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LUBRICANT COMPOSITIONS

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ABSTRACT OF THE DISCLOSURE

Lubricant compositions containing a polyester are prepared from short-chain dicarboxylic acids containing from 6 to 13 carbon atoms, polyols, and monobasic carboxylic acid chain terminators refined with a glycidyl ester of an aliphatic acid having from about 5 to 22 carbon atoms.

BACKGROUND OF INVENTION

(1) Field of invention

This invention is concerned with lubricant compositions and, more particularly, with synthetic lubricant compositions containing a polyester thickening agent prepared from short-chain dicarboxylic acids, polyols, and monobasic carboxylic acid chain terminators that have been refined with a glycidyl ester of an aliphatic acid having from 5 to 22 carbon atoms.

(2) Prior art

Synthetic ester lubricants have been widely used in gas turbine and internal combustion engines. However, one disadvantage of these lubricants is that the viscosity tends to thin out excessively at high temperatures. Because of the wide temperature range encountered in the operation of an engine, it is desirable that the viscosity should vary only slightly with the temperature of the fluid. The resistance of a lubricant to viscosity change is known as the V.I. (viscosity index). In general, the greater the V.I., the more desirable the lubricant because of the greater resistance to thickening at low temperatures and to thinning out at high temperatures.

A number of additives have been used in the past in an attempt to improve the V.I. of synthetic lubricants. Included among these additives have been a number of polymeric materials, such as polyisobutenes, polymethacrylates, polyvinyl acetates, and polyacrylates.

However, the polymeric V.I. improving compounds mentioned above have been unsatisfactory in engines in which there is great shear since under high shear conditions, they tend to break apart and lost their ability to thicken the lubricating fluid.

Polyester additives prepared from polyols, short-chain dicarboxylic acids, and monobasic carboxylic acid terminators have been found to resist shear and have as a result been used to some extent as lubricant V.I. improvers. One problem with these esters is that they generally contain an excessive amount of acidity in the form of partially reacted dicarboxylic acid.

A number of methods have been attempted in the past to reduce the acidity of esters. One method involves the treatment of the polyester with an aqueous alkali solution, such as potassium carbonate or sodium hydroxide, or the percolating of the ester reaction product through an alkaline bed. While being effective in reducing the acid value, the alkali treatment has not been entirely satisfactory. The short-chain partially reacted dicarboxylic acids form salts or soaps which are more soluble in the ester than in water and thus cannot be removed when conventional alkali refining is attempted.

Another method which has been for the removal of excess acidity has been the treatment of the unrefined polyester lubricant with short-chain alkylene oxides, such as propylene oxide or ethylene oxide. One of the drawbacks to this process is that the reaction product of alkylene oxides, particularly the lower alkylene oxides, and the free acid is a material which is generally unstable under high temperature operating conditions and either decomposes, resulting in the contamination of the lubricant composition, or gives a lubricant having excessive volatility. Another problem connected with the use of alkylene oxides is their tendency to polymerize to materials which are difficult to remove.

In addition, in many applications the synthetic lubricant must exhibit good oxidative and thermal stability.

Surprisingly, we have now found that new synthetic lubricant thickener compositions exhibiting good oxidative and thermal stability, excellent shear resistance, and low acid value can be prepared by refining a short-chain polyester with an aliphatic glycidyl ester.

SUMMARY OF INVENTION

This invention relates to lubricant compositions containing novel thickeners comprising relatively low molecular weight polyesters which have been refined with a glycidyl ester of an aliphatic acid having from 5 to 22 carbon atoms.

DESCRIPTION OF INVENTION

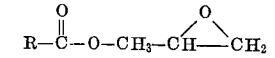
The polyester thickeners suitable for refining with a glycidyl ester in accordance with the present invention may be prepared by reacting short-chain dicarboxylic acids, polyols, and monobasic carboxylic acids. Dibasic acids used in the preparation of the polyesters contain from about 6 to 13 carbon atoms, including the following: azelaic, adipic, sebacic, dodecanedioic, and brassylic. The preferred acid is azelaic.

The monobasic acids used in the preparation of the polyesters contain from about 3 to 10 carbons. Preferably the acids are saturated aliphatic monobasic acids.

The applicable polyols may be short-chain glycols, such as propylene glycol, ethylene glycol and diethylene glycol, preferably hindered glycols, such as neopentyl glycol, and 2,2,4-trimethylpentanediol-1,3 and polyols such as trimethylolpropane, trimethylolethane, and pentaerythritol. After the preparation the polyester contains excess acidity in the form of partially reacted dicarboxylic acid which cannot be introduced into a lubricant composition. The glycidyl ester is added in excess of the amount needed to neutralize the excess acidity of the polyester and acts as an acid "scavenger."

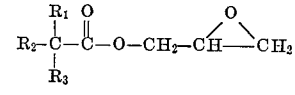
The glycidyl esters which may be used in the practice of the present invention include the glycidyl esters of normal or branched-chain aliphatic acids containing from 5 to about 22 carbon atoms. Preferably, the compounds utilized to form the acid moiety of suitable glycidyl esters are neo-acids; that is, acids in which the alpha carbon atoms are completely substituted with alkyl groups.

The glycidyl esters, as shown in the following formula, may be formed by the reaction of epichlorohydrin and an alkali metal salt of the selected acid.



wherein R is a branched or straight chain alkyl group having from 4 to 21 carbon atoms.

The preferred acid scavenging glycidyl ester is a compound having the general formula:



wherein R_1, R_2 and R_3 are all saturated alkyl groups and contain a total of about 7 to 9 carbon atoms. The glycidyl esters having this structure produce, when reacted with

the free acid of the ester thickener, materials which are particularly stable and compatible with the lubricant system. Other glycidyl esters which may be used as acid scavengers, in addition to the neo-acid esters, are the glycidyl esters of pelargonic acid, valeric acid, isostearic acid, and oleic acid.

The polyester thickeners are refined in accordance with the method described in United States patent application Ser. No. 650,185, filed June 30, 1967, now U.S. Pat. 3,485,754 dated Dec. 23, 1967. In general, the excess monobasic acid preparation of the polyester is removed by heating the reaction mixture to a temperature of from 180 to 240° C. with a pressure of about 1 to 2 torr. The polyester is then cooled and the acid value measured. A 100% excess of the stoichiometric amount of the glycidyl ester needed to neutralize the acid present is added to the polyester. Reaction is continued until the acid value of the polyester is reduced to a value of less than 0.05. The excess glycidyl ester is removed by heating the mixture from about 240 to 260° C. and applying a pressure of 1 to 2 torr.

The base fluids for the lubricant compositions may be either paraffins or synthetic esters. When the base fluid is an ester it may be prepared from various combinations of aliphatic monocarboxylic acids and dicarboxylic acids having from about 6 to 36 carbon atoms, and monohydric and polyhydric aliphatic alcohols having from about 4 to about 20 carbon atoms. Specific examples of esters which may be used are: di-iso-octyl adipate, di-2 ethylhexyl azelate, didecyl azelate, di-isodecyl azelate, diisodecyl adipate, ditridecyl adipate, di-iso-octyl sebacate, di-isodecyl sebacate, di-2-ethylhexyl sebacate, di-iso-octyl dodecanedioate, di-2-ethylhexyl brassylate, and di-iso-octyl brassylate. Other esters may also be used as synthetic lubricants that are made from hindered polyols and mono- and polycarboxylic acids of about 5 to 13 carbon atoms. Specific examples include: esters of monopentaerythritol and an acid blend comprised of 70% by weight of isovaleric acid and 30% by weight pelargonic acid; monopentaerythritol and an acid blend of 50% by weight of isovaleric acid, 25% by weight of pelargonic acid, and 25% by weight of a mixture of acids with an average of C_7 chain length; trimethylol pentane and two moles of pelargonic acid and one mole of a mixture of monocarboxylic acids with C_7 chain length; and trimethylol pentane and three moles of a mixture of monocarboxylic acids with an average of C_7 chain length. Among other complex esters used are those having a structural formula such as X—Y—Z—Y—X, wherein X is a monohydric alcohol radical, Y is a dibasic acid radical, and Z is a glycol radical; or a structural formula such as A—B—C—B—A, wherein A is a monocarboxylic acid radical, B is a polyol radical, and C is a dicarboxylic acid radical. Examples of these complex esters are the reaction products of 2-ethyl-1, 3-hexanediol, sebacic acid, and 2-ethylhexanoic acid; adipic acid, diethylene glycol, and pelargonic acid; sebacic acid, trimethylolethane, and hexanoic acid; sebacic acid, 1,3,5,7 - octanetetrol and pentanoic acid; and diethylene glycol, adipic acid, and ethylhexanol.

The average molecular weight of the polyester thickeners used in the lubricant compositions of this invention have a range of about 500 to 1500 and preferably from about 900 to 1200.

The polyester thickeners may be used in an amount of about 0.5 to 30.0 percent by weight of the composition and preferably from about 1.0 to 15.0 percent by weight based on the total weight of the lubricant composition.

To further illustrate the lubricant compositions of the present invention, the following examples are provided. It is to be understood that these examples are illustrative only and are not to be considered as limiting the scope of this invention in any manner.

EXAMPLE 1

Preparation of polyester (1336–48)

Azelaic acid (1 mole), pentaerythritol (2 moles), and 4 moles of a low molecular weight monobasic acid averaging seven carbon atoms (sold under the trademark EMFAC 1210) were charged to a 5-liter/3-neck flask equipped with a stirrer, thermometer, water trap, and condenser. Reaction was continued until most of the theoretical water of esterification was removed, that is, for about 4 hours at a maximum temperature of 235° C. under 28–29 inches mercury vacuum. An additional 3 moles of EMFAC 1210 was added. The reaction was maintained at 235° C. under 28–29 inches mercury vacuum for about another 4 hours and additional water of reaction removed. Excess acids were stripped off at 225° C. under 1–2 mm. mercury pressure. At this point the acid value was 5.65 and the hydroxyl value 8.2. 100% excess of the glycidyl ester of a neo-acid (sold under the trademark Cardura E) was added and the mixture heated to 220° C. for 1 hour. At this time the acid value had dropped to 0.05. The excess Cardura E was stripped off at a temperature of 245° C. at 1–2 mm. Hg pressure. The final acid value was 0.04, and the final hydroxyl value 12.4.

EXAMPLE 2

Preparation of polyester (1336–27)

The procedure of Example 1 was followed except 2 moles of trimethylol pentane were substituted for the amount of pentaerythritol and only 4 moles of EMFAC 1210 were utilized.

EXAMPLES 3–6

Lubricant compositions were prepared in accordance with the formulations shown in Table I below. These compositions were evaluated using Federal Test 791, Corrosion and Oxidation Stability, Method 5308, run at 450° F. for 48 hours, using the following five metals; bronze, titanium alloy, steel, aluminum alloy, and silver. The data in Table II shows that the change in metal weight was not great, and that the change in metal weight was within the specifications of General Electric D50TF3–S3, which allows a maximum change in metal weight of ±0.2 mg./cm.^{2}.

Lubricant compositions containing polyesters which have not been refined have excess acidity and consequent corrosive propensities which render them of marginal utility.

TABLE I

Example number	4	5	6
Pentaerythritol ester [1]	[2] 94.93	87.81	87.80
Polyester of—			
Example 1		7.12	
Example 2			7.12
Commercial anti-oxidants	2.00	2.00	2.00
Commercial metal deactivator	.05	.05	.05
Quinizarin	.02	.02	.02
Tri-cresyl phosphate	3.00	3.00	3.00

[1] An ester prepared from monopentaerythritol and a mixture of C_5 to C_9 monobasic acids.
[2] Weight percent.

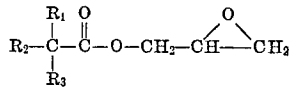
TABLE II

Example number	4	5	6
Bronze corrosion (mg./cm.^{2})	0	−0.02	−0.02
Steel corrosion (mg./cm.^{2})	+0.02	+0.01	+0.02
Silver corrosion (mg./cm.)	−0.04	−0.04	−0.05
Titanium corrosion (mg./cm.^{2})	−0.02	0	0
Aluminum corrosion (mg./cm.)	−0.02	−0.01	−0.02
Viscosity change at 100° F. (percent)	71.4	61.0	72.6
Acid value (mg. KOH/g.)	9.2	8.5	11.4

As will be evident to those skilled in the art, various modifications of the present invention can be made or followed in the light of the foregoing disclosure and discussion without departing from the spirit or scope of the following claims.

I claim:

1. In a process for preparing lubricant compositions comprised of a synthetic ester fluid prepared from aliphatic monocarboxylic acids and dicarboxylic acids having from 6 to 36 carbon atoms and monohydric and polyhydric aliphatic alcohols having from 4 to 20 carbon atoms and from about 0.5% to 30% by weight of a polyester thickener having a molecular weight of 500 to 1500 and prepared by the reaction of (a) azelaic acid, (b) a polyol selected from the group consisting of neopentyl glycol, trimethylolpropane, pentaerythritol, diethylene glycol and trimethylolethane and (c) a saturated aliphatic monobasic acid containing from 3 to 10 carbon atoms, in a molar ratio 1:2:4–7, respectively, the improvement comprising refining said polyester thickener to an acid value less than about 0.05 by treating with a glycidyl ester of a neo-acid having the formula



wherein R_1, R_2 and R_3 are saturated alkyl groups having a total of 7 to 9 carbon atoms.

2. The process of claim 1 wherein the molecular weight of the polyester thickener is about 900 to 1200 and said thickener comprises about 1% to 15% by weight of the lubricant composition.

3. The process of claim 1 wherein the polyester thickener is prepared from azelaic acid, pentaerythritol and a mixture of saturated aliphatic monobasic acids averaging about 7 carbon atoms.

4. The process of claim 1 wherein the polyester thickener is prepared from azelaic acid, trimethylolpropane and a mixture of saturated aliphatic monobasic acids averaging about 7 carbon atoms.

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U.S. Cl. X.R.

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