures. Upon cooling each of the hot gelatinous mixtures it was observed that some of the toluene and decalin separated from its corresponding gelatinous mixture.

The following examples which are identified by a Roman numeral are illustrative of compositions within the scope of this invention. The examples which are identified by a Roman numeral and capital letter, are illustrative of compositions containing oil and a polymeric material other than polyethylenes having a molecular weight within the range of over 1,000,000 to about 5,000,000 and are set forth for comparative purposes. The examples serve to show the very different types of compositions or properties thereof that are obtained when utilizing the high molecular weight polyethylenes of this invention as opposed to polyethylenes having lower molecular weight or other polymers.

Example I below is illustrative of the rigid gel composition of this invention and that method of preparation which requires heating but no mixing.

EXAMPLE I

120 grams of a hydrocarbon mineral oil (Sunvis 941 — Sun Oil Co.) and 40 grams of polyethylene of molecular weight 1.5–2.5 million (AC—1221 — Allied Chemical.) were cold mixed until the polyethylene particles were well distributed throughout the oil. This mixture, which comprised 75 percent oil and 25 percent polyethylene, was placed in a preheated oven at 350°F. and maintained therein for about 1 hour after which it was removed and allowed to cool. The cool product was removed from the beaker. The product which had assumed the cylindrical shape of the beaker was a tough, solid, oil-exuding, rigid gel. Upon cutting a pie-shaped piece extending the length of the cylindrical shape, visual observation showed the composition appeared homogeneous throughout and had a smooth, oily surface. A socket of a ball joint is a product that could be made from the composition of Example I.

EXAMPLE IA

one hundred-twenty grams of a hydrocarbon mineral oil (Sunvis 941 — Sun oil Co.) and 40 grams of polyethylene of molecular weight 500,000 (Hi-Fax 1806 — Hercules) were cold mixed until the polyethylene particles were well distributed throughout the oil. This mixture, which comprised 75 percent oil and 25 percent polyethylene was placed in a preheated oven at 350°F. and maintained therein for about 1 hour after which it was removed and allowed to cool. The product was very different from the product described in Example I above, although it was made in exactly the same way and contained the same relative proportions of oil and polymeric components. The product consisted of a very definite oily liquid phase and a solid mass of polymer and oil phase which was not nearly as tough as the product of Example I and which was much more easily penetrated with a pointed object than the product of Example I. Unlike the product of Example I, the product of Example IA upon standing was observed to be unstable in that the top surface of the polymer/oil mass, except for a small center area, seemed to separate into small irregular shaped globs of material. The aforesaid small center area diminished in size as it broke down into the aforesaid globs upon continued standing. The small center area was not nearly as tough and was much more easily penetrated by a point instrument than the tough, rigid, homogeneous product of Example I. Thus, it was observed that whereas the product of Example I remained homogenous, tough, smooth and rigid, the product of Example IA was not homogeneous upon preparation in that it consisted of an oil phase, a very mushy top surface comprised of small globs of oil and polymer, and a small center area that was not nearly as tough as the product of Example I and that was also unstable. The product of Example IA in this form would not be suitable for use as the socket portion of a ball joint.

EXAMPLE IB

A composition consisting of the same components as Example IA was prepared according to the same method set forth in Examples I and IA, except that the mixture was heated for two hours rather than one. The product upon standing was found to be of a solid mass that had a very porous, oily surface from which globs of mushy-like particles could be easily separated. Moreover, the surface of the product was very easily penetrated by a pointed instrument. Thus, this product, even though heated during preparation for 100% longer than the product of Example IA or Example I, was not tough and smooth-surfaced.

Example II below is illustrative of the preparation of a self-lubricating product within the scope of this invention.

EXAMPLE II

Thirty-five grams of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 were mixed with 65 grams of Harmony oil No. 44 (SAE No. 10) at room temperature. The mixture was poured into the space between the inner and outer rings of a ball bearing to cover the balls. The bearing was then covered on the top and bottom with aluminum plates and heated at 350°F. for 45 minutes in a forced air oven. The bearing

was then cooled to room temperature. The mixture in the bearing had solidified to a tough, oil-dispensing, rigid composition. The balls in the bearing had free and efficient movement and the bearing was self-lubricating.

EXAMPLE IIA

Thirty-five grams of a polyethylene having a molecular weight of 23,000 were mixed well with 65 grams of SAE No. 10 oil while heating to 300°F. A non-stringy liquid solution was produced. When the solution was cooled to room temperature a weak solid was produced. Within 10 hours a substantial amount of the oil slowly separated out from the solid and left a cake which was easily crumbled to a powder. Thus, this composition was not similar to the self-lubricating composition of Example II above.

As illustrative of the jelly-like gel compositions of this invention, which can be processed to a grease-like consistency, three compositions were made up and their oil separation properties were tested in accordance with a standard grease test, namely ASTM D-1742. The results of the test are reported in Table 1 below. The oil comprising the compositions of Examples III, IV and V reported in Table 1 was Sun Oil Company's Sunvis 941, a hydrocarbon mineral oil, and the polyethylene in said compositions was of molecular weight 1.5 - 2.5 million (AC 1221 - Allied Chemical Company).

There is also reported in Table 1 below, for comparative purposes, the compositions of Examples IIIA, IVA and VA, which compositions were comprised of the same oil utilized in compositions III, IV and V and a polyethylene having a molecular weight of 500,000 (Hi-Fax 1806 - Hercules Powder Company).

Except as noted below, each of the compositions reported in Table 1 was made according to the same method. This method included heating a mixture of the oil and polyethylene components in a container to a temperature of 320°F. - 350°F. and maintaining the composition within this temperature range for about 3 minutes. The mixture was agitated continuously during the heating cycle. The composition was then poured into trays and allowed to cool to room temperature. Each of the compositions, except those of Examples IVA and VA, was then passed through a meat grinder having a small mesh screen of one-sixteenth inch diameter holes. Thereafter, Examples III, IIIA, and IV and V were tested for oil separation properties. The compositions of Examples IVA and VA, which were not passed through the meat grinder, were not so tested, the reason for this being given immediately following the table.

TABLE 1

Ex. No.	Components of Composition weight %		Polymer, Mol. Wt.	Oil Separation Test
	Oil	500,000	1.5-2.5 Mil.	ASTM D-1742
III	99		1	16.8
IIIA	99	1		25.0
IV	97		3	9.7
IVA	97	3		
V	95		5	6.9

As mentioned above, the compositions of Examples IVA and VA, which contained polyethylene of molecular weight 500,000 were not tested for oil separation properties. The reason for this is that after these compositions were cooled, they were found to be of two phases, an oil phase and dispersed therein large hard lumps of polyethylene. (Some of the lumps in composition VA were 2 inches long.) Whereas compositions IV and V were homogeneous gel-like gels which were capable of being converted into a homogeneous composition of grease-like consistency by passing them through the meat grinder, the compositions of IVA and VA were not homogeneous gel-like compositions that were capable of being converted by the grinder into a homogeneous composition of grease-like consistency. It is further noted that attempts were made to prepare homogeneous compositions containing the components of Examples IVA and VA by heating the oil and polyethylene mixture to higher temperature, that is above 320°F. - 350°F. and increasing the time of heating and mixing. However, it was found that compositions containing no lumps could be made utilizing the increased temperatures and length of mixing time.

With respect to the compositions of III and IIIA reported in Table 1 above, it can be seen that the oil separation properties of Example III of the composition of this invention were much better—to the extent of being 30 percent lower. The significance of this is that a lubricating grease composition made from the composition of Example III would have a much longer lubricating life than one made from the composition of Example IIIA.

EXAMPLE IVB

An oil-polymer composition wherein the polymer was isotactic polypropylene was prepared. The composition was made from a mixture containing 3 percent isotactic polypropylene and 97 percent Sunvis 941 oil by heating, while stirring the mixture, to a temperature of about 350°F. Upon cooling, there was obtained an oily liquid phase which contained suspended undissolved globs of polypropylene. With reference to Example IV in Table 1 above, which identifies a homogeneous composition of grease-like consistency, it should be appreciated that the isotactic polypropylene/oil composition was of a vastly different type of product.

In the comparative tests reported in Table 2 below, the oil viscosity and oil separation properties of a liquid-like gel composition of this invention containing a polyethylene of molecular weight 1.5 - 2.5 million (AC 1221 of Allied Chemical Company) and a mineral oil were compared with those of a composition consisting of a polyethylene having a molecular weight of 500,000 (Hi-Fax 1806 of Hercules Powder Company) and a mineral oil. The mineral oil in each of the compositions was Sunvis 941 of Sun Oil Co.

Each of the compositions set forth in Table 2 below was prepared in the same way. They were each prepared by heating a mixture of the oil and polyethylene components to 320°F. - 350°F. and maintaining the composition within this temperature range for about 3 minutes. The mixture was agitated continuously during the heating cycle. The composition was

then cooled, placed in a mixing bowl and blended with a wire heater at No. 8 speed for about 10 minutes. (A Kitchen Aid mixer Model K5-A was used.)

The viscosity of each of the compositions was then determined. Also, each of the compositions was allowed to stand in a beaker for over a month and periodic observations were made to determine whether any oil separated from the composition. Upon observing that a layer of oil separated from the composition, the depth of the layer was measured with a ruler.

The results are reported in Table 2 below.

TABLE 2

Ex. No.	Components of Composition, Wt. %			Visc. at 75°F. c.p.s.	Oil Separation in inches			
	Oil	500 K	1.5 - 2.5 Mil.		1 day	8 days	18 days	35 days
VI	99.5		0.5	1439	none	none	1/32	1/32
VIA	99.5	0.5		925	none	none	1/8	1/8

Prior to discussing the viscosity and oil separation properties reported in the above table, it is first noted that small undissolved particles of polyethylene were present in the composition of Example VIA. On the other hand, the composition of Example VI was clear and homogeneous throughout, and contained no visible particles of polyethylene. In lubricating applications wherein the lubricant had to pass through small openings, for example screens or orifices, the composition of Example VIA could tend to block the openings. This problem would not be encountered using the composition of Example VI.

From the Table 2 above, it can be seen that the lubricating composition within the scope of the invention had a viscosity of over 55% greater than that of the composition of Example VIA. In view of these results, it is evident that the addition of high molecular weight polyethylene to oil in very small amounts is much more effective in producing a composition of increased viscosity than a polyethylene of lower molecular weight.

With respect to the stability properties of the compositions of Examples VI and VIA, Table 2 shows that after 8 days, the composition of Example VIA was unstable to the extent that a one-eighth inch layer of oil had separated from the composition. On the other hand, after 18 days, there was only a one thirty-second inch layer of oil on the top of composition VI. The significance of this is that the composition of Example VI would have a much longer lubricating life than that of the composition of Example VIA.

The following examples are further illustrations of the rigid gel compositions of this invention.

EXAMPLE VII

Sixty-seven grams of Harmony oil No. 44 (SAE No. 10 oil) and 33 grams of polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 were mixed at room temperature. The mixture was poured in a 0.625 inch

by 5.50 inch glass tube. Both ends of the tube were covered. The filled tube was placed in a preheated forced air oven at 350°F. for 40 minutes. The filled tube was removed from the oven and cooled for 5 minutes at room temperature and quenched in a water bath. A gel composition was obtained and removed from the tube. The result was a smooth, tough, flexible and voidless composition which had assumed the shape of the tube.

EXAMPLE VIII

Seventy-five grams of Security oil No. 53 (SAE No. 20 oil) and 25 grams of polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 were mixed thoroughly and put in a forced air oven at 300°F. for 5 minutes. The mixture was then removed from the oven and cooled to room temperature. The result was a strong, uniform, solid product.

EXAMPLE IX

The procedure of Example VIII was followed except that the polyethylene had a molecular weight of 3×10^6 . The result was substantially the same as that in Example VIII.

The following examples are illustrative of the rigid gel compositions of this invention containing a finely divided filler material which minimizes dimensional instability.

EXAMPLE X

8.4 grams of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 , 8.4 grams of a nylon powder, and 83.2 grams of Harmony oil (SAE No. 10 oil) were mixed at room temperature. The mixture was poured into the annular space of a ball bearing which was preheated to 350°F. The filled bearing was placed in a forced air oven at 350°F. for 45 minutes. The bearing was cooled to 200°F. The result was a tough, oil-exuding, rigid gel in the bearing which did not hinder the ball movement. There was no shrinkage of the rigid gel in the bearing after 6 months.

EXAMPLE XI

8.4 grams of polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 , 8.4 grams of a nylon powder, and 83.2 grams of SAE No. 90 oil were mixed and heated at 380°F. until a rubbery gel was formed. The gel was cooled slowly until it solidified. The product was a firm, rigid gel.

EXAMPLE XII

Two grams of polyethylene having a molecular weight of 3×10^6 , 75 grams of Security oil No. 53 (SAE No. 20 oil), 11 grams of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 and 8 grams of a nylon powder were mixed. The mixture was heated to about 350°F. until a thick gel was formed. The gel was cooled until solidified at about 200°F. The gel was left set at room temperature for 6 months and no dimensional changes were observed.

The next example is illustrative of another jelly-like gel composition within the scope of this invention.

EXAMPLE XIII

Ninety-six grams of Sunoco No. 280 (SAE No. 20 oil) and 2 grams of a polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 were mixed and slowly heated to 300°F. At 300°F., the mixing process was continued for one hour. The hot mixture was then poured into a container and cooled to room temperature. The result was a stable, oil-retaining and rather smooth looking jelly-like product.

For the purposes of illustration, there is set forth below compositions containing a low molecular weight polyethylene component in addition to the high molecular weight polyethylene component and lubricant component. It is noted that compositions of this type can be prepared according to any of the methods discussed above in connection with the two component composition, that is one containing lubricant and high molecular weight polyethylene components.

EXAMPLE XIV

Five quarts of SAE No. 20 oil containing 0.25 percent of a polyethylene having a molecular weight of 23,000 and 0.25 percent of a polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 was mixed and heated to 300°F. to form a homogeneous low viscosity gel. Upon cooling to 200°F., the gel became more viscous and was a homogeneous fluid gel. This product was used as a motor oil and placed into the engine crank case of an old, worn-out 1957 Mercury automobile. The combustion efficiency of the automobile increased 50 percent and its oil consumption decreased by a factor of 10. When the product was used as a motor oil additive in an automobile, the combustion efficiency increased and its oil consumption decreased.

EXAMPLE XV

6.5 grams of a polyethylene having a molecular weight of 23,000 was mixed with 91.5 grams of Gulf SAE No. 10 motor oil in a beaker and heated to 180°F. The viscosity of the mixture was about the same as that of the oil. This mixture was then cooled to room temperature and 2 grams of a polyethylene of an average molecular weight of 1.5×10^6 to 2.5×10^6 was mixed with it to form a dispersion of the polyethylenes and the oil. The dispersion was then slowly heated and stirred in the beaker to 300°F. After about 3 minutes a homogeneous mixture was formed which was cooled to form a jelly-like, oil-retaining homogeneous gel.

EXAMPLE XVI

91.5 grams of Gulf SAE No 10 regular motor oil, 6.5 grams of a polyethylene having a molecular weight of 23,000 and 2.0 grams of a polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 were thoroughly mixed. Sixty lbs./hour of this mixture was passed through an extruder (1.5 inch Thermatic extruder) having a screw diameter of 1.5 inches and a barrel temperature of 270°F. and a nozzle temperature of 350°F. The product was similar to that of Example XV at room temperature.

EXAMPLE XV

91.5 grams of Security No. 53 (SAE No. 20 oil), a pure hydrocarbon oil containing no additives, 6.5 grams of a polyethylene having a molecular weight of 23,000 2.0 grams of polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 were thoroughly mixed and 20 lbs./hour of this mixture was passed through a 1.5 inch Thermatic extruder with a barrel temperature of 400°F. and a nozzle temperature of 350°F. The product was similar to that of Example XV at room temperature.

EXAMPLE XVIII

Ninety-two grams of Sunoco No. 280 (SAE No. 20 oil), a petroleum oil containing additives and inhibitors, 4 grams of a polyethylene having a molecular weight of 23,000, and 4 grams of a polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 were thoroughly mixed. Twenty lbs./hour of this mixture was passed through the same extruder as in Example XVI. At room temperature, the product was similar to that of Example XV, except that it was more cohesive and stronger gel.

EXAMPLE XIX

The procedure of Example XVIII was followed except that the oil used was Shell oil No. 66602, a bearing infusion oil. The product was similar to that of Example XVIII.

EXAMPLE XX

94.50 grams of the petroleum oil used in Example XVIII, 2.75 grams of a polyethylene having a molecular weight of 23,000, and 2.75 grams of a polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 were mixed and then passed through a heat exchanger (Votator) at 17 - 19 p.s.i. and a temperature of 300°- 330°F. The product was a thick, cohesive liquid and upon cooling it became a homogeneous jelly-like product. This jelly-like product was passed through an ordinary meat grinder. The result was a smooth, grease-like gel.

EXAMPLE XXI

Ninety-six grams of the petroleum oil used in Example XVIII, 2.0 grams of a polyethylene having an average molecular weight of 1.5×10^6 and 2.5×10^6 and 2.0 grams of polyethylene having a molecular weight of 23,000, were mixed and slowly heated to 300°F. At 300°F. the mixing process continued for one hour. The hot mixture was then poured into a container and cooled to room temperature. The result was a stable, oil-retaining, and rather smooth-looking jelly-like product.

EXAMPLE XXII

71.7 grams of Harmony oil No. 44 (SAE No. 10) and 0.7 grams of a polyethylene having a molecular weight of 23,000 were thoroughly mixed and heated until the polyethylene was dissolved in the oil. The mixture was cooled to room temperature and 27.6 grams of polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 was added to and mixed thoroughly therewith. The result was creamy, pourable and thick dispersion mixture.

A ball bearing was dipped into some SAE No. 40 oil and then placed in a container and preheated for 15 minutes at 300°F. in an oven. The bearing was then removed from the oven and the space between its inner and outer rings was filled with the dispersion mixture which embodied the balls. The bearing was then returned to the oven for 45 minutes at 300°F. The bearing was cooled to about 200°F., the dispersion mixture solidified to a rigid gel and the balls therein were completely free to rotate. The bearing was self-lubricating and the rigid gel in the bearing was oil-retaining.

EXAMPLE XXIII

Seventy-five grams of SAE No. 40 oil, 0.375 gram of a polyethylene having a molecular weight of 23,000, 0.375 gram of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 and 8 grams of a dried nylon powder were mixed and heated to 300°F. while agitating the mixture. The heated mixture was cooled to about 200°F. and formed a fluid, weak gel product. At room temperature, 12 grams of the 1.5×10^6 to 2.5×10^6 molecular weight polyethylene was added to the fluid product and mixed thoroughly. This produced a dispersion mixture of the product and the polyethylene. A ball bearing was dipped into some SAE No. 40 oil and then placed in a container and preheated for 15 minutes at 300°F. in an oven. The bearing was removed from the oven and the space between its inner and outer rings was filled with the dispersion mixture which embodied the balls. The bearing was returned to the oven for 45 minutes at 300°F. After cooling to about 200°F., the dispersion mixture had solidified and the balls therein were completely free to rotate. The bearing was self-lubricating and the solidified mixture herein was oil-retaining. After 6 months there was no shrinkage of the mixture in the bearing.

EXAMPLE XXIV

81.7 grams of Harmony oil No. 44 (SAE No. 10) and 1.5 grams of a polyethylene having a molecular weight of 23,000 were mixed while heating to 200°F. The mixture was cooled to room temperature. To this cooled mixture, 8.4 grams of a nylon powder, and 8.4 grams of polyethylene having an average molecular weight of 1.5×10^6 to 2.5×10^6 were added and mixed thoroughly and heated to 350°F. for 15 minutes and then cooled slowly to room temperature. The product was a tough, strong solid. The product was left set for 6 months and the oil was still well retained therein.

EXAMPLE XXV

Ninty-nine grams of Security oil No. 53 (SAE No. 20), 0.5 gram of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 , and 0.5 gram of polyethylene having a molecular weight of 23,000 were mixed and heated. Upon cooling, the mixture formed a gel. There were mixed 75 grams of this gel with 12 grams of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 and 13 grams of a nylon powder.

The mixture was poured into a mold and then heated at 325°F. for 30 minutes. Upon cooling, the product was a tough, rigid gel.

EXAMPLE XXVI

Ninety-nine grams of Harmony oil No. 44 (SAE No. 10), 0.5 gram of a polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 and 0.5 gram of polyethylene having a molecular weight of 23,000 were mixed and heated to 300°F. Of the heated mixture 77.8 grams were cooled to room temperature and mixed with 13.4 grams of a the polyethylene having a molecular weight of 1.5×10^6 to 2.5×10^6 and 8.8 grams of a nylon powder. A dispersion mixture was obtained which was mixed thoroughly, poured into a mold and then heated to 300°F. for 60 minutes. Upon cooling, a rigid, homogeneous gel composition was produced in the shape of the mold which had no shrinkage after 6 months.

We claim:

1. A lubricating composition comprising a hydrocarbon oil of lubricating viscosity and polyethylene having an average molecular weight within the range of about 1.5 million to about 5 million wherein said ingredients are present in proportions such that the composition is a jelly-like gel.

2. A composition according to claim 1 wherein said oil is mineral oil.

3. A composition according to claim 1 wherein the molecular weight of said polyethylene is within the range of about 1.5 million to about 3.5 million.

4. A composition according to claim 1 wherein the amount of said oil comprising said composition is about 95 to less than about 99% by weight and wherein the amount of said polyethylene comprising said composition is greater than about 1 to about 5 percent by weight.

5. A composition according to claim 1 comprising about 79 to about 99 percent by weight of said oil; greater than about 1 to about 5 percent by weight of said polyethylene; and about 1% to about 20 percent by weight of filler.

6. A composition according to claim 5 wherein said filler is a finely divided material selected from the group consisting of molybdenum disulfide; nylon; polyaryl carbonate and polyacetal.

7. A composition according to claim 5 wherein said oil is a mineral oil.

8. A composition according to claim 7 wherein the molecular weight of said polyethylene is about 1.5 million to about 3.5 million.

9. A jelly-like gel lubricating composition comprising about 95 to less than about 99 percent by weight of a mineral oil of lubricating viscosity and greater than about 1 to about 5 percent by weight of polyethylene having an average molecular weight within the range of of about 1.5 million to about 5 million.

10. A composition according to claim 9 wherein the molecular weight of said polyethylene is within the range of about 1.5 million to about 3.5 million.

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