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USE OF ESTERS IN A LUBRICANT COMPOSITION FOR IMPROVING THE CLEANLINESS OF AN ENGINE

The present invention relates to the use of esters in a lubricant composition for improving the cleanliness of an engine, in particular marine engines. The present invention also relates to a method for improving the cleanliness of an engine, in particular marine engines, comprising the use of a lubricant composition comprising esters.

In the maritime field, significant efforts are concentrated on lubricant compositions in order to neutralize sulfuric acid formed during the combustion of fuel oil which allows corrosive wear of engine parts to be significantly diminished.

To respond to said issue of decreasing corrosive wear, many additives have been used in the lubricant compositions and can have deleterious effects on the cleanliness of engine parts, in particular the cleanliness of the crankcase. Indeed, when said additives are in contact with sulfuric acid and/or are subject to applied temperature and pressure stresses within the engine, they can partially or totally decompose and form deposits that clog said parts. The result of this is that the deposits formed induce an increase of wear and rapid clogging of engine parts and consequently a more rapid decrease of the service life of engines.

WO 2013/045648 describes lubricant compositions for marine engines allowing fuel oil consumption to be decreased while improving the cleanliness of an engine. Said compositions comprise at least one base oil, at least one detergent, at least one copolymer olefin and at least one glycol ester. EP1344769 discloses polyol esters functionalized by polyamines and their use as dispersant additives which have greater cleanliness while improving the control of deposits.

However, there is an interest in providing lubricant compositions even more efficient in terms of cleanliness of an engine.

The invention is defined in the attached claims.

Thereby, a first subject of the present invention relates to the use, in a lubricant composition for improving the cleanliness of an engine, preferably the cleanliness of the crankcase, said composition comprising at least one base oil of 2 to 12 % by weight with respect to the weight of the lubricant composition, an ester having a viscosity at 100°C between 200 and 1,000 cSt, wherein the alcohol forming the esters is trimethylolpropane and the acid forming the esters is chosen from the fatty acids comprising 6 to 24 carbon atoms.

In the framework of the present invention, the improvement of the cleanliness of an engine is defined by the increase of thermal stability of the lubricant which thus implies a decrease of varnish on engine parts. The thermal stability of the lubricant is determined by the ECBT test such as described below. It should be understood that the improvement of the cleanliness of an engine is understood with respect to what is observed in the absence in the lubricant composition of the invention, of esters according to the invention. Surprisingly, the inventors have shown that with less than 1% by weight of ester or a plurality of than 14% by weight of ester, the cleanliness of an engine was not improved.

Preferably, the ester is in a proportion of 2 to 11%, preferably 3 to 11 %, more preferably 3 to 10%, even more preferably 4 to 10% by weight with respect to the weight of the lubricant composition.

Preferably, the viscosity at 100°C of the ester measured according to the standard ASTM D445, is between 200 and 900 cSt, preferably between 200 and 800, more preferably between 250 and 700 cSt.

The esters of the present invention can be all types of esters obtained by reaction between an alcohol and an acid. The alcohol is trimethylolpropane and the acid forming the esters is chosen from fatty acids comprising from 6 to 24 carbon atoms.

The esters can be chosen in particular from amongst mono-, di- or triesters.

The acids are chosen from amongst acid anhydrides or fatty acids.

Furthermore, advantageously the fatty acids comprise from 6 to 24 carbon atoms. Said fatty acids can be saturated, mono and/or polyunsaturated.

According to a particular embodiment of the invention, the fatty acids used for the reaction with alcohols, are e.g. fatty acids coming from vegetable oils and can be saturated, mono and/or polyunsaturated. Said fatty acids are e.g. chosen from amongst caprylic, pelargonic, capric, undecylenic, lauric, tridecylenics, myristics, pentadecylic, palmitic, margaric, stearic, nonadecylic, arachic, heneicosanoic, behenic, tricosanoic, lignoceric fatty acids, or unsaturated fatty acids such as palmitoleic, oleic, erucic, nervonic, linoleic, a-linolenic, c-linolenic, di-homo-c-linolenic, arachidonic, eicosapentaenoic, docosahexanoic acid. Preferably, the fatty acids come from the hydrolysis of triglycerides present in vegetable and animal oils such as copra, palm, olive, peanut, rapeseed, sunflower, soybean, castor, wood, corn, squash, grapenut, jojoba, sesame, walnut, walnut, almond, shea, macadamia, alfalfa, rye, safflower, coconut, cottonseed, linseed, tallow, or any mixtures thereof. Natural oils can have been genetically modified in order to enrich the concentration thereof of certain fatty acids, e.g. rapeseed oil or oleic sunflower oil.

The esters according to the invention can be mixed esters, i.e. esters obtained by mixtures of different alcohols and/or different acids.

The ester according to the invention is not a glycerol ester.

Preferably, the lubricant composition further comprises at least one detergent. With regard to detergents used in the lubricant compositions according to the present invention, same are well known to a person skilled in the art.

According to a particular embodiment of the invention, the detergents commonly used in the formulation of lubricant compositions are typically anionic compounds comprising a long lipophilic hydrocarbon chain and a hydrophilic head. The associated cation is typically a metal cation of an alkali or alkaline earth metal.

The detergents are preferentially chosen from alkali metal or alkaline earth metal salts of carboxylic acids, sulfonates, salicylates and naphthenates, as well as the phenate salts.

The alkali metals and alkaline earth metals are preferentially calcium, magnesium, sodium or barium.

Said metal salts can contain the metal in an approximately stoichiometric quantity. In such case, one refers to non-overbased or “neutral” detergents, although same also bring a certain basicity. Said “neutral” detergents typically have a Base Number (BN), measured according to ASTM D2896, of less than 150 mg KOH/g, or less than 100, or else less than 80 mg KOH/g.

Said so-called neutral detergents could contribute in part to the BN of the lubricants according to the present invention. Neutral detergents such as carboxylates, sulfonates, salicylates, phenates, naphthenates of alkali metals and alkaline earth metals, e.g. calcium, sodium, magnesium or barium type, will be used.

When the metal is in excess (in a quantity greater than the stoichiometric quantity), one is dealing with so-called overbased detergents. The BN thereof is high, greater than 150 mg KOH/g, typically comprised between 200 and 700 mg KOH/g, generally between 250 and 450 mg KOH/g.

The excess metal that provides the overbased character to the detergent is in the form of metal salts insoluble in oil, e.g. carbonate, hydroxide, oxalate, acetate, glutamate, preferentially carbonate.

In the same overbased detergent, the metals of said insoluble salts can be either the same as the metals of oil-soluble detergents or different. The metals are preferentially chosen from calcium, magnesium, sodium or barium.

Thereby, the overbased detergents are in the form of micelles composed of insoluble metal salts held in suspension in the lubricant composition by the detergents in the form of oil-soluble metal salts.

Said micelles can contain one or a plurality of types of insoluble metal salts, stabilized by one or a plurality of types of detergent.

Overbased detergents comprising a single type of soluble metal salt detergent will generally be named after the nature of the hydrophobic chain of said latter detergent.

Thereby, the detergents will be referred to as carboxylate, phenate, salicylate, sulfonate or naphthenate type depending on whether said detergent is respectively a carboxylate, a phenate, a salicylate, a sulfonate or a naphthenate.

Overbased detergents will be referred to as mixed type if the micelles comprise a plurality of types of detergents, different from each other by the nature of the hydrophobic chain thereof.

For use in the lubricant compositions according to the present invention, the oil-soluble metal salts will preferentially be carboxylates, phenates, sulfonates, salicylates, and mixed phenate-sulfonate and/or salicylates detergents of calcium, magnesium, sodium or barium.

The insoluble metal salts bringing the overbased character are alkali metal and alkaline earth metal carbonates, preferentially calcium carbonate.

The overbased detergents used in the lubricant compositions according to the present invention will preferentially be carboxylates, phenates, sulfonates, salicylates and mixed phenate-sulfonate-salicylate detergents overbased with calcium carbonate.

According to the invention, the lubricant composition may further comprise detergents that may in particular be chosen from amongst:

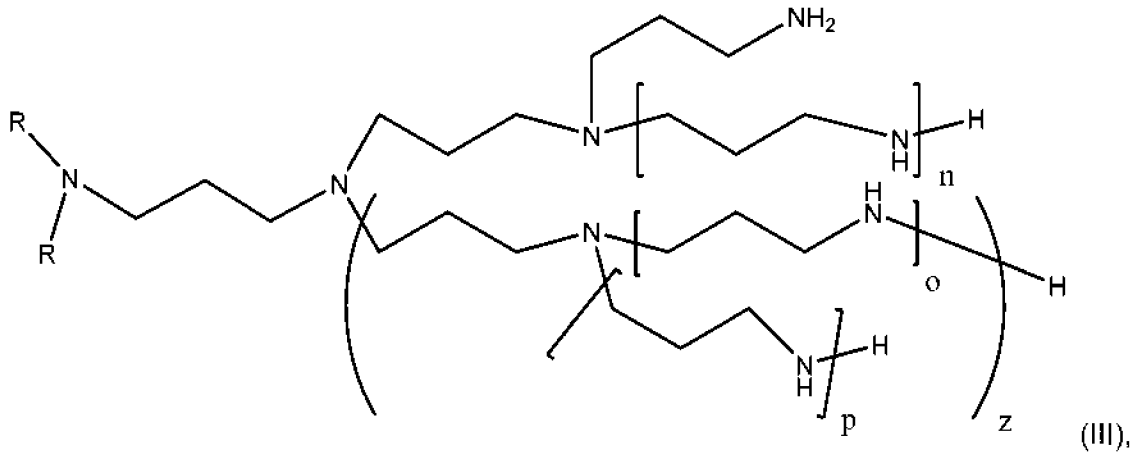
- soluble fatty amines chosen from amongst
  - compounds with the formula (I):

$$R_1\text{-}[(NR_2)\text{-}R_3]_m\text{-}NR_4R_5,$$

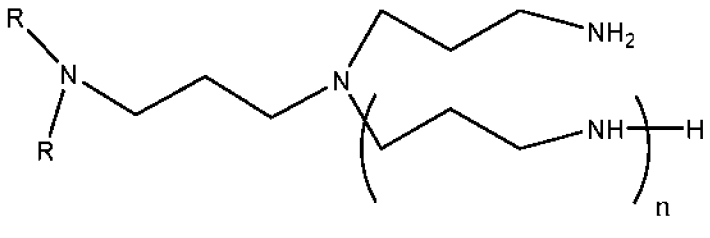
wherein,

- R_1 represents a saturated or unsaturated, linear or branched hydrocarbon moiety comprising at least 12 carbon atoms and optionally at least one heteroatom chosen from amongst nitrogen, sulfur or oxygen,
- R_2, R_4 or R_5 independently represents a hydrogen atom or a saturated or unsaturated, linear or branched hydrocarbon moiety and optionally comprising at least one heteroatom chosen from amongst nitrogen, sulfur or oxygen,

- R_3 represents a saturated or unsaturated, linear or branched hydrocarbon moiety comprising one or a plurality of carbon atoms and optionally comprising at least one heteroatom chosen from amongst nitrogen, sulfur or oxygen, preferably oxygen,
- m is a whole number greater than or equal to 1, preferably between 1 and 10, more preferably between 1 and 6, even more preferably being chosen from amongst 1, 2 or 3 or
  - a mixture of fatty polyalkylamines comprising one or a plurality of polyalkylamines with the formulae (III) and/or (IV):



(III),



(IV)

wherein

- R, identical or different represents a linear or branched alkyl group comprising from 8 to 22 carbon atoms,
- n and z independently from each other, represent 0, 1, 2 or 3 and
- when z is greater than 0, o and p, independently of each other, represent 0, 1, 2 or 3,

said mixture comprising at least 3% by weight of branched compounds such that at least one of n or z is greater than or equal to 1, or the derivatives thereof, or

- mixtures of fatty amines with formulae (I), (III) and/or (IV),

- a detergent containing alkali metals or alkaline earth metals overbased by metal carbonate salts

Fatty amines of formula (I) comprised in the lubricant composition are described in particular in the applications WO 2009/153453 and WO 2014/180843 filed by the applicant.

According to a preferred embodiment, the fatty amine of formula (I) or the mixture of fatty amines of formulae (III) and/or (IV) or the mixture of fatty amines of formulae (I), (III) and/or (IV) is added in a quantity from 0.1 to 15%, preferably from 0.5 to 10%, preferably from 0.5 to 8% or from 3 to 10% by weight with respect to the total weight of the lubricant composition.

By “fatty amine” according to the invention is understood to be an amine of formula (I), (III) or (IV) comprising one or a plurality of saturated or unsaturated, linear or branched hydrocarbon moieties and optionally comprising at least one heteroatom chosen from amongst nitrogen, sulfur or oxygen, preferably oxygen. By “a plurality of fatty amines” according to the invention, a mixture of fatty amines including at least one fatty amine of formula (I), (III) and/or (IV) is understood.

The fatty amines of formula (I), (III) and/or (IV) are in particular such as described in WO2017021426.

In a particularly advantageous manner, the present invention is intended to improve the cleanliness of a marine engine, in particular a 2-stroke or 4-stroke marine engine. More particularly, the invention is intended to improve the cleanliness of a 2-stroke marine engine, in particular the cleanliness of the crankcase. For 2-stroke engines, the lubricant composition is used in particular as cylinder oil or system oil, preferably as cylinder oil.

According to a particular embodiment of the invention, the base oil included in the lubricant composition is chosen from mineral, synthetic or vegetable oils, as well as mixtures thereof.

The mineral or synthetic oils generally used in the application belong to one of the classes defined in the API classification as summarized in the table below.

	Concentration of saturates	Concentration of sulfur	Viscosity index
Group 1 Mineral oils	< 90%	> 0.03%	80 ≤ VI < 120
Group 2 Hydrocracked oils	≥ 90%	≤ 0.03%	80 ≤ VI < 120
Group 3 Hydro-isomerized oils	≥ 90%	≤ 0.03%	≥ 120
Group 4	PAO		
Group 5	Other bases not included in groups 1 to 4 bases		

Group 1 mineral oils can be obtained by distillation of chosen naphthenic or paraffinic crude oils followed by purification of said distillates by methods such as solvent extraction, solvent or catalytic dewaxing, hydrotreatment or hydrogenation.

Group 2 and 3 oils are obtained by more demanding purification methods, e.g. a combination of hydrotreatment, hydrocracking, hydrogenation and catalytic dewaxing.

Examples of Group 4 and 5 synthetic base oils include poly-alpha olefins such as polybutenes, polyisobutenes, alkylbenzenes.

Said base oils can be used alone or as a mixture. A mineral oil can be combined with a synthetic oil.

Cylinder oils for marine diesel 2-stroke engines typically have a viscosity grade SAE-40 to SAE-60, generally SAE-50 equivalent to a kinematic viscosity at 100°C comprised between 16.3 and 21.9 mm^2/s.

Grade 40 oils have a kinematic viscosity at 100°C comprised between 12.5 and 16.3 mm^2/s.

Grade 50 oils have a kinematic viscosity at 100°C comprised between 16.3 and 21.9 mm^2/s.

Grade 60 oils have a kinematic viscosity at 100°C comprised between 21.9 and 26.1 mm^2/s.

Said viscosity can be obtained by mixing additives and base oils e.g. containing Group 1 mineral bases such as Neutral Solvent bases (e.g. 500NS or 600 NS) and Brightstock. Any other combination of mineral, synthetic or vegetable bases having, in admixture with the additives, a viscosity compatible with SAE-50 grade can be used.

Typically, a conventional formulation of cylinder lubricant for slow 2-stroke marine diesel engines is of grade SAE 40 to SAE60, preferentially SAE50 (according to the SAE J300 classification) and comprises at least 50% by weight of a lubricant base oil of mineral and/or synthetic origin, suitable for use in marine engines, e.g. API Group 1 i.e. obtained by distillation of chosen crude oils followed by purification of said distillates by methods such as solvent extraction, solvent or catalytic dewaxing, hydrotreatment or hydrogenation. The Viscosity Index (VI) thereof is comprised between 80 and 120; the concentration of sulfur thereof is greater than 0.03% and the concentration of saturates thereof is less than 90%.

According to a particular embodiment of the invention, the lubricant composition may further comprise one or a plurality of thickening additives the role of which is to increase the viscosity of the composition, both hot and cold, or of additives improving the Viscosity Index (VI).

Preferably, said additives are most often polymers with low molecular weight, on the order of 2,000 to 50,000 dalton (Mn). The additives can be chosen from PIBs (on the order of 2,000 dalton), Polyacrylate or Polymethacrylates (on the order of 30,000 dalton), Olefin copolymers, Copolymers of olefin and Alpha Olefins, EPDM, Polybutenes, Poly-alphaolefins with high molecular weight (viscosity 100°C >150), Styrene-Olefin copolymers, whether or not hydrogenated. According to a particular embodiment of the invention, the base oil(s) included in the lubricant composition according to the invention can be partially or totally substituted by said additives. Henceforth, the polymers used to partially or totally substitute one or a plurality of the base oils are preferentially the abovementioned thickeners such as PIB type (e.g. marketed under the name of Indopol H2100).

According to a particular embodiment of the invention, the lubricant composition can further comprise at least one anti-wear additive. Preferably, the anti-wear additive is Zinc di-thiophosphate or DTPZn. Various phosphorus, sulfur, nitrogen, chlorine and boron compounds are also found in said category. There is a wide variety of anti-wear additives, but the most widely used category is that of phosphor-sulfur additives such as metal alkylthiophosphates, in particular Zinc alkylthiophosphates, and more specifically Zinc dialkyldithiophosphates or DTPZn. Amine phosphates, polysulfides, in particular sulfur olefins, are also commonly used anti-wear additives.

Anti-wear and extreme pressure additives such as nitrogen and sulfur additives are also usually found in lubricant compositions, such as e.g. metal dithiocarbamates, in particular molybdenum dithiocarbamate. Glycerol esters are also anti-wear additives. Examples include e.g. of mono-, di- and trioleates, monopalmitates and monomyristates.

According to a particular embodiment of the invention, the lubricant composition can further comprise at least one dispersant. Dispersants are well known additives used in the formulation of lubricant compositions, in particular for application in the marine field. The primary role thereof is to maintain in suspension the particles initially present or appearing in the lubricant composition during the use thereof in the engine. Dispersants prevent the agglomeration of particles by playing on steric congestion. Dispersants can also have a synergistic effect on neutralization. The dispersants used as lubricant additives typically contain a polar group, associated with a relatively long hydrocarbon chain, generally containing from 50 to 400 carbon atoms. The polar group typically contains at least one nitrogen, oxygen or phosphorus element. Derivative compounds of succinic acid are dispersants particularly used as lubricant additives. Succinimides, obtained by condensation of succinic anhydrides and amines, succinic esters obtained

by condensation of succinic anhydrides and alcohols or polyols, are more particularly used. Said compounds can then be treated with various compounds, in particular sulfur, oxygen, formaldehyde, carboxylic acids and compounds containing boron or zinc for producing e.g. borated succinimides or zinc-blocked succinimides. Mannich bases, obtained by polycondensation of alkyl-substituted phenols, formaldehyde and primary or secondary amines, are also compounds used as dispersants in lubricants. It is possible to use a dispersant of the family of PIB succinimides e.g. borated or blocked with zinc.

According to a particular embodiment of the invention, the lubricant composition can further comprise any type of functional additives suitable for the use thereof, e.g. anti-foam additives, to counter the effect of detergents, which can be e.g. polar polymers such as polymethylsiloxanes, polyacrylates, antioxidant and/or rust-inhibiting additives, e.g. organo-metallic detergents or thiadiazoles. Same are known to a person skilled in the art.

According to the present invention, the compositions of the lubricants described refer to the compounds taken separately before mixing, it being understood that said compounds may or may not retain the same chemical form before and after mixing. Preferably, the lubricants according to the present invention obtained by mixing the compounds taken separately are not in emulsion nor in microemulsion form.

Preferably, the base oil of the composition according to the invention is a base oil from group 2.

The subject of the present invention is also the use of a lubricant composition comprising at least one base oil and from 2 to 12% by weight with respect to the weight of the lubricant composition, of an ester compound having a viscosity at 100°C measured according to the standard ASTM D445 comprised between 100 and 1000 cSt to improve the cleanliness of an engine. The quantities of the ester, the esters, the base oil and the lubricant composition and the possible additives thereof are such as defined above.

The present invention also relates to a lubricant composition comprising at least one base oil and from 2 to 12% by weight with respect to the weight of the lubricant composition, of an ester having a viscosity at 100°C measured according to the standard ASTM D445 comprised between 100 and 1000 cSt to improve the cleanliness of an engine. The quantities of the ester, the esters, the base oil and the lubricant composition and the possible additives thereof are such as defined above.

The present application will now be described using non-limiting examples.

Example 1: Compositions according to the invention and comparative compositions

The esters of table 1 have been used in the following lubricant compositions tested.

Ester	Viscosity at 100°C (cSt)
Ester 1 (according to the invention)	600
Ester 2 (according to the invention)	315
Ester 3 (according to the invention)	589
Ester 4 (comparative)	98
Ester 5 (comparative)	36
Ester 6 (comparative)	12

The following compositions (CI: composition according to the invention and CC: comparative composition) were prepared:

	CI1	CI2	CI3	CI4	CI5	CI6	CC1	CC2	CC3	CC4	CC5	CC6
Base oil (% by weight)	79.7	79.7	79.7	84.2	84.2	84.2	88.6	79.7	79.7	79.7	87.71	75.3
Ester 1 (% by weight)	8.9			4.4							0.89	13.3
Ester 2 (% by weight)		8.9			4.4							
Ester 3 (% by weight)			8.9			4.4						
Ester 4 (% by weight)								8.9				
Ester 5 (% by weight)									8.9			
Ester 6 (% by weight)										8.9		
Detergent package	11.4	11.4	11.4	11.4	11.4	11.4	11.4	11.4	11.4	11.4	11.4	11.4

Table 2

The properties of said compositions are described in table 3 below

Composition	CI1	CI2	CI3	CI4	CI5	CI6	CC1	CC2	CC3	CC4	CC5	CC6
Viscosity at 40°C (mm2/s)	117.5	189.2	170.2	198	181	200.5	217.6	196.8	189	201.6	216.0	151.9
Viscosity at 100°C (mm2/s)	19.7	19.74	18.1	19.36	17.72	19.3	19.02	19	18.08	18.8	19.6	19.6
VI	128	131	118	111	107	109	98	109	105	104	103	148
BN (KOH/g)	25.1	24.9	25.1	25.2	25	25.3	25.3	25.1	25	25.3	25.2	25

Table 3

The viscosity at 40°C was measured according to the standard ASTM D7279.

The viscosity at 100°C was measured according to the standard ASTM D7279.

VI corresponds to the index of viscosity and was calculated according to the standard NF ISO 2909.

BN corresponds to the Base Number measured according to the standard ASTM D2896.

Example 2: Evaluation of properties of the lubricant compositions according to the invention

ECBT tests were performed to evaluate the properties of the lubricant compositions according to the invention.

Said tests allow the appearance of a varnish on engine parts to be simulated.

The thermal resistance of the compositions was thereby measured by means of the ECBT test. A detailed description of said test is given in the publication entitled “Research and Development of Marine Lubricants in ELF ANTAR France – The relevance of laboratory tests in simulating field performance” by Jean-Philippe ROMAN, MARINE PROPULSION CONFERENCE 2000 – AMSTERDAM – 29-30 MARCH 2000.

The results are listed in table 4 below.

The results show that the compositions according to the invention, have a good thermal resistance and thus allow the cleanliness of an engine to be improved.

	COMPOSITIONS					
	CI1	CI2	CI3	CI4	CI5	CI6
Measurement of the rating at 280°C	73.6	92.3	88.5	62.9	63.9	45.7

Critical temperature measured at the rating 50	289°C	289°C	295°C	285°C	283°C	282°C

	COMPOSITIONS					
	CC1	CC2	CC3	CC4	CC5	CC6
Measurement of the rating at 280°C	32.2	40.1	34.1	28.8	12.2	47
Critical temperature measured at the rating 50	277°C	278°C	277°C	276°C	274°C	279°C

Table 4

With regard to the measurement of the rating at 280°C, if the surface is without varnish, then the rating is 100. In other words, the lower the rating, the more varnish on the surface.

The critical temperature corresponds to the temperature at which the surface has a rating of 50.

Said results show that the esters having a viscosity according to the invention advantageously allow the cleanliness of an engine to be improved with respect to lubricant compositions without said esters and with respect to lubricant composition comprising esters of different viscosities. Indeed, the varnish rating is much closer to 100 for the esters according to the invention. Moreover, the critical temperature is much higher for esters according to the invention.

Said results also highlight the influence of the quantity of ester used.

ANVENDELSE AF ESTERE I EN SMØREMIDDELSAMMENSÆTNING TIL FORBEDRING AF RENHEDEN AF EN MOTOR

PATENTKRAV

1.- Anvendelse, i en smøremiddelsammensætning omfattende mindst én baseolie, af en ester med 2 til 12 vægtprocent i forhold til smøremiddelsammensætningens vægt med en viskositet ved 100 °C på mellem 200 og 1000 cSt for at forbedre motorens renhed, hvor den alkohol, der danner esterne, er trimethylolpropan, og den syre, der danner esterne, udvælges blandt fedtsyrer omfattende fra 6 til 24 carbonatomer.

2.- Anvendelse ifølge krav 1, hvor esterens viskositet ved 100 °C er mellem 200 og 900 cSt, fortrinsvis mellem 200 og 800, mere fortrinsvis mellem 250 og 700 cSt.

3.- Anvendelse ifølge krav 1 eller 2, hvor esteren findes i en andel på 2 til 11 vægtprocent, fortrinsvis fra 3 til 11 vægtprocent, mere fortrinsvis fra 3 til 10 vægtprocent, endnu mere fortrinsvis fra 4 til 10 vægtprocent i forhold til vægten af smøremiddelsammensætningen.

4.- Anvendelse i henhold til et hvilket som helst af kravene 1 til 3, hvor motoren er en marinemotor, fortrinsvis en 2-takts marinemotor.

5.- Fremgangsmåde til forbedring af renheden af en motor omfattende anvendelse i nævnte motor af et smøremiddelsammensætning, der omfatter mindst én baseolie og en ester med 2 til 12 vægtprocent i forhold til smøremiddelsammensætningens vægt med en viskositet ved 100 °C på mellem 200 og 1000 cSt, idet esteren er som defineret i krav 1.