

5 FATTY ACID METHYL ESTER COMPOSITION HAVING A LOW POUR
POINT

10 FIELD OF THE INVENTION

The present invention relates to a composition based on fatty acid methyl esters, derived from castor oil, having a low pour point. This composition is of use especially as lubricant or to facilitate the release of concrete.

15 TECHNICAL BACKGROUND

It is known to transesterify plant or animal oils, which are mixtures of different fatty acids, in order to produce products of use for example for lubrication.

20 The composition of fatty acids varies considerably from one plant or
animal oil to another, to the extent that each transesterified oil has different
properties.

A particularly useful product, especially of use as lubricant or as concrete release agent, is a mixture of fatty acid methyl esters derived from castor oil. This product predominantly contains C11-C22 fatty acid methyl esters, the main examples of which are methyl linoleate and methyl oleate. This product also contains two specific compounds which are generally not found in products derived from other plant oils, namely 10-undecylenic acid and methyl ricinoleate.

30 This product is especially sold by Arkema under the name Oleris® Esterol A. However, one drawback of this product is that it has a relatively high pour point, around +6°C (according to standard ASTM D97). For this reason, the use of the product is difficult in cold weather.

It is known to use pour point depressant additives for lowering the pour
35 point of various products derived from plant or animal oils.

Thus, documents EP 0543356, EP 0626442, US 6,409,778, US 6,051,538 and US 2011/296743 describe various compositions in which pour point depressant additives are used.

However, generally speaking, the possibility of depressing the pour point of a product derived from a plant or animal oil in order to enable the use thereof in cold weather depends on the nature and the properties of this product. Consequently, there is a need to be able to improve a mixture of fatty acid methyl esters derived from castor oil so as to enable the use thereof in cold weather.

SUMMARY

In an aspect, there is provided a composition comprising:

- at least 95% by weight of a mixture of castor oil fatty acid methyl esters containing 10-undecylenic acid and methyl ricinoleate;
- a pour point depressant additive;

the pour point of the composition being less than or equal to 0°C according to standard ASTM D97, wherein the pour point depressant additive is an acrylic polymer dissolved in an oil.

There is provided a composition comprising:

- at least 95% by weight of a mixture of castor oil fatty acid methyl esters;
- a pour point depressant additive;

the pour point of the composition being less than or equal to 0°C.

According to one embodiment, the pour point of the composition is less than or equal to -5°C, preferably less than or equal to -9°C.

According to one embodiment, the mixture of castor oil fatty acid methyl esters has an iodine number of 100 to 120 g / 100 g and comprises, in proportions by weight:

- from 5 to 20%, preferably from 8 to 12%, of methyl stearate;
- from 20 to 40%, preferably from 25 to 35%, of methyl oleate;
- from 30 to 50%, preferably from 35 to 45%, of methyl linoleate;
- less than 10%, preferably less than 8.5%, of methyl palmitate;
- from 1.5 to 4%, preferably from 2 to 3%, of methyl ricinoleate;
- from 0.4 to 1.5%, preferably from 0.6 to 1%, of 10-undecylenic acid.

According to one embodiment, the composition comprises a proportion by weight of pour point depressant additive of 1 to 2%, preferably 1.2 to 1.8%.

According to one embodiment, the pour point depressant additive is an acrylic polymer (preferably in accordance with the general formula presented below) dissolved in an oil, the proportions preferably being from 60 to 100 parts of acrylic polymer per 15 to 40 parts of oil.

According to one embodiment, the oil is a paraffinic distillate.

According to one embodiment, the additive has:

- a viscosity at 100°C according to standard ASTM D445 of 200 to 500 mm²/s, preferably 300 to 400 mm²/s;
- a pour point according to standard ASTM D97 of less than or equal to -30°C, preferably less than or equal to -36°C;

- an acid number according to standard ASTM D974 of less than or equal to 0.6 mg KOH/g, preferably less than or equal to 0.5 mg KOH/g.

In another aspect, there is provided the use of the above composition as lubricant, concrete release agent, degreasing agent or cleaning agent.

5 The present disclosure makes it possible to overcome the disadvantages of the state of the art. It more particularly provides a mixture of fatty acid methyl esters derived from castor oil, which may be used in cold weather.

10 This is achieved by virtue of the use of an additive making it possible to depress the pour point of the mixture to less than 0°C, preferably to less than -5°C or to less than -9°C.

BRIEF DESCRIPTION OF THE FIGURES

15 **Figure 1** represents the crystallization kinetics of various compositions, estimated by the thickness of the layer of crystals formed (on the y-axis, in mm) as a function of the temperature (on the x-axis, in °C). The details are featured in example 1 below.

DESCRIPTION OF EMBODIMENTS OF THE INVENTION

20 The invention is now described in greater detail and in a nonlimiting manner in the description which follows.

 The composition according to the invention is obtained by mixing a mixture of fatty acid methyl esters derived from castor oil and a pour point depressant additive.

25 The mixture of fatty acid methyl esters derived from castor oil is obtained by transesterification of a castor oil with methanol. The degree of transesterification of the mixture is not necessarily 100%, which means that the mixture of methyl esters in question may comprise a certain proportion of non-esterified fatty acids.

30 The mixture of fatty acid methyl esters derived from castor oil is preferably characterized by a pour point according to standard ASTM D97 of 5 to 8°C; and/or by an iodine number of 100 to 120 g/100 g. The iodine number may be determined according to standard ISO 3961.

 The mixture of fatty acid methyl esters in question preferably comprises:

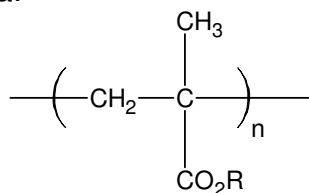
- from 5 to 20%, preferably from 8 to 12%, of methyl stearate (C18:0);

- from 20 to 40%, preferably from 25 to 35%, of methyl oleate (C18:1);
- from 30 to 50%, preferably from 35 to 45%, of methyl linoleate (C18:2);
- 5 - less than 10%, preferably less than 8.5%, of methyl palmitate (C16:0);
- from 1.5 to 4%, preferably from 2 to 3%, of methyl ricinoleate (C18:1(OH));
- from 0.4 to 1.5%, preferably from 0.6 to 1%, of 10-undecylenic acid (C11:1).

The pour point depressant additive is chosen such that it makes it possible to depress the pour point of the composition according to the invention below 0°C (preferably below -1°C, or -2°C, or -3°C, or -4°C, or -5°C, or -6°C, or -7°C, or -8°C, or -9°C, according to standard ASTM D97).

15 It may especially be an acrylic polymer (which includes acrylic copolymers) dissolved in an oil. The relative proportions are preferably 60 to 100 parts of acrylic polymer per 15 to 40 parts of oil.

The acrylic polymer is preferably a polyalkyl methacrylate of general formula:



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wherein n is an integer preferably from 200 to 2000, and each R independently represents a C1 to C22 alkyl group.

The oil may for example be obtained by catalytic hydrogenation of a petroleum fraction, and predominantly comprise predominantly saturated C15 to C30 hydrocarbons and have a viscosity of less than 19 cSt at 40°C. It may especially be the oil with the reference number CAS 64742-55-8.

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The oil may also be obtained by catalytic hydrogenation of a petroleum fraction, and predominantly comprise predominantly saturated C20 to C30 hydrocarbons and have a viscosity of greater than 19 cSt at 40°C. It may especially be the oil with the reference number CAS 64742-54-7.

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The oil may also be obtained from a petroleum fraction by eliminating paraffins by crystallization in a solvent, and predominantly comprise C20 to C30 hydrocarbons and have a viscosity of greater than 19 cSt at 40°C. It may especially be the oil with the reference number CAS 64742-65-0.

It is also possible to use a mixture of the above oils.

The additive preferably has:

- a viscosity at 100°C according to standard ASTM D445 of 200 to 500 mm²/s, preferably 300 to 400 mm²/s; and/or
- 5 – a pour point according to standard ASTM D97 of less than or equal to -30°C, preferably less than or equal to -36°C; and/or
- an acid number according to standard ASTM D974 of less than or equal to 0.6 mg KOH/g, preferably less than or equal to 0.5 mg KOH/g.

10 A particularly suitable additive is the product sold by Evonik under the reference VISCOPLEX® 1-211.

The additive may be present in the composition in a proportion by weight from 0.1 to 5% and preferably from 1 to 2%. Preferred content ranges are: from 1.0 to 1.1%; from 1.1 to 1.2%; from 1.2 to 1.3%; from 1.3 to 1.4%;
15 from 1.4 to 1.5%; from 1.5 to 1.6%; from 1.6 to 1.7%; from 1.7 to 1.8%; from 1.8 to 1.9% and from 1.9 to 2.0%. A content by weight of approximately 1.5% is particularly suitable.

The content of acrylic copolymer or polymer in the composition of the invention is preferably from 0.5 to 2%, more particularly preferably from 0.8 to 1.4% (especially from 0.8 to 0.9%, or from 0.9 to 1.0%, or from 1.0 to 1.1%,
20 or from 1.1 to 1.2%, or from 1.2 to 1.3%, or from 1.3 to 1.4%).

According to one embodiment, the composition essentially consists of the mixture of fatty acid methyl esters derived from castor oil and the pour point depressant additive. According to another embodiment, the composition
25 consists of the mixture of fatty acid methyl esters derived from castor oil and a pour point depressant additive.

EXAMPLES

The following examples illustrate the invention without limiting it.

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Example 1

Various pour point depressant additives are added at an amount of 1 to 2.5% into a mixture of fatty acid methyl esters derived from castor oil sold under the name Oleris® Esterol A by Arkema (pour point: +6°C).

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The additives used are as follows:

- VISCOPLEX® 10-310 (Evonik): acrylic copolymer in an amount of greater than or equal to 60%, combined with rapeseed oil, having a viscosity of 1800 mm²/s at 100°C;

- VISCOPLEX® 1-211 (Evonik): acrylic polymer in an amount of greater than or equal to 60%, combined with a neutral oil, having a viscosity of 400 mm²/s at 100°C;
- 5 - VISCOPLEX® 1-180 (Evonik): acrylic copolymer in an amount from 40 to 70%, combined with a neutral oil, having a viscosity of 300 mm²/s at 100°C;
- VISCOPLEX® 1-255 (Evonik): acrylic copolymer in an amount from 15 to 40%, combined with a neutral oil, having a viscosity of 45 mm²/s at 100°C.

10 The pour points of the compositions obtained in this way are summarized in the following table:

	Additive			
Concentration of the additive	VISCOPLEX® 10-310	VISCOPLEX® 1-211	VISCOPLEX® 1-180	VISCOPLEX® 1-255
1%	+1°C	-2°C	0°C	+4°C
1.5%	-4°C	-9°C	-5°C	-1°C
2%	-6°C	-10°C	-8°C	-4°C
2.5%	-4°C	-5°C	-10°C	-6°C

Pour point (ASTM D97)

15 It is observed that the additive VISCOPLEX® 1-211 is particularly beneficial since it makes it possible to depress the pour point to -9°C at a concentration of just 1.5%.

Moreover, measurements of crystallization kinetics were carried out between 5 and -3°C, by measuring the thickness of the layer of crystals formed in a sample kept in a test tube (filling height of 55 mm). These measurements related to four compositions produced with 1.5% of the four additives above, and also to a control sample of Oleris® Esterol A. The results are shown in **figure 1**. They demonstrate that the additive VISCOPLEX® 1-211 is that which offers the slowest crystallization kinetics.

25 The curve 1 represents the control composition without additive, the curve 2 represents the composition produced with VISCOPLEX® 1-255, the curve 3 represents the composition produced with VISCOPLEX® 1-180, the curve 4 represents the composition produced with VISCOPLEX® 10-310 and the curve 5 represents the composition produced with VISCOPLEX® 1-211.

Example 2

The consistency of various compositions prepared with the same products as in example 1 was evaluated after storing under static conditions at -7°C for 7 days.

The results are presented in the table below:

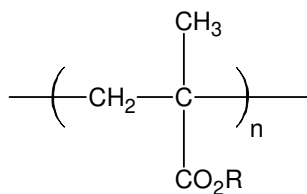
Product	State
Control (no additive)	Crystallized
VISCOPLEX® 10-310 at 1%	Set, but flows if vial is shaken
VISCOPLEX® 1-211 at 1%	Very fluid, flows by itself
VISCOPLEX® 1-180 at 1%	Heterogeneous, flows slightly, liquid if vial is shaken
VISCOPLEX® 1-255 at 1%	Set
VISCOPLEX® 10-310 at 1.5%	Pasty, but fluid if vial is shaken
VISCOPLEX® 1-211 at 1.5%	Very fluid, flows by itself
VISCOPLEX® 1-180 at 1.5%	Heterogeneous liquid, large clumps
VISCOPLEX® 1-255 at 1.5%	Very pasty but flows if vial is shaken
VISCOPLEX® 10-310 at 2%	Set and only very slightly flows if vial is shaken
VISCOPLEX® 1-211 at 2%	Relatively fluid
VISCOPLEX® 1-180 at 2%	Fluid
VISCOPLEX® 1-255 at 2%	Very fluid
VISCOPLEX® 10-310 at 2.5%	Set
VISCOPLEX® 1-211 at 2.5%	Relatively fluid
VISCOPLEX® 1-180 at 2.5%	Fluid
VISCOPLEX® 1-255 at 2.5%	Very fluid

Homogeneity and fluidity of the products

The compositions obtained with VISCOPLEX® 1-211 are the most advantageous in terms of homogeneity and fluidity, for a content of additive which remains moderate (1.5 %).

CLAIMS

1. A composition comprising:
 - at least 95% by weight of a mixture of castor oil fatty acid methyl esters containing 10-undecylenic acid and methyl ricinoleate;
 - a pour point depressant additive;the pour point of the composition being less than or equal to 0°C according to standard ASTM D97, wherein the pour point depressant additive is an acrylic polymer dissolved in an oil.
2. The composition as claimed in claim 1, the pour point of which is less than or equal to -5°C, preferably less than or equal to -9°C.
3. The composition as claimed in claim 1 or 2, wherein the mixture of castor oil fatty acid methyl esters has an iodine number of 100 to 120 g / 100 g and comprises, in proportions by weight:
 - from 5 to 20%, preferably from 8 to 12%, of methyl stearate;
 - from 20 to 40%, preferably from 25 to 35%, of methyl oleate;
 - from 30 to 50%, preferably from 35 to 45%, of methyl linoleate;
 - less than 10%, preferably less than 8.5%, of methyl palmitate;
 - from 1.5 to 4%, preferably from 2 to 3%, of methyl ricinoleate;
 - from 0.4 to 1.5%, preferably from 0.6 to 1%, of 10-undecylenic acid.
4. The composition as claimed in one of claims 1 to 3, comprising a proportion by weight of pour point depressant additive of 1 to 2%, preferably 1.2 to 1.8%.
5. The composition as claimed in one of claims 1 to 4, wherein the pour point depressant additive has proportions of 60 to 100 parts of acrylic polymer per 15 to 40 parts of oil.
6. The composition as claimed in one of claims 1 to 5, wherein the acrylic polymer is of formula:



wherein n is an integer preferably from 200 to 2000, and each R represents a C1 to C22 alkyl group.

7. The composition as claimed in claim 5 or 6, wherein the oil is a paraffinic distillate.
8. The composition as claimed in one of claims 1 to 7, wherein the additive has:
 - a viscosity at 100°C according to standard ASTM D445 of 200 to 500 mm²/s, preferably 300 to 400 mm²/s;
 - a pour point according to standard ASTM D97 of less than or equal to -30°C, preferably less than or equal to -36°C;
 - an acid number according to standard ASTM D974 of less than or equal to 0.6 mg KOH/g, preferably less than or equal to 0.5 mg KOH/g.
9. The use of the composition as claimed in one of claims 1 to 8 as lubricant, concrete release agent, degreasing agent or cleaning agent.