

Description

This invention relates to a method for producing a multi-functional lubricant additive which is a VI improver and a dispersant additive when employed in a lubricating oil composition.

The art is replete with disclosures on the use of polymer additives in lubricating oil compositions. Ethylene-propylene copolymers and ethylene-alpha olefin non-conjugated diene terpolymers, which have been further derivatized to provide bifunctional properties in lubricating oil compositions, illustrate this type of oil additive.

US-A-3-522-180 discloses a method for the preparation of an ethylene-propylene copolymer substrate effective as a viscosity index improver for lubricating oils.

US-A-4-026-809 discloses graft copolymers of a methacrylate ester and an ethylene-propylene-alkylidene norbornene terpolymer as a viscosity index improver for lubricating oils.

US-A-4-089-794 discloses ethylene copolymers derived from ethylene and one or more C₃ to C₂₈ alpha olefins, solution grafted with an ethylenically-unsaturated carboxylic acid material, followed by a reaction with a polyfunctional material reactive with carboxyl groups, such as a polyamine, a polyol, or a hydroxyamine. The reaction product is useful as a sludge-and varnish-control additive in lubricating oils.

US-A-4-137-185 discloses a stabilized imide graft of an ethylene copolymer additive for lubricants.

US-A-4-146-489 discloses a graft copolymer where the backbone polymer is an oil-soluble ethylene-propylene copolymer or an ethylene-propylene-diene modified terpolymer, and the graft monomer is C-vinylpyrrolidone or N-vinylpyrrolidone, providing a dispersant VI improver for lubricating oils.

US-A-4-320-019 discloses a multipurpose lubricating additive prepared by the reaction of an interpolymer of ethylene and a C₃ - C₈ alpha-monoolefin with an olefinic carboxylic acid acylating agent to form an acylating reaction intermediate, which is then reacted with an amine.

US-A-4-340-689 discloses a process for grafting a functional organic group onto an ethylene copolymer or an ethylene-propylene-diene terpolymer.

US-A-4-357-250 discloses a reaction product of a copolymer and an olefin carboxylic acid obtained via the "ene" reaction, followed by reaction with a monoamine-polyamine mixture.

US-A-4-382-007 discloses a dispersant - VI improver prepared by reacting a polyamine-derived dispersant with an oxidized ethylene-propylene polymer or an ethylene-propylene diene terpolymer.

US-A-4-144-181 discloses polymer additives for fuels and lubricants comprising a grafted ethylene copolymer reacted with a polyamine, polyol or hydroxyamine and finally reacted with an alkaryl sulfonic acid.

EP-A-0225048 (Exxon) describes a VI improver wherein hydrocarbon polymers are reacted with unsaturated nitrogen-containing monomers or unsaturated carboxylic acids in the presence of a free-radical initiator. The monomers include pyridines.

EP-A-0171167 (Exxon) describes a VI improver wherein hydrocarbon polymers are reacted with unsaturated nitrogen-containing monomers or unsaturated carboxylic acids in the presence of a free-radical initiator. The monomers include pyridines and pyrimidines.

EP-A-0002286 (Rohm and Haas) describes graft copolymers wherein the backbone comprises a hydrocarbon polymer, the backbone being treated with, e.g. maleic anhydride and a polyamino compound. The polyamino compound may be a pyridine.

US-A-4-735-736 discloses grafted hydrocarbon polymers subsequently reacted with a polymamine and treated and/or reacted with a monoamine.

US-A-4-780-228 discloses the grafting of a hydrocarbon polymer, in the absence of a solvent and in the presence of a free radical initiator, and a claim-stopping agent followed by a reaction with an amine, polyol or an aminoalcohol.

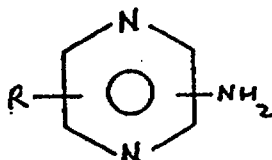
An object of this invention is to provide a novel derivatized graft copolymer composition.

Another object of the invention is to provide a multi-functional lubricant additive effective for imparting viscosity index, and dispersancy properties to a lubricating oil composition.

The present invention provides:

A) reacting a polymer comprising from 15 to 80 mole percent of ethylene, from 20 to 85 mole percent of a C₃-C₁₀ alphanonoolefin, and from 0 to 15 mole percent of a non-conjugated diene or triene, said polymer having a number average molecular weight from 5,000 to 500,000, with at least one olefinic carboxylic acid acylating agent to form one or more acylating reaction intermediates having a carboxylic acid acylating function within their structure; and,

B) reacting said reaction intermediate from (A) with an aminopyrazines having the formula:



in which R is hydrogen or an alkyl or alkoxy radical having 1 to 18 carbon atoms.

The invention also provides a lubricating oil composition which comprises an oil of lubricating viscosity and an effective amount of the above-described reaction product. The lubricating oil has viscosity index improver and dispersancy properties.

The invention also provides a concentrate for a lubricating oil comprising a diluent oil of lubricating viscosity and 1 to 50 weight percent, of the reaction product described above.

The polymer or copolymer substrate employed in the invention may be prepared from ethylene and propylene or it may be prepared from ethylene and a higher olefin within the range of C_3 to C_{10} alpha-monoolefins.

More complex polymer substrates, often designated as interpolymers, may be prepared using a third component. The third component generally used to prepare an interpolymer substrate is a non-conjugated diene or triene. The non-conjugated diene component preferably has 5 to 14 carbon atoms in the chain. Preferably, the diene monomer has a vinyl group in its structure and can be a cyclic or bi-cyclic compound. Representative dienes include 1,4-hexadiene, 1,4-cyclohexadiene, dicyclopentadiene, 5-ethylidene-2-norbornene, 5-methylene-2-norbornene, 1,5-heptadiene, and 1,6-octadiene. A mixture of more than one diene can be used in the preparation of the interpolymer. A preferred non-conjugated diene for preparing a terpolymer or interpolymer substrate is 1,4-hexadiene.

The triene component will have at least three non-conjugated double bonds, and preferably have up to 30 carbon atoms in the chain. Typical trienes useful in preparing the interpolymer of the invention are 1-isopropylidene-3a,4,7,7a-tetrahydroindene, 1-isopropylidenedicyclopentadiene, dehydro-isodicyclopentadiene, and 2-(2-methylene-4-methyl-3-pentenyl)-[2.2.1] bicyclo-5-heptene.

The polymerization reaction to form the polymer substrate is generally carried out in the presence of a catalyst in a solvent medium. The polymerization solvent may be any suitable inert organic solvent that is liquid under reaction conditions for solution polymerization of monoolefins, which is generally conducted in the presence of a Ziegler type catalyst. Examples of satisfactory hydrocarbon solvents include straight chain paraffins having 5 to 8 carbon atoms, with hexane being preferred. Aromatic hydrocarbons, preferably aromatic hydrocarbons having a single benzene nucleus, such as benzene, toluene and the like; and saturated cyclic hydrocarbons having boiling point ranges approximating those of the straight chain paraffinic hydrocarbons and aromatic hydrocarbons described above, are particularly suitable. The solvent selected may be a mixture of one or more of the foregoing hydrocarbons. It is desirable that the solvent be free of substances that will interfere with a Ziegler polymerization reaction.

In a typical preparation of a polymer substrate, hexane is first introduced into a reactor and the temperature in the reactor is raised moderately to 30°C. Dry propylene is fed to the reactor until the pressure reaches 40-45 inches of mercury (135 to 152 KPa). The pressure is then increased to 60 inches of mercury (203 KPa), and dry ethylene and 5-ethylidene-2-norbornene are fed to the reactor. The monomer feeds are stopped and a mixture of aluminum sesquichloride and vanadium oxytrichloride are added to initiate the polymerization reaction. Completion of the polymerization reaction is evidenced by a drop in the pressure in the reactor.

It is preferred that the polymer comprises from 15 to 80 mole percent ethylene and from 20 to 85 mole percent of a C_3 to C_8 alpha-monoolefin.

Most preferably polymer comprises from 25 to 55 mole percent of ethylene and from 45 to 75 mole percent of propylene.

Terpolymer variations of the foregoing polymers may contain from 0.1 to 10 mole percent of a non-conjugated diene or triene.

The polymer substrate, that is the ethylene copolymer or terpolymer is an oil-soluble, substantially linear, rubbery material, having a number average molecular weight from 5,000 to 500,000, with a preferred number average molecular weight range of 25,000 to 250,000, and a most preferred range from 50,000 to 150,000.

The terms polymer and copolymer are used generically to encompass ethylene copolymers, terpolymers or interpolymers. These materials may contain minor amounts of other olefinic monomers so long as their basic characteristics are not materially changed.

An ethylenically unsaturated carboxylic acid material is next grafted onto the prescribed polymer backbone. These materials which are attached to the polymer contain at least one ethylenic bond and at least one, preferably two, carboxylic acid or its anhydride groups or a polar group which is convertible into said carboxyl

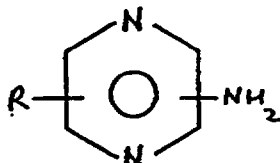
groups by oxidation or hydrolysis. Maleic anhydride or a derivative thereof is preferred. It grafts onto the ethylene copolymer or terpolymer to give two carboxylic acid functionalities per monomer unit. Examples of additional unsaturated carboxylic materials include chloromaleic anhydride, itaconic anhydride, or the corresponding dicarboxylic acids, such as maleic acid, fumaric acid, and their monoesters.

The ethylenically unsaturated carboxylic acid material may be grafted onto the polymer backbone in a number of ways. It may be grafted onto the backbone by a thermal process known as the "ene" process, or by grafting in solution form using a radical initiator. The free-radical induced grafting of ethylenically unsaturated carboxylic acid materials in solvents, such as benzene, is a preferred method. It is generally carried out at an elevated temperature in the range of 100°C to 250°C preferably 120°C to 190°C, and more preferably at 150 to 180°C, e.g. above 160°C, in a solvent, preferably a mineral lubricating oil solution, containing, e.g. 1 to 50 wt.%, more preferably 5 to 30 wt.%, based on the initial total oil solution, of the ethylene polymer, and preferably under an inert environment.

The free-radical initiators which may be used are peroxides, hydroperoxides, and azo compounds and preferably those which have a boiling point greater than 100°C and decompose thermally within the grafting temperature range to provide free radicals. Representative of these free-radical initiators are azobutyronitrile and -2,5-dimethyl-hex-3-yne-2, 5 bis-tertiary-butyl peroxide. The initiator is generally used in an amount of between 0.005% and 1% by weight, based on the weight of the reaction mixture solution. The grafting is preferably carried out in an inert atmosphere, such as under nitrogen. The resulting polymer intermediate is characterized by having carboxylic acid acylating functions within its structure.

Polymer substrates or interpolymers are available commercially. Particularly useful are those containing from 40 to 60 mole percent of ethylene units, and 60 to 40 mole percent of propylene units. Examples are "Ortholeum 2052" - Trade Marks and "PL-1256" - Trade Marks, available from E.I. duPont de Nemours and Co. The former is a terpolymer containing 48 mole percent of ethylene units, 48 mole percent of propylene units and 4 mole percent of 1,4-hexadiene units. The average molecular weights of the two are of the order of 200,000 and 280,000, respectively.

The polymer intermediate possessing carboxylic acid acylating functions is reacted with an amino pyrazines represented by the formulae:



in which R represents hydrogen or an alkyl or alkoxy radical having from 1 to 18 carbon atoms, more preferably 1 to 5 carbon atoms.

The reaction between the polymer substrate intermediate with the grafted carboxylic acid acylating function and the prescribed aminopyrazine compound is conducted by heating a solution of the polymer substrate under inert conditions and then adding the aminopyrazine to the heated solution, generally with mixing to effect the reaction. It is convenient to employ an oil solution of the polymer substrate heated at 140 to 175°C while maintaining the solution under nitrogen.

The following examples illustrate the preparation of the novel reaction product additive of the invention.

Example

72.0 grams of the solid maleic anhydride graft polymer of Example 1 were mixed in 528 grams of solvent neutral oil at 160°C with stirring under nitrogen until dissolved. Stirring was continued for an additional hour under the same conditions. Aminopyrazine (0.80 grams) was then added to the rubber-oil solution. The reaction was continued with stirring at 160°C for three hours under nitrogen. The reaction mixture was cooled to 100°C and filtered.

The graft and derivatized polymers obtained according to the invention are useful as an additive for lubricating oils. They are multi-functional additives for lubricants, being effective to provide dispersancy, and viscosity index improvement properties to lubricating oils. They can be employed in a variety of oils of lubricating viscosity, including natural and synthetic lubricating oils, and mixtures thereof. The novel additives can be employed in crankcase lubricating oils for spark-ignition and compression-ignition internal combustion engines. The compositions can also be used in gas engines, or turbines, automatic transmission fluids, gear lubricants, metal-working lubricants, hydraulic fluids and other lubricating oil and grease compositions. They may also be

used in motor fuel compositions.

The base oil may be a natural oil including liquid petroleum oils and solvent-treated or acid-treated mineral lubricating oils of the paraffinic, naphthenic and mixed paraffinic-naphthenic types.

In general, the lubricating oil composition of the invention will contain the novel reaction product in an amount from 0.1 to 30 weight percent. A preferred concentration range for the additive is from 0.1 to 10 weight percent, based on the total weight of the oil composition.

Oil concentrates of the additives may contain from 1 to 50 weight percent of the additive reaction product in a carrier or diluent oil of lubricating oil viscosity.

The reaction product of the invention may be employed in lubricant compositions together with conventional lubricant additives. Such additives may include additional dispersants, detergents, anti-oxidants, pour point depressants, anti-wear agents, and the like.

The additive reaction product of the invention was tested for its effectiveness as a dispersant in a formulated lubricating oil composition. In the examples, the polymer substrate employed was the MA-EPM-rubber comprising 60 mole percent ethylene, 40 mole percent propylene having a number average molecular weight of 80,000, and 1.0 weight percent of maleic anhydride. The base lubricating oil used in the dispersancy test was a typical formulated lubricating oil as represented by the values set forth in Table 1.

Table 1

<u>Component</u>	<u>Parts by Weight</u>
Solvent neutral oil A	75.25
Solvent neutral oil B	21.64
Zinc Dialkyldithiophosphate	1.22
4,4'-dinonyldiphenylamine	0.39
Overbasedmagnesium sulfonate	1.50
Silicone Antifoamant	150 PPM
<u>Product Analyses</u>	
Viscosity Kin 40°C CS (m ² /s)	30.4 (30.4x10 ⁻⁶)
Viscosity Kin 100°C CS (m ² /s)	5.33 (5.33x10 ⁻⁶)
Pour Point, °F/°C	+10/-12
Ash Sulfated, % D874	0.88
Phosphorus, % X-Ray	0.12
Sulfar, % X-Ray Total	0.32
Zinc, % X-Ray	0.13

Oil A had a specific gravity 60/60°F (15/15°C) of 0.858 to 0.868; a viscosity at 100°F (40°C) of 123 to 133 mm²/s and a Pour Point of 0°F (-18°C). Oil B had a specific gravity of 0.871 to 0.887; a viscosity of 325-350 mm²/s and a Pour Point of +10°F (-12°C). Zinc salt is a salt of mixed alcohols-isopropanol and P₂S₅ product as de-

scribed in US-A-3-292-181. The overbased magnesium sulfonate had a total base number of 395 and is a salt of branched C₂₀-C₄₀ monoalkylbenzene sulfuric acid (MW 530-540) together with about 10% of magnesium carbonate, 14% of magnesium hydroxide and 4% of magnesium sulfate.

The dispersant properties of the additive-containing oil are determined in the Bench VC Dispersancy Test (BVCT). Dispersancy of a lubricating oil is determined relative to three references which are the results from three standard blends tested along with the unknown. The test additives were blended into a formulated oil containing no dispersant. The additive reaction product was employed in the oil at a concentration of 12.0 weight percent polymer solution.

The product prepared in the Example was blended into formulated oils not containing a dispersant to form 1.20 wt% polymer solutions. These blends were tested for dispersancy in the Bench VC Test (Table 2). In this test, dispersancy is compared to that of three reference oils which are tested along with the experimental samples. The numerical value of a test result decreases with an increase of dispersant effectiveness, i.e. the lower the value the better the performance.

TABLE 2

VI Improver Performance

<u>VI Improver</u>	<u>BVCT</u> ¹
Example	38
EPM - (Ethylene Propylene Monomer)	99
MA-EPM (underivatized) - (Maleic Anhydride EPM)	88
Commercial NVP (N-vinylpyrrolidone)	
grafted DOCP (Dispersant Olefin Copolymer)	68

The EPM base rubber and molecular weight average are similar in all examples in Table 2.

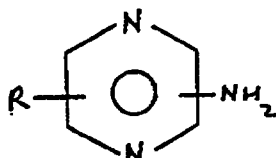
The results from the BVCT Test show that the dispersant VI improvers prepared in the Example gave better dispersancy performance than the corresponding EPM (without the aminoaromatic compound derivative) and equivalent performance to a commercial NVP-grafted DOCP VI Improver.

Claims

1. A method of preparing a polymeric lubricant additive which comprises:

A) reacting a polymer comprising from 15 to 80 mole percent of ethylene, from 20 to 85 mole percent of a C₃-C₁₀ alpha-monoolefin, and from 0 to 15 mole percent of a non-conjugated diene or triene, said polymer having a number average molecular weight from 5,000 to 500,000, with at least one olefinic carboxylic acid acylating agent to form one or more acylating reaction intermediates having a carboxylic acid acylating function within their structure; and,

B) reacting said reaction intermediate from (A) with an aminopyrazine having the formula:



in which R is hydrogen or an alkyl or alkoxy radical having 1 to 18 carbon atoms.

2. A method according to Claim 1 characterised in that the polymer has a number average molecular weight from 25,000 to 500,000.



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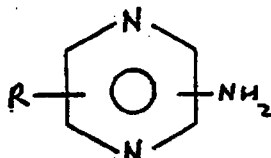
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Claims

1. A method of preparing a polymeric lubricant additive which comprises:

A) reacting a polymer comprising from 15 to 80 mole percent of ethylene, from 20 to 85 mole percent of a C₃-C₁₀ alpha-monoolefin, and from 0 to 15 mole percent of a non-conjugated diene or triene, said polymer having a number average molecular weight from 5,000 to 500,000, with at least one olefinic carboxylic acid acylating agent to form one or more acylating reaction intermediates having a carboxylic acid acylating function within their structure; and,

B) reacting said reaction intermediate from (A) with an aminopyrazine having the formula:



in which R is hydrogen or an alkyl or alkoxy radical having 1 to 18 carbon atoms.

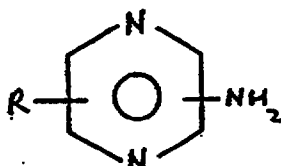
2. A method according to Claim 1 characterised in that the polymer has a number average molecular weight from 25,000 to 500,000.



3. A method according to Claim 1 or 2 characterised in that the polymer comprises from 15 to 80 mole percent ethylene and from 20 to 85 mole percent of a C₃ to C₈ alpha-monoolefin.
4. A method according to Claim 3 characterised in that the polymer contains from 0.1 to 10 mole percent of the non-conjugated diene or triene.
5. A method according to Claim 1 characterised in that the polymer comprises from 25 to 55 mole percent of ethylene and from 45 to 75 mole percent of propylene.
6. A method according to any one of Claims 1 to 5 characterised in that the olefinic carboxylic acid acylating agent is maleic anhydride or itaconic anhydride, chloromaleic anhydride, maleic acid, fumaric acid or a corresponding ester.
7. A lubricating oil composition comprising an oil of lubricating viscosity and from 1 to 50 wt%, based on the total weight of composition of an additive produced by a method according to any one of Claims 1 to 6.
8. A lubricating oil composition according to Claim 7 characterised in that it contains from 0.1 to 10 weight percent of the additive, based on the total weight of the oil composition.

Patentansprüche

1. Verfahren zur Herstellung eines polymeren Schmiermittelzusatzes, welches umfaßt, daß:
 - A) ein Polymer, das von 15 bis 80 Molprozent Ethylen, von 20 bis 85 Molprozent eines C₃-C₁₀-Alpha-Monoolefins und von 0 bis 15 Molprozent eines nicht-konjugierten Diens oder Triens umfaßt, wobei besagtes Polymer im Zahlenmittel ein Molekulargewicht von 5000 bis 500.000 besitzt, mit wenigstens einem Acylierungsmittel auf der Basis einer olefinischen Carbonsäure zur Reaktion gebracht wird, um eine oder mehrere Acylierungsreaktionszwischenprodukte mit einer Carbonsäure-Acylierungsfunktion in ihrer Struktur zu bilden; und
 - B) besagtes Reaktionszwischenprodukt aus (A) mit einem Aminopyrazin mit der Formel:



in der R Wasserstoff oder ein Alkyl- oder Alkoxyrest mit 1 bis 18 Kohlenstoffatomen ist, zur Reaktion gebracht wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Polymer im Zahlenmittel ein Molekulargewicht von 25.000 bis 500.000 hat.
3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß das Polymer von 15 bis 80 Molprozent Ethylen und von 20 bis 85 Molprozent eines C₃-C₈-Alpha-Monoolefins umfaßt.
4. Verfahren nach Anspruch 3, dadurch gekennzeichnet, daß das Polymer von 0,1 bis 10 Molprozent des nicht-konjugierten Diens oder Triens enthält.
5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Polymer von 25 bis 55 Molprozent Ethylen und von 45 bis 75 Molprozent Propylen umfaßt.
6. Verfahren nach einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß das Acylierungsmittel auf der Basis einer olefinischen Carbonsäure Maleinsäureanhydrid oder Itaconsäureanhydrid, Chlormaleinsäureanhydrid, Maleinsäure, Fumarsäure oder ein entsprechender Ester ist.
7. Schmierölzusammensetzung, die ein Öl mit Schmierviskosität und von 1 bis 50 Gew.-%, bezogen auf das Gesamtgewicht der Zusammensetzung, eines nach einem Verfahren gemäß einem der Ansprüche 1 bis



6 hergestellten Zusatzes umfaßt.

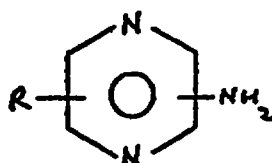
8. Schmierölzusammensetzung nach Anspruch 7, dadurch gekennzeichnet, daß sie von 0,1 bis 10 Gew.-% des Zusatzes enthält, bezogen auf das Gesamtgewicht der Ölzusammensetzung.

Revendications

1. Procédé de préparation d'un additif lubrifiant polymérique qui consiste à :

A) faire réagir un polymère comprenant de 15 à 80 pour cent en mole d'éthylène, de 20 à 85 pour cent en mole d'une alphamonooléfine C_3-C_{10} , et de 0 à 15 pour cent en mole d'un diène non conjugué ou d'un triène, ledit polymère ayant un poids moléculaire moyen numérique de 5000 à 500 000, ayant au moins un agent d'acylation de l'acide carboxylique oléfinique pour former un intermédiaire de réaction d'acylation ou plus ayant une fonction d'acylation de l'acide carboxylique à l'intérieur de leur structure ; et

B) faire réagir ledit intermédiaire de réaction issu de (A) avec une aminopyrazine ayant la formule :



dans laquelle R est un hydrogène ou un radical alkyle ou alkoyle ayant de 1 à 18 atomes de carbone.

2. Procédé selon la revendication 1 caractérisé en ce que le polymère a un poids moléculaire moyen en nombre allant de 25 000 à 500 000.
3. Procédé selon la revendication 1 ou 2 caractérisé en ce que le polymère comprend de 15 à 80 pour cent en mole d'éthylène et de 20 à 85 pour cent en mole d'une alpha-monooléfine C_3-C_8 .
4. Procédé selon la revendication 3 caractérisé en ce que le polymère contient de 0,1 à 10 pour cent en mole du diène non conjugué ou du triène.
5. Procédé selon la revendication 1 caractérisé en ce que le polymère comporte de 25 à 55 pour cent en mole d'éthylène et de 45 à 75 pour cent en mole de propylène.
6. Procédé selon l'une quelconque des revendication 1 à 5 caractérisé en ce que l'agent d'acylation de l'acide carboxylique oléfinique est l'anhydride maléique, ou l'anhydride itaconique, l'anhydride chloromaléique, l'acide maléique, l'acide fumérique ou un ester correspondant.
7. Composition d'huile lubrifiante comprenant une huile d'une viscosité lubrifiante et de 1 à 50% en poids, sur la base du poids total de la composition d'un additif produit par un procédé suivant l'une quelconque des revendications 1 à 6.
8. Composition d'huile lubrifiante selon la revendication 7, caractérisée en ce qu'elle contient de 0,1 à 10 pour cent en poids de l'additif, sur la base du poids total de la composition d'huile.