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(54) Title: LUBRICATING COMPOSITION

(57) Abstract: The present invention provides a lubricating composition comprising: - a base oil; and - a viscosity index improver containing poly (meth) acrylate having hydroxyl groups in the structure and having a hydroxyl value of from 22 to 37.

LUBRICATING COMPOSITION

The present invention relates to a lubricating composition of excellent water compatibility, in particular, a lubricating composition that is suitable for use as driving system lubricating composition of vehicles such as automobiles, agricultural vehicles and construction machinery.

As a lubricating composition that is employed for vehicles such as automobiles or for industrial use, a lubricating composition is sought that is of high viscosity index, whose viscosity does not change over a temperature region that is as wide as possible. As such lubricating compositions, lubricating compositions are employed in which viscosity index improvers, such as poly(meth)acrylate, are blended with a base oil such as mineral oil or synthetic oil: in order to obtain a lubricating composition with a high viscosity index, a poly(meth)acrylate having various functional groups is employed, and a poly(meth)acrylate having hydroxyl groups is also employed.

A lubricating composition is used in rolling contact systems or sliding contact systems in order to prevent interference between sliding elements, by forming an oil film between the elements constituting the contact system. In such lubrication systems, there is a possibility of infiltration of water into the apparatus during use; if the oil film is broken by such infiltration of water, lubricating performance is degraded. In order to prevent such degradation of lubricating performance, measures are typically taken such as the provision in the apparatus of means for removing separated water, such as a drain cock,

and removal of the separated water may be facilitated by employing a demulsifying lubricating composition.

Laid-open Japanese Patent Application No. 2005-290182 proposes a gear lubricating composition of improved water separating performance, using viscosity index improvers containing poly(meth)acrylate as a demulsifier. However, this reference merely discloses, as demulsifiers, poly(meth)acrylate not having hydroxyl groups: obtaining a lubricating composition that has been given compatibility with water by use of poly(meth)acrylate having hydroxyl groups is not disclosed.

Also, Laid-open Japanese Patent Application No. 2007-197509 proposes the use of poly(meth)acrylate having a polar group as a sludge separating agent for a lubricating composition having high demulsifying performance: a hydroxyl group is also disclosed as an example of a polar group. However, the polymer employed herein is only a single type of polymer, constituted of different monomers, and no Practical Example of a polymer having hydroxyl groups as a polar group is disclosed. It is only disclosed that this polymer has excellent sludge dispersibility and high water separating performance: obtaining a lubricating composition of emulsifying properties that has been given compatibility with water by poly(meth)acrylate having hydroxyl groups is not disclosed.

As described above, many lubricating systems are constructed to permit discharge of separated water, so the lubricating composition that is used therein is required to have water separating properties. In contrast, the driving system of an automobile has a construction that is difficult for water to penetrate from outside, so

means for removing separated water, such as a drain cock, is not provided. Even in such lubricating systems, infiltration of water from outside may occur due to incidents such as immersion; however, so long as the quantity is small, it is arranged that, by continued use, the infiltrated water can be removed by spontaneous evaporation. However, when a large quantity of water infiltrates into the lubricating composition, there is the problem that the water is not emulsified and phase separation occurs; in this condition, the oil film is broken and lubricating performance is degraded: the sliding parts are flooded with water and seizure easily takes place.

Also in the case of multigrade lubricating compositions, polymers may be added to the base oil as viscosity index improvers. The properties that such polymers are required to have include: 1) a high viscosity-increasing effect (viscosity index improving effect); 2) excellent low-temperature viscosity properties; and 3) excellent shear stability. In addition, a pour point depressant function is also sometimes demanded. The above items 1) to 3) are in a relationship having a close mutual connection: if the viscosity increasing effect of 1) is increased, the shear stability of 3) in general becomes poor; this is because the molecular weight of the polymer becomes large. Also 2) is related to the type, structure and molecular weight of the polymer.

In recent years, lubricating compositions having the functions 1) to 3) at the same time are being demanded, but it is difficult for a single polymer to satisfy these functions. For example, in order to improve the shear stability, a polymer of low molecular weight may be

employed, but, as a result, the viscosity-increasing effect of 1) becomes poor, making it necessary to employ a large quantity of polymer. The effect of this is not only that the cost of the product is increased, but also that the benefits thereof in terms of improving the viscosity index cannot be fully realised. Specifically, viscosity is increased by the addition of polymer, but, in the relationship between the amount of viscosity increase α at high temperature (100°C) and the amount of increase β of viscosity at low temperature (40°C), if α is larger than β , the viscosity index improving effect is large and the desired viscosity grade can be realised by addition of a small amount of polymer. However, if α is smaller than β , the problem arose that even if the increase in viscosity at low temperature is excessive, the increase in viscosity at high temperature does not achieve the target, so the viscosity index improving effect is small, with the result that either an enormous addition of polymer was necessary, or the desired viscosity grade could not be achieved.

In order to solve the above problems, an object of the present invention is to provide a lubricating composition that is suited for use as a driving system lubricating composition of for example automobiles, agricultural vehicles, or construction machinery, that has excellent compatibility with water, so that, even in the event of infiltration of water, emulsification takes place, preventing breakage of the oil film from being caused by phase separation, making it possible to prevent seizure of sliding parts being thereby produced and providing excellent viscosity properties as multigrade lubricating composition.

The present invention provides the following:

(1) Lubricating composition comprising:

- a base oil; and
- a viscosity index improver containing

poly(meth)acrylate having hydroxyl groups in the
5 structure and having a hydroxyl value of from 22 to 37.

(2) Preferably the viscosity index improver contains
50 to 100 mass parts of poly(meth)acrylate having
hydroxyl groups in the structure, and from 50 to 0 mass
10 parts of poly(meth)acrylate not having hydroxyl groups in
the structure.

(3) Further it is preferred that the
poly(meth)acrylate of the viscosity index improver has a
mass average molecular weight of from 5,000 to 50,000.

(4) Also it is preferred that the viscosity index
15 improver is present in an amount of from 1 to 8 mass%,
based on the total weight of the lubricating composition,
as net polymer, excluding diluent oil.

(5) Preferably the lubricating composition contains
20 another additive selected from extreme pressure agents,
anti-wear additives, pour point depressants, antioxidants,
metal deactivators, oiliness agents, defoaming agents and
rust inhibitors.

(6) Preferably the lubricating composition is for
use in the drive systems of vehicles.

25 The lubricating composition according to the present
invention is has excellent water compatibility and may be
employed in particular in lubrication systems such as
automobile driving systems where no means for removal of
separated water is provided, to prevent breakage of the
30 oil film from occurring due to phase separation: this is
achieved by emulsification of infiltrated water.

As the base oil that is employed in the present
invention, base oils that are commonly used as base oil

for lubricating composition may be employed, there being no restriction regarding origin or method of refining etc. As the base oil that may be employed, base oil such as mineral oil or synthetic oil, called highly refined base oil, can be employed. One type of base oil belonging to group I, group II, group III, group IV or group V etc of the base oil category of the API (American Petroleum Institute) or a mixture of a plurality of types may be employed.

As the mineral oil-based base oil that is employed in the present invention, there may be given as specific examples a base oil obtained by refining the oil fraction obtained by reduced-pressure distillation of normal-pressure residue oil obtained by normal-pressure distillation of petroleum, that has then been subjected to one or more treatments such as solvent deasphalting, solvent extraction, hydrocracking, solvent desulfurizing, or hydrofining; or wax-isomerized mineral oil. A preferred example of base oil employed in the present invention is highly hydrofined base oil.

There is no particular restriction concerning the base oil other than mineral oil-based oil referred to above and the previously employed synthetic oils may be used: depending on the application, these may be suitably selected or chosen and employed in any desired ratio with mineral oil. Examples that may be given include: poly- α -olefins, α -olefin copolymers, polybutene, polyol esters, DBE, polyhydric alcohol esters, polyoxyalkylene glycols, polyoxyalkylene glycol esters, polyoxyalkylene glycol ethers, and cycloalkane-based compounds. Lubricating composition base oils etc manufactured by the technique of isomerizing FT wax manufactured by the Fischer-Tropsch

process from natural gas may also be mentioned by way of example.

There is no particular restriction regarding the viscosity index of the base of oil, but, in order to obtain excellent viscosity performance from low to high temperature, its value is preferably at least 90, even more preferably at least 100, and most preferably at least 110. If the viscosity index of the lubricating base oil is less than 90, the added amount of poly(meth)acrylates constituting the added viscosity index improver is preferably increased or the shear stability is degraded, due to the use of poly(meth)acrylates of large molecular weight. There is no particular restriction regarding the upper limit of the viscosity index, and base oil of viscosity index about 120 to 180, such as normal paraffins, slack wax, or GTL (gas to liquid) wax etc, or isoparaffin-based mineral oil obtained by isomerization of these, or complex ester-based base oil or PAO (poly- α -olefin)-based base oil of viscosity index about 150 to 250 may be employed.

The kinematic viscosity of the base oil at 100°C is preferably 2 to 6 mm²/s, more preferably 2 to 5 mm²/s. If the kinematic viscosity at 100°C is less than 2 mm²/s, oil film formation at the lubricated locations is insufficient, so the lubrication performance is degraded, with the risk that seizure or wear of the gear surfaces may occur, or evaporation loss of the base oil may become large.

The kinematic viscosity of the base oil at 40° C is preferably 5 to 35 mm²/s, more preferably 6 to 27 mm²/s. If the kinematic viscosity at 40°C is less than 5 mm²/s, oil film formation at the lubricated locations is insufficient, so the lubrication performance is degraded,

with the risk that seizure or wear of the gear surfaces may occur, or evaporation loss of the base oil may become large.

The viscosity index improvers that are blended with the above base oil include poly(meth)acrylates having hydroxyl groups in their structure and having a hydroxyl value of from 22 to 37. As poly(meth)acrylates containing hydroxyl groups as referred to above, there may be mentioned by way of example alkyl(meth)acrylates which are copolymers constructed of structural units having hydroxyl groups and that have alkyl groups of carbon number 1 to 20; and copolymers having as required constituent monomers hydroxyl group-containing vinyl monomers. In the present invention, "(meth)acryl" means "acryl" or "methacryl".

As the alkyl(meth)acrylates (a) having alkyl groups of carbon number 1 to 20 referred to above, specifically, there may be mentioned by way of example:

(a1) alkyl(meth)acrylates having alkyl groups of carbon number 1 to 4: for example, methyl(meth)acrylate, ethyl(meth)acrylate, n-, or isopropyl(meth)acrylate, n-, iso-, or sec-butyl(meth)acrylate;

(a2) alkyl(meth)acrylates having alkyl groups of carbon number of from 8 to 20: for example, n-octyl(meth)acrylate, 2-ethylhexyl(meth)acrylate, n-decyl(meth)acrylate, n-isodecyl(meth)acrylate, n-undecyl(meth)acrylate, n-dodecyl(meth)acrylate, n-methylundecyl(meth)acrylate, n-tridecyl(meth)acrylate, n-methyldodecyl(meth)acrylate, 2-methyldodecyl(meth)acrylate, n-tetradecyl(meth)acrylate, 2-methyltridecyl(meth)acrylate, n-pentadecyl(meth)acrylate, n-methyltetradecyl(meth)acrylate, n-hexadecyl(meth)acrylate,

and n-octadecyl(meth)acrylate, n-eicosyl(meth)acrylate, n-docosyl(meth)acrylate, dobanol 23 [mixture of oxoalcohols having a carbon number of 12/13 manufactured by Mitsubishi Chemicals Ltd] methacrylate, dobanol 45
5 [mixture of oxoalcohols having a carbon number of 13/14 manufactured by Mitsubishi Chemicals Ltd] methacrylate, etc., and

(a3) alkyl(meth)acrylates having alkyl groups having a carbon number of from 5 to 7: for example, n-
10 pentyl(meth)acrylate and n-hexyl(meth)acrylate.

Of the above monomers (a1) to (a3), the preferred monomers belong to (a1) and (a2); of these, the monomer (a2) is preferred. Of the above (a1), the monomers that are preferred from the point of view of viscosity index
15 are those with alkyl groups having a carbon number of 1 to 2. And of the above (a2), the monomers that are preferred from the point of view of solubility with the base oil and low-temperature performance are those with alkyl groups having a carbon number of from 10 to 20, or,
20 even more preferably, a carbon number of from 12 to 14.

The hydroxyl group-containing vinyl monomers (b) that form a copolymer with the alkyl(meth)acrylate having an alkyl group having a carbon number 1 to 20 referred to above are vinyl monomers containing one or more
25 (preferably one or two) hydroxyl groups per molecule. Specific examples that may be given include:

(b1) hydroxyalkyl (having a carbon number of from 2 to 6) (meth)acrylate: for example, 2-
hydroxyethyl(meth)acrylate, 2 or 3-
30 hydroxypropyl(meth)acrylate, 2-hydroxybutyl(meth)acrylate, 1-methyl-2-hydroxyethyl(meth)acrylate etc.,

(b2) mono- or dihydroxyalkyl (having a carbon number of from 1 to 4) substituted (meth)acrylamide: for example,

N, N-dihydroxymethyl(meth)acrylamide, N, N-dihydroxypropyl(meth)acrylamide, N, N-di-2-hydroxybutyl(meth)acrylamide etc.,

(b3) vinyl alcohols (formed by hydrolysis of vinyl acetate units),

(b4) alkenols having a carbon number of from 3 to 12: for example (meth)aryl alcohols, crotyl alcohol, isocrotyl alcohol, 1-octenol, 1-undecenol etc.,

(b5) alkene diols having a carbon number of from 4 to 12: for example, 1-butene-3-ol, 2-butene-1-ol, 2-butene-1,4-diol etc.,

(b6) hydroxyalkyl (having a carbon number of from 1 to 6) alkenyl (having a carbon number of from 3 to 10) ethers: for example, 2-hydroxyethylpropenyl ether etc.,

(b7) hydroxyl group-containing aromatic monomers: for example, o-, m- or p-hydroxystyrene etc.,

(b8) polyhydric (3- to 8- hydric) alcohols: for example, alkane polyols, intramolecular or intermolecular anhydrides thereof, sugars (for example, glycerine, pentaerythritol, sorbitol, sorbitane, diglycerine, cane sugar) alkenyl (having a carbon number of from 3 to 10) ethers or (meth)acrylates (for example, cane sugar (meth)aryl ether) etc.

(b9) mono(meth)acrylates or mono(meth)aryl ethers of polyoxyalkylene chains and hydroxyl group-containing vinyl monomers: for example, polyoxyalkylene glycol (carbon number of alkylene group of from 2 to 4, degree of polymerization 2 to 50) or polyoxyalkylene polyols {polyoxyalkylene ethers (alkyl group carbon number of from 2 to 4, degree of polymerization of from 2 to 100) of 3- to 8-hydric alcohols}{for example, polyethylene glycol (degree of polymerization of from 2 to 9) mono(meth)acrylate, or polypropylene glycol (degree of

polymerization of from 2 to 12) mono(meth)acrylate, or polyethylene glycol (degree of polymerization of from 2 to 30) mono(meth)aryl ether.

Of the above monomers (b1) to (b9), monomers (b1),
5 in particular 2-hydroxyethylmethacrylate, are preferable from the point of view of viscosity index improving effect.

From the point of view of the viscosity index, it is desirable that the ratio of the monomers (a) and monomers
10 (b) constituting the hydroxyl group-containing poly(meth)acrylate copolymer referred to above should be as follows.

The above monomers (a) are preferably present in an amount of from 50 to 95 mass%, and even more preferably
15 75 to 85 mass%. When the monomers (a1) are employed as the above monomers (a), the monomers (a1) are preferably present in an amount of from 0 to 20 mass% and even more preferably 1 to 10 mass%. When the monomers (a2) are employed as the above monomers (a), the monomers (a2) are
20 preferably present in an amount of from 50 to 95 mass% and even more preferably 70 to 90 mass%.

The above monomers (b) are preferably present in an amount of from 5 to 50 mass%, and even more preferably 7 to 30 mass%; 11 to 15 mass% is particularly preferred.

25 The ratio of the totals of the above monomers (a) and monomers (b) is preferably 55 to 100 mass% and even more preferably 82 to 100 mass%; the balance may be the following other monomers.

Other monomers can be copolymerized together with
30 the above monomers (a) and monomers (b) in the hydroxyl group-containing poly(meth)acrylate copolymer; monomers (c) containing nitrogen atoms may be employed as such

other monomers. Specific examples that may be given include:

(c1) nitro group-containing monomers: for example, 4-nitrostyrene;

5 (c2) primary to tertiary amino group-containing vinyl monomers: for example

(c2-1) primary amino group-containing vinyl monomers: for example, alkenylamines having a carbon number of from 3 to 6 [(meth)arylamines, or crotylamines etc], aminoalkyl (having a carbon number of from 2 to 6) (meth)acrylates [aminoethyl(meth)acrylate etc],

(c2-2) secondary amino group-containing vinyl monomers: for example, alkyl (having a carbon number of from 1 to 6) aminoalkyl (having a carbon number of from 2 to 6) (meth)acrylates [t-butylaminoethyl methacrylate, methylaminoethyl(meth)acrylate etc], diphenylamine(meth)acrylamides [4-diphenylamine(meth)acrylamide, 2-diphenylamine(meth)acrylamide etc], and dialkenylamines having a carbon number of from 6 to 12 [di(meth)arylamines etc.]

(c2-3) tertiary amino group-containing vinyl monomers: for example, dialkyl (having a carbon number of from 1 to 4) aminoalkyl (having a carbon number of from 2 to 6) (meth)acrylates [dimethylaminoethyl (meth)acrylate, diethylaminoethyl(meth)acrylate etc.], dialkyl (having a carbon number of from 1 to 4) aminoalkyl (having a carbon number of from 2 to 6) (meth)acrylamides [dimethylaminoethyl (meth) acrylamide, diethylaminoethyl(meth)acrylamide, dimethylaminopropyl(meth)acrylamide etc] or tertiary amino group-containing aromatic vinyl monomers [N, N-dimethylaminostyrene, etc.], or

(c2-4) nitrogen-containing heterocyclic ring-containing vinyl monomers [morpholinoethyl(meth)acrylate, 4-vinylpyridine, 2-vinylpyridine, N-vinylpyrrole, N-vinylpyrrolidone, N-vinylthiopyrrolidone etc.];

5 (c3) amphoteric vinyl monomers:

for example, N-(meth)acryloyloxy (or amino) alkyl (having a carbon number of from 1 to 10) N, N-dialkyl (having a carbon number of from 1 to 5) ammonium-N-alkyl (having a carbon number of from 1 to 5) carboxylates (or sulfates),
10 for example N-(meth)acryloyloxyethyl N, N-dimethylammonium N-methylcarboxylates, N-(meth)acryloylaminopropyl N,N-dimethylammonium N-methylcarboxylates, and N-(meth)acryloyloxyethyl N, N-dimethylammoniumpropyl sulfate etc.; and

15 (c4) nitrile group-containing monomers: for example, N-(meth)acrylonitrile etc.

Also, other such monomers are aliphatic hydrocarbon vinyl monomers (d). Examples are alkenes having a carbon number of from 2 to 20 [ethylene, propylene, butene,
20 isobutylene, pentene, heptene, diisobutylene, octene, dodecene, octadecene etc.] and alkadienes having a carbon number of from 4 to 12 [butadiene, isoprene, 1,4-pentadiene, 1,6-heptadiene, 1,7-octadiene etc.].

Further such monomers are alicyclic hydrocarbon vinyl monomers (e): examples are cyclohexene,
25 (di)cycloheptadiene, pinene, limonene, indene, vinylcyclohexene, and ethylidene bicycloheptene.

Yet further such monomers are aromatic hydrocarbon vinyl monomers (f): examples are styrene, α -methylstyrene,
30 vinyltoluene, 2,4-dimethylstyrene, 4-ethylstyrene, 4-isopropylstyrene, 4-butylstyrene, 4-phenylstyrene, 4-cyclohexylstyrene, 4-benzylstyrene, 4-crotylbenzene, and 2-vinylnaphthalene.

Yet further such monomers are vinyl esters, vinyl ethers or vinyl ketones (g): examples are vinyl esters of saturated fatty acids having a carbon number of from 2 to 12 [vinyl acetate, vinyl propionate, vinyl butyrate, vinyl octanoate etc.], alkyl, allyl or alkoxyalkyl having a carbon number of from 1 to 12 vinyl ethers [methylvinyl ether, ethylvinyl ether, propylvinyl ether, butylvinyl ether, 2-ethylhexylvinyl ether, phenylvinyl ether, vinyl 2-methoxyethyl ether, vinyl 2-butoxyethyl ether etc.] and alkyl or allyl having a carbon number of from 1 to 8, vinyl ketones [methylvinyl ketone, ethylvinyl ketone, phenylvinyl ketone etc.]

Yet further such monomers are esters of unsaturated carboxylic acids (h); examples are alkyl, cycloalkyl or aralkyl esters of unsaturated carboxylic acids; of these, the alkyl diesters [dimethyl maleate, dimethyl fumarate, diethyl maleate, or dioctyl maleate etc.] having a carbon number of from 1 to 8 of unsaturated carboxylic acids [maleic acid, fumaric acid, and itaconic acid etc.] may be mentioned.

Also, polyoxyalkylene chain-containing vinyl monomers (not containing hydroxyl groups) (i) may be employed: for example, the mono(meth)acrylates [for example, methoxy polyethylene glycol (molecular weight of from 110 to 310) (meth)acrylates, or lauryl alcohol ethylene oxide adduct (2 to 30 mol) (meth)acrylates etc.] of monoalkyl (having a carbon number of from 1 to 18) ethers of polyoxyalkylene glycols (carbon number of alkylene groups of from 2 to 4, degree of polymerization 2 to 50) or polyoxyalkylene polyols [polyoxyalkylene ethers of the above 3 to 8-hydric alcohols (carbon number of alkyl groups of from 2 to 4, degree of polymerization 2 to 100)].

Also, carboxyl group-containing vinyl monomers (j) may be employed as monomers for copolymerization, such as for example monocarboxyl group-containing vinyl monomers such as for example monoalkyl (having a carbon number of from 1 to 8) esters [maleic acid monoalkyl esters, fumaric acid monoalkyl esters, itaconic acid monoalkyl esters, etc] of unsaturated monocarboxylic acids [(meth)acrylic acid, α -methyl(meth)acrylic acid, crotonic acid or cinnamic acid etc.] or unsaturated dicarboxylic acids; or vinyl monomers containing two or more carboxyl groups, such as for example maleic acid, fumaric acid, itaconic acid, or citraconic acid.

Of the above additional monomers (c), (d), (e), (f), (g), (h), (i) and (j), monomers (c) are preferred; two or more of the monomers (c) may be used in combination. Of the above monomers (c), monomers (c2) are preferred and dimethylaminoethyl(meth)acrylate and diethylaminoethyl(meth)acrylate are even more preferred.

The mass average molecular weight of the above poly(meth)acrylates containing hydroxyl groups is preferably from 10,000 to 50,000, more preferably from 7,500 to 35,000, and particularly preferably from 5,000 to 30,000. If the mass average molecular weight is in the above ranges, excellent shear stability can be achieved. It should be noted that this mass average molecular weight (Mw) is that obtained using gel permeation chromatography and is found by calculating as polystyrene. The above mass average molecular weight can be adjusted by means of the temperature on polymerisation, the monomer concentration (solvent concentration), and the amount of catalyst or the amount of chain transfer agent etc.

The dispersion (M_w/M_n) of this hydroxyl group-containing poly(meth)acrylate is preferably from 1 to 2.5, more preferably from 1.2 to 2, and particularly preferably from 1.5 to 1.7. Smaller dispersion implies better shear stability. The numerical average molecular weight (M_n) is found in the same way as the mass average molecular weight (M_w).

The solubility parameter of this hydroxyl group-containing poly(meth)acrylate is preferably 8.6 to 11, more preferably from 9.2 to 10.5, and particularly preferably from 9.3 to 9.7. Solubility in the base oil is even better if the solubility parameter's value is in the above ranges. This solubility parameter value is calculated by Fedor's method (Polym. Eng. Sci. 14(2), 152, (1974)).

In addition the HLB of the hydroxyl group-containing poly(meth)acrylates is preferably from 0.5 to 7, more preferably from 1 to 6.5, and particularly preferably from 1.5 to 6. If the HLB is in these ranges, particularly good emulsification properties are achieved. This HLB value is a value calculated by the HLB according to Oda's method, based on the organic/inorganic concept ("New Introduction to Surfactants", published by Sanyo Chemical Industries Ltd, p128).

The hydroxyl value of the hydroxyl group-containing poly(meth)acrylates employed as viscosity index improvers is from 22 to 37, preferably from 25 to 35 and more preferably from 28 to 33. The hydroxyl value is a numerical value obtained by measurements based on JIS K3342 (1961), and is a value indicating the amount of hydroxyl groups in the additive.

The viscosity index improver according to the present invention contains poly(meth)acrylates containing

hydroxyl groups in the structure referred to above: it may contain only poly(meth)acrylates containing hydroxyl groups in the structure, but preferably contains both poly(meth)acrylates that have hydroxyl groups in the structure and poly(meth)acrylates that do not have hydroxyl groups in their structure. As poly(meth)acrylates that do not have hydroxyl groups in their structure, poly(meth)acrylates that are generally employed as viscosity index improvers for lubricating compositions may be employed: for example, one or two or more polymers of the above monomers (a) may be mentioned by way of example.

In the case where both poly(meth)acrylates that have hydroxyl groups in the structure and poly(meth)acrylates that do not have hydroxyl groups in their structure are present, the poly(meth)acrylates that have hydroxyl groups in their structure are from 50 to 100 mass parts, preferably from 60 to 100 mass parts and even more preferably from 70 to 100 mass parts, while the poly(meth)acrylates that do not have hydroxyl groups in their structure are from 50 to 0 mass parts, preferably from 40 to 0 mass parts and even more preferably from 30 to 0 mass parts.

The lubricating composition is formed by blending the above viscosity improver with the base oil: the added amount of viscosity improver, as net polymer excluding diluent oil, is from 1 to 8 mass%, preferably from 1 to 6 mass%, based on the total weight of the lubricating composition.

The lubricating composition comprising the viscosity index improver and the base oil has a viscosity index of from 90 to 220, preferably from 100 to 200, a kinematic viscosity at 100°C of preferably from 4 to 13 mm²/s, even

more preferably from 6 to 10 mm²/s, a kinematic viscosity at 40°C of preferably from 20 to 61 mm²/s, and even more preferably from 38 to 48 mm²/s; in particular, the SAE viscosity grade is preferably 75W-80 to 75W-85: the
5 viscosity index improver is blended so that a lubricating composition having such a viscosity characteristic is obtained.

The other additives that are typically employed as additives for lubricating composition may be blended with
10 the lubricating oil composition of the present invention: there may be cited by way of example typical extreme pressure agents, anti-wear additives, pour point depressants, antioxidants, metal deactivators, oiliness agents, defoaming agents, rust inhibitors and other
15 publicly known additives.

As extreme pressure agents added to the lubricating composition according to the present invention, there may be employed sulfur-based extreme pressure agents,
phosphorus-based extreme pressure agents,
20 sulfur/phosphorus-based extreme pressure agents, or mixtures of these.

As sulfur-based additives, there may be mentioned by way of example additives containing hydrocarbon sulfide compounds typified by olefin sulfides, and at least one
25 type of sulfur compound selected from the group consisting of sulfurated fats and oils.

Hydrocarbon sulfide compounds include monosulfide compounds, disulfide compounds, trisulfide compounds and polysulfide compounds. Particularly suitable polysulfide
30 compounds are olefin sulfides (diisobutylene polysulfides). As olefin sulfides, the product obtained by sulfuration of olefins such as polyisobutylene using a sulfurating agent may be employed; in the lubricating

composition according to the present invention, suitably, from 1 to 5 weight%, in particular from 1.5 to 3 weight%, of polysulfides may be employed, in terms of elemental sulfur.

5 Also, as sulfurated oils and fats, there may be mentioned by way of example the reaction products of oils and fats with sulfur; products having a sulfur element content of from 5 to 20 weight% may be employed.

10 Such sulfur-based additives may be blended in a range of from 1 to 5 mass% in terms of elementary sulfur content, based on the total weight of the lubricating composition: a particularly preferred blending content is from 1.5 to 3 mass%.

15 As phosphorus-based extreme pressure agents, there may be mentioned by way of example phosphoric acid esters, hypophosphoric acid esters, and amine salts etc of these. As phosphoric acid esters or hypophosphoric acid esters, any of the mono-, di- or tri-esters may be employed; as the alcohol residues thereof, there may be mentioned by
20 way of example: alkyl groups having a carbon number of from 4 to 30 such as for example butyl, octyl, lauryl, stearyl or oleyl groups; aryl groups having a carbon number of from 6 to 30, such as for example phenyl groups; or alkyl-substituted aryl groups having a carbon
25 number of from 7 to 30, such as for example methylphenyl or octylphenyl groups.

30 As sulfur/phosphorus-based extreme pressure agents, there may be mentioned by way of example blends obtained by combining the above sulfur-based extreme pressure agents and phosphorus-based extreme pressure agents, or sulfur/phosphorus-based compounds.

 As sulfur/phosphorus-based compounds, there may be mentioned by way of example thiophosphoric acid ester-

based compounds. As the thiophosphoric acid esters any of the mono-, di- or tri-esters may be employed; as the alcohol residues thereof, there may be mentioned by way of example: alkyl groups having a carbon number of from 4 to 30 such as for example butyl, octyl, lauryl, stearyl or oleyl groups; aryl groups having a carbon number of from 6 to 30, such as for example phenyl groups; or alkyl-substituted aryl groups having a carbon number of from 7 to 30, such as for example methylphenyl or octylphenyl groups.

One or two or more of these phosphorus-based additives may be employed: their blending amount may be from 0.05 to 0.3 mass% in terms of the amount of phosphorus, based on the total weight of the lubricating composition; preferably they may be employed in a range of from 0.1 to 0.25 mass%. The phosphorus-based additives have a considerable wear-preventing effect and also have an action as adjuvants in promoting the beneficial effect of sulfur-based additives as extreme pressure agents; acidic phosphoric acid esters and amine salts of acidic hypophosphoric acid esters are excellent in particular in terms of wear prevention.

As wear-preventing agents that may be employed in the present invention, there may be mentioned by way of example organometallic compounds, such as Zn-dialkyl dithiophosphates, Zn-dialkyl dithiocarbamates, Mo-dialkyl dithiophosphates or Mo-dialkyl dithiocarbamates. These can be employed alone or in combination and may be blended in the amount of from 0.01 to 5 mass% based on the total weight of the lubricating composition.

As the pour point depressants employed in the present invention, there may be mentioned by way of example polymers such as polymethacrylates. These

additives may be employed either alone or in combination in the range of from 0.01 to 5 mass% with respect to the total weight of the lubricating composition. The polymethacrylates employed as pour point depressants normally have an average molecular weight of about 100,000 and small molecular weight distribution; since the pour point improving effect differs depending on the length of the alkyl groups of the side chains, polymethacrylates with long side chains are effective in regard to base oils of high pour point, while polymethacrylates with short side chains are effective in regard to base oils of low pour point.

As antioxidants that may be employed in the present invention, there may be mentioned by way of example amine-based antioxidants, phenol-based antioxidants, sulfur-based antioxidants, or phosphorus-based antioxidants. The antioxidants that are ordinarily actually employed in lubricating compositions may be employed directly as these antioxidants. These antioxidants may be employed alone or in combination, in the range of from 0.01 to 5 mass% based on the total quantity of lubricating composition.

As metal deactivators that may be employed in the present invention, there may be mentioned by way of example benzotriazole derivatives, benzoimidazole derivatives, benzothiazole derivatives, benzo-oxazole derivatives, thiadiazole derivatives, and triazole derivatives. These metal deactivators may be employed either alone or in combination in a range of from 0.01 to 0.5 mass% based on the total quantity of lubricating composition.

As oiliness improvers that can be used in the present invention, for example fatty acid esters of

polyhydric alcohols may be employed. For example, partial or complete esters of saturated or unsaturated fatty acids having a carbon number of from 1 to 24 of polyhydric alcohols such as glycerol, sorbitol, alkylene glycols, neopentyl glycol, trimethylol propane, 5 pentaerythritol, or xylitol may be employed. These oiliness improvers may be employed alone or in combination in a range of from 0.01 to 5 mass% based on the total quantity of lubricating composition.

10 As antifoaming agents that can be employed in order to confer antifoaming properties according to the present invention, there may be mentioned by way of example organosilicates such as dimethyl polysiloxane, diethyl silicate, or fluorosilicones, or non-silicone-based 15 antifoaming agents such as polyalkyl acrylates. These antifoaming agents may be employed either alone or in combination in a range of from 0.0001 to 0.1 mass% based on the total quantity of lubricating composition.

20 As rust inhibitors that can be employed in the present invention, at least one type of additive may be employed selected from for example, chiefly, acid amines having a rust-preventing effect, sarcosinic acid or aspartic acid derivatives, succinic acid esters, or sulfonates. These rust inhibitors may be employed alone 25 or in combination in a range of from 0.01 to 0.1 mass% based on the total quantity of lubricating composition.

Lubricating compositions according to the present invention may be manufactured in which the base oil is blended with viscosity index improving agents as referred 30 to above and, in addition, if necessary, other additives may be blended therewith, the amounts and combinations of additives, etc being selected so as to achieve the aforementioned viscosity index and kinematic viscosity.

It is also preferable that the emulsibility of the lubricating composition according to the present invention is such that an emulsified layer persists for at least 60 even after 30 minutes, in accordance with
5 JISK2520.

The lubricating composition according to the present invention manufactured in this way can be employed in the same way as ordinary lubricating composition in ordinary lubrication systems, but is in particular suitable for
10 use as lubricating composition in lubricating systems that are not provided with means for removal of separated water, such as the drive systems of for example automobiles or agricultural vehicles, or constructional machinery vehicles. If the lubricating composition
15 according to the present invention is employed in such lubricating systems in which no means for removal of separated water is provided, even when infiltration of water occurs, emulsification takes place, with the result that there is no possibility of phase separation: seizure
20 of the sliding parts can thereby be prevented. The drive systems of vehicles such as automobiles, agricultural vehicles, or construction machinery comprise power transmission systems from the engine to the vehicle wheels or endless tracks and systems that include sliding
25 parts, such as manual gearboxes or differential devices or automatic gearboxes.

Although the viscosity properties of the lubricating composition according to the present invention may be somewhat lowered when infiltration of water into the
30 lubricating composition takes place, thanks to the blending of the aforementioned viscosity index improvers, the lubricating composition according to the present invention has excellent viscosity properties as

multigrade lubricating composition, so, albeit the viscosity properties are somewhat lowered, the lubricating composition according to the present invention can still maintain viscosity properties that enable use thereof as a drive system lubricating composition for vehicles such as automobiles. Such moisture that has infiltrated the lubricating composition is removed by evaporation during use, so the viscosity properties of the lubricating composition are recovered, making possible use over a long period.

Thanks to blending with the base oil of the lubricating composition according to the present invention with a viscosity index improver containing poly(meth)acrylate having hydroxyl groups in the structure and having a hydroxyl value of from 22 to 37, the lubricating composition according to the present invention has excellent compatibility for water so that, even if water infiltrates into the lubricating composition according to the present invention, such water is emulsified, preventing breakage of the oil film due to phase separation from occurring: seizure of sliding parts can thereby be prevented and, in addition, the lubricating composition according to the present invention has excellent viscosity properties as a multigrade lubricating composition; a lubricating composition capable of being suitably employed as a drive system lubricating composition for vehicles such as automobiles, agricultural vehicles or building machinery can thus be obtained.

The present invention is further described below specifically with reference to Reference Examples, Practical Examples and Comparative Examples: however, the present invention is not restricted solely to these

Practical Examples. In all the examples, % indicates weight%, and the methods of testing are as follows.

(1) Kinematic viscosity (Vk40): kinematic viscosity (mm²/s) at 40°C in accordance with JIS-K-2283

5 (2) Kinematic viscosity (Vk100): kinematic viscosity (mm²/s) at 100°C in accordance with JIS-K-2283

(3) Viscosity index: viscosity index in accordance with JIS-K-2283

Emulsibility Test Method:

10 This means the emulsibility of the lubricating composition with water, and is evaluated in accordance with the water separation test method of JIS K2520.

Test condition 1: Water (60 ml) + test oil (20 ml) are transferred to a test container, and allowed to
15 stabilise by resting for at least 30 minutes in a constant-temperature bath that is temperature-regulated to 54°C ± 1°C. Once the temperature is stabilised, a stirring paddle is inserted into the container containing the sample and stirring performed for five minutes at
20 1500 rpm and when stirring is completed a quantitative evaluation of the emulsified layer is carried out in terms of the time for which the emulsified layer persists and the emulsification condition.

Test condition 2: a test is conducted using the same
25 test procedure and evaluation criteria as in the case of Test Condition 1, apart from the fact that the sample of Test Condition 1 is replaced by a sample prepared using water (40 ml) + test oil (40 ml), and the resting period is changed from 20 to 30 minutes.

30 Four-Ball Wear Test:

Using a Shell-type four-ball test machine, the diameter of the wear pits on the fixed balls were found by performing a test using steel ball bearings, under the

conditions: rotational speeds 1200 rpm and 1500 rpm, load 400 N, temperature 75°C (1200 rpm) and temperature 80°C (1500 rpm), for a time of 30 minutes. Wear tests were also conducted using new oil according to the Practical Examples and Comparative Examples and emulsified oil on which the emulsification test had been performed.

The base oil and additives employed in the examples were as follows.

Base oil A: high-grade hydrogenated refined base oil, kinematic viscosity at 100°C, about 4 mm²/s, kinematic viscosity at 40°C: 19.5 mm²/s, viscosity index: 123.

Hydroxyl group-containing polymethacrylate (PA): hydroxyl value: 28, numerical average molecular weight: 10,000, mass average molecular weight: 15,000, net polymer: 54 mass%.

Polymethacrylate (PB) not containing hydroxyl groups: Aclube 728 (trade name, manufactured by Sanyo Chemical Industries Ltd): numerical average molecular weight: 22,000, mass average molecular weight: 40,000, net polymer: 30 mass%.

Polymethacrylate (PC) not containing hydroxyl groups: Aclube 504 (trade name, manufactured by Sanyo Chemical Industries Ltd): numerical average molecular weight: 70,000, mass average molecular weight: 180,000, net polymer: 20 mass%.

Sulfur-phosphorus-based extreme pressure agent: Anglamol 99 (trade name, manufactured by Lubrizol Ltd).

Pour point depressant: Lubran VI-721 (trade name, manufactured by Toho Chemicals Ltd).

Examples

Reference Example 1

A sample prepared by mixing 20 weight% of hydroxyl group-containing polymethacrylate (PA) with the above

base oil A was subjected to an emulsification test in accordance with the above Test Condition 1, and the emulsification condition was evaluated. The volume of the emulsified layer 30 minutes after stirring emulsification was 70 ml.

Reference Example 2

A sample prepared by mixing 15 weight% of hydroxyl group-containing polymethacrylate (PA) with the above base oil A and 2.5 weight% of ordinary polymethacrylate (PB) not containing hydroxyl groups was subjected to an emulsification test in accordance with the above Test Condition 1. The volume of the emulsified layer 30 minutes after stirring emulsification was 67 ml.

Comparative Reference Example 1

A sample prepared by mixing 10 weight% of ordinary polymethacrylate (PB) not containing hydroxyl groups with the above base oil A was subjected to an evaluation in accordance with the above Test Condition 1. The volume of the emulsified layer 30 minutes after stirring emulsification was 0 ml.

The results obtained with the above Reference Examples and Comparison Reference Examples are shown in Table 1; whereas the Reference Example 1 and Reference Example 2 show an emulsified layer of 60 ml or more, in the case of the Comparison Reference Example 1, which does not contain hydroxyl group-containing polymethacrylate (PA), no emulsified layer at all was generated.

Table 1

		Reference Example 1	Reference Example 2	Comparison Reference Example 1
Blending composition	Base oil A (%)	80	82.5	90
	PA (%)	20	15	0
	PB (%)	0	2.5	10
Before water separation test	Aqueous layer (ml)	60	60	60
	Oil layer (ml)	20	20	20
	Emulsified layer (ml)	0	0	0
After water separation test	Aqueous layer (ml)	10	13	60
	Oil layer (ml)	0	0	20
	Emulsified layer (ml)	70	67	0

Practical Examples 1 to 3

As the lubricating composition of Practical Examples 1 to 3, lubricating composition was manufactured by admixing, so as to give a viscosity grade of 75W-80, 10% of hydroxyl group-containing polymethacrylate (PA), 7.5 weight% of ordinary polymethacrylate, not containing hydroxyl groups (PC), 1% of pour point depressant, and, in addition, sulfur-phosphorus-based extreme pressure agents in an added amount, in terms of the oil performance classification, based on the API service classification GL-3 (Practical Example 1), GL-4 (Practical Example 2) and G-5 (Practical Example 3). In respect of these lubricating compositions, the kinematic viscosity (Vk100) at 100°C and the kinematic viscosity (Vk40) at 40°C, and the viscosity index (VI) were measured, and the diameter of the wear pits (mm) at a temperature of 75°C (1200 rpm) and at a temperature of 80°C (1500 rpm) were found by the four-ball wear test referred to above. The results are shown in Table 2.

Table 2

	Practical Example 1	Practical Example 2	Practical Example 3
Base oil A (%)	75.5	77.5	79.5
PA (%)	10	10	10
PC (%)	7.5	7.5	7.5
Pour point depressant (%)	1	1	1
Extreme pressure agent (%)	6	4	2
Total additives (%)	24.5	22.5	20.5
Vk100 (mm ² /s)	10.18	9.999	9.779
Vk40 (mm ² /s)	42.85	41.46	40.07
VI	236	240	242
Wear pit diameter 75°C (mm)	0.47	0.41	0.40
Wear pit diameter 80°C (mm)	0.42	0.44	0.38

Comparative Examples 1 to 3

The lubricating composition of Comparative Examples 1 to 3 was manufactured by, in Practical Examples 1 to 3, dispensing with blending with hydroxyl group-containing polymethacrylate (PA) and blending only with ordinary polymethacrylate (PC). In respect of these lubricating compositions, the kinematic viscosity (Vk100) at 100°C and the kinematic viscosity (Vk40) at 40°C, and the viscosity index (VI) were measured in the same way, and the diameter of the wear pits (mm) at a temperature of 75°C (1200 rpm) and at a temperature of 80°C (1500 rpm) were found by the four-ball wear test referred to above. The results are shown in Table 3.

Table 3

	Comparative Example 1	Comparative Example 2	Comparative Example 3
Base oil A (%)	83	85	87
PA (%)	0	0	0
PC (%)	10	10	10
Pour point depressant (%)	1	1	1
Extreme pressure agent (%)	6	4	2
Total additives (%)	17	15	13
Vk100 (mm ² /s)	9.870	9.674	9.475
Vk40 (mm ² /s)	39.47	38.29	37.03
VI	249	252	255
Wear pit diameter 75°C (mm)	0.49	0.43	0.41
Wear pit diameter 80°C (mm)	0.45	0.48	0.39

From the results of the above Table 1 and Table 2, it was found that whereas the extreme pressure agent added amounts were the same and the wear conditions were the same for both Practical Example 1 and Comparative Example 1, Practical Example 2 and Comparative Example 2 and Practical Example 3 and Comparative Example 3, by comparing these examples, superior results were obtained for the wear resistance in the case of Practical Examples 1 to 3, in which hydroxyl group-containing polymethacrylate (PA) was added, compared with Comparative Examples 1 to 3, under both test conditions of the four-ball wear test.

For the lubricating compositions of the above Practical Example 1 and Comparative Example 1, Table 4 shows the results of performance of a four-ball wear test (temperature 75°C (1200 rpm)) after emulsification in accordance with the test condition 2 of the above-mentioned emulsification test, and also shows the

emulsification (separation) condition after 20 minutes and after 30 minutes of emulsification.

Table 4

		Practical Example 1	Comparative Example 1
After resting for 20 minutes	Aqueous layer (ml)	00	40
	Oil layer (ml)	00	40
	Emulsified layer (ml)	80	0
After resting for 20 minutes	Aqueous layer (ml)	0	40
	Oil layer (ml)	0	40
	Emulsified layer (ml)	80	0
Emulsification condition		Completely emulsified	Completely separated
Wear pit diameter	75°C (mm)	1.29	Severe seizure

As shown in Table 4, although, in Practical Example 1, the wear pit diameter was larger than in the case where no water had entered (Table 2), in Comparative Example 1, seizure took place immediately after rotation. The reason for this is inferred to be that, whereas, in the case of Practical Example 1, an oil film was formed because the water was completely emulsified, in the case of Comparative Example 1, oil/water separation took place, with the result that no oil film was formed on the sliding parts. As a result, with the lubricating composition according to the present invention, it is believed that seizure was prevented due to incorporation and emulsification of the water.

The present invention can be employed as lubricating composition in typical lubrication systems, and, in particular, can be employed as lubricating composition in lubrication systems in which no means are provided for removal of separated water, such as for example as the lubricating composition for the drive systems of vehicles

such as automobiles, agricultural vehicles, or
constructional machines.

C L A I M S

1. Lubricating composition comprising:
- a base oil; and
- a viscosity index improver containing poly(meth)acrylate having hydroxyl groups in the structure and having a hydroxyl value of from 22 to 37.
- 5
2. The lubricating composition according to claim 1 wherein the viscosity index improver contains from 50 to 100 mass parts of poly(meth)acrylate having hydroxyl groups in the structure, and from 50 to 0 mass parts of poly(meth)acrylate not having hydroxyl groups in the structure.
- 10
3. The lubricating composition according to claim 1 or 2 wherein the poly(meth)acrylate of the viscosity index improver has a mass average molecular weight of from 5,000 to 50,000.
- 15
4. The lubricating composition according to any of claims 1 to 3 wherein the viscosity index improver is present in an amount of from 1 to 8 mass%, based on the total weight of the lubricating composition, as net polymer, excluding diluent oil.
- 20
5. The lubricating composition according to any of claims 1 to 4, further containing another additive selected from extreme pressure agents, anti-wear additives, pour point depressants, antioxidants, metal deactivators, oiliness agents, defoaming agents and rust inhibitors.
- 25
6. Lubricating composition according to any of claims 1 to 5 for use in the drive systems of vehicles.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2010/057990

A. CLASSIFICATION OF SUBJECT MATTER INV. C10M145/14 ADD. C10N40/04 According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) C10M Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2008/053033 A2 (SHELL INT RESEARCH [NL]; MARUYAMA RYUJI [JP]; NAGATOMI EIJI [JP]; WAKI) 8 May 2008 (2008-05-08) page 1, line 1 - line 15 page 7, line 1 - page 10, line 25 page 15, line 26 - page 16; claims; table 1	1-6
X	DE 103 35 360 A1 (SANYO CHEMICAL IND LTD [JP]) 12 February 2004 (2004-02-12) paragraphs [0016], [0020]; claims; table 1	1-6
A	EP 0 569 639 A1 (ROHM & HAAS [US]) 18 November 1993 (1993-11-18) page 2, line 1 - line 16; claims ----- -/--	1-6
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents :		
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family	
Date of the actual completion of the international search	Date of mailing of the international search report	
17 August 2010	25/08/2010	
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Glod, Guy	

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2010/057990

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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