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FATTY ACID COMPOSITIONS OF METHYL ESTER FOR ESTERQUATS
PRODUCTION SUITABLE FOR FABRIC SOFTENER

5 FIELD OF INVENTION

The present invention relates to a composition of saturated fatty acid and unsaturated fatty acid methyl ester for the production of esterquats. In more particular, this invention relates to a composition of methyl ester for the production of esterquats
10 having good fabric softening and rewetting properties. Accordingly, they are suitable for the production of fabric softeners.

BACKGROUND OF THE INVENTION

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Esterquats are cationic surfactants generally known by those skilled in the art to be quaternized fatty acid triethanolamine ester salts. Esterquats are typically used for softening fibers or preparing cosmetics owing to their excellent fabric-softening effect and high ecotoxicological compatibility.

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The esterquats are generally produced in a two-stage process, in which methyl ester is transesterified with triethanolamine and the resulting triethanolamine esters are subsequently alkylated or quaternized with dimethyl sulfate in the presence of a catalyst, such as isopropanol, to yield esterquats as a biodegradable cationic
25 surfactant.

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There are a few patented technologies over the prior arts relating to esterquats with fabric-softening properties. These patented technologies include a wide range of the production and applications of esterquats. Of interest in connection with a fabric
30 softening composition containing esterquats with specific ester distribution and

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sequestrant is U. S. Patent No. US2004097395. This invention discloses an aqueous
rinse cycle fabric softening composition containing compound which remains
physically stable and is characterized by a stable viscosity over a wide range of
ambient temperatures and for extended periods of time. The composition comprises
5 1% to 25% of quaternary ammonium compound and 0.001% to 2% of sequestrant.

There is an European Patent No. EP1160238 disclosing an esterquat produced from
methyldiethanolamine using dibasic acids and fatty acids. The esterquats invented
have improved properties, such as softness, dispersibility and high biodegradability;
10 therefore suitable to be used as a component in rinse cycle fabric softeners, dryer
sheets, hair conditioners, paper debonders and other applications that requires a high
degree of softness.

U. S. Patent No. 5296622 discloses an invention of esterquats obtained from
15 unsaturated fatty acids containing at least 40 mol-% trans-configured double bonds or
ester thereof with alkanolamines. The reaction products are subsequently quaternized
with alkylating agents to produce an end products as fabric softeners. Esterquats of
saturated fatty acids and alkanolamines used as fabric softeners are also described in
European Patent No. DE1619058 and DE179468. Particular significance is attributed
20 in this regard to products obtained by reaction between 2mol of saturated fatty acid
with 1mol of triethanolamine, and subsequent quaternization with dimethyl sulfate or
methyl chloride. Although fabrics treated with quaternized esters of this type show
good softness, they often exhibit unwanted hydrophobicity, which is reflected in poor
rewettability or water adsorption of the treated fabrics.

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Another U. S. Patent No. US3915867 also describes the quaternized esters of a
mixture of saturated and unsaturated fatty acids, for example of the type readily
obtained by hydrolysis of natural fats and oils, showing good softening power with
triethanolamine. However, the quaternized esters based on unsaturated fatty acids and
30 alkanolamines have shown no fabric-softening properties. Therefore, mixtures of

quaternized esters based on both saturated and unsaturated fatty acids significantly exhibit poorer performance than those quaternized esters produced by using only saturated fatty acids.

5 Nowadays, vegetable-based raw materials are gaining acceptance in the fabric softener market over the animal-based raw material. The vegetable-based feedstock can be obtained from various seeds such as soybean, sunflower, oil palm fruits, coconut and rapeseed. Nevertheless, most vegetable-based oils and fats have low saturated long chain fatty acid and therefore not applicable for the use in the
10 formulation of fabric softener. Partial or full hydrogenation is required for most of the vegetable oils to obtain acceptable fabric softening performance due to their high degree of unsaturation, especially polyunsaturates and lesser saturated C₁₆ and C₁₈ as compared to those of the fatty acids from animal fats. Despite the better softening power found in long and fully saturated hydrocarbon-based esterquats, increase in the
15 degree of saturation will tend to increase the hydrophobicity effect on the fabric and results in poor rewetting properties.

A major drawback of the esterquats disclosed by the prior arts is that the esterquats invented do not possess good rewetting properties while providing good fabric-
20 softening effect. In view of this drawback of the prior arts, it is desirable for the present invention to develop an innovative and improved composition of esterquats that free from the above-mentioned disadvantages by incorporating a certain amount of unsaturated hydrocarbon in the composition of esterquats.

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SUMMARY OF INVENTION

The primary object of the present invention is to develop a composition of esterquats which possesses good softening and rewetting properties which enables it to be used
30 in the formulation of a fabric softener.

Another object of the present invention is to utilize raw material with suitable fatty acid compositions which contains both saturated and unsaturated fatty acid methyl esters in a suitable ratio for the production of esterquats.

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Yet another object of the present invention is to provide a saturated and unsaturated fatty acid composition for the production of esterquats that offers optimum rewetting characteristics, provides water repellency and at the same time exhibits good fabric-softening power.

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At least one of the preceding objects is met, in whole or in part, by the present invention, in which one of the embodiments of the present invention describes a composition of esterquats, comprising a mixture of methyl esters, characterized by 40% to 95% of saturated fatty acid methyl esters and 5% to 60% of unsaturated fatty acid methyl esters, transesterified with triethanolamine and quaternized with dimethyl sulfate.

Another embodiment of the present invention is a fabric softener comprising a composition of esterquats, comprising a mixture of methyl esters, characterized by 40% to 95% of saturated fatty acid methyl esters and 5% to 60% of unsaturated fatty acid methyl esters, transesterified with triethanolamine and quaternized with dimethyl sulfate.

One skilled in the art will readily appreciate that the present invention is well adapted to carry out the objects and obtain the ends and advantages mentioned, as well as those inherent therein. The embodiments described herein are not intended as limitations on the scope of the invention.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a composition of saturated fatty acid and unsaturated fatty acid methyl ester for the production of esterquats. In more particular, the present invention relates to a composition of methyl ester for the production of esterquats having good fabric softening and rewetting properties. Accordingly, they are suitable for the production of fabric softeners.

Hereinafter, the invention shall be described according to the preferred embodiments of the present invention and by referring to the accompanying description and drawings. However, it is to be understood that limiting the description to the preferred embodiments of the invention and to the drawings is merely to facilitate discussion of the present invention and it is envisioned that those skilled in the art may devise various modifications without departing from the scope of the appended claim.

The present invention discloses a composition of esterquats, comprising a mixture of methyl esters, characterized by 40% to 95% of saturated fatty acid methyl esters and 5% to 60% of unsaturated fatty acid methyl esters, transesterified with triethanolamine and subsequently quaternized with dimethyl sulfate.

The present invention is characterized by the incorporation of a certain amount of unsaturated fatty acid methyl esters into the saturated fatty acid methyl esters to be used in the production of esterquats. According to the preferred embodiment of the present invention, the mixture of methyl esters contains 40% to 95% of saturated fatty acid methyl esters and 5% to 60% of unsaturated fatty acid methyl esters. Preferably, the saturated fatty acid is present in an amount of 60% to 90% whereas the unsaturated acid is present in an amount of 5% to 26% by weight of the methyl esters.

In accordance with the preferred embodiment of the present invention, the methyl esters are derived from vegetable oils or animal fats. Preferably, the methyl esters are

derived from palm oil, soybean oil, corn oil, cottonseed oil, peanut oil, olive oil, canola oil, sunflower oil, coconut oil, palm stearin, palm kernel oil, lard, tallow or derivatives thereof or a combination of one or more thereof. A person skilled in the art should appreciate the fact that oils from other sources can also be used if they possess
5 suitable ratio of saturated fatty acids to unsaturated fatty acids.

The starting material for the production of esterquats, which is capable both of providing fabrics with good softness and imparting good rewetting performance is methyl esters having certain ratio of palmitic, stearic and oleic along with other minor
10 amounts of fatty acid substances. As described by the most preferred embodiment of the present invention, suitable saturated fatty acid is palmitic acid, stearic acid or a combination thereof, whereas the suitable unsaturated fatty acid is oleic acid. In the most preferred embodiment of the present invention, the mixture of methyl esters contains 68% of palmitic and stearic acid as its saturated fatty acids component and
15 26% of oleic acid as its unsaturated fatty acids component.

Another embodiment of the present invention is a fabric softener comprising a composition of esterquats, comprising a mixture of methyl esters, characterized by 40% to 95% of saturated fatty acid methyl esters and 5% to 60% of unsaturated fatty
20 acid methyl esters, transesterified with triethanolamine and quaternized with dimethyl sulfate. The fabric softener invented is capable of offering optimum rewetting characteristics, providing water repellency as well as exhibiting good fabric-softening power.

25 The fatty acids of the methyl ester suitable for fabric softening formulations are obtained by pre-mixing of hydrogenated methyl ester (full saturation) with 98.7% mixture of palmitic acid and stearic acid, and non-hydrogenated methyl ester with 36.4% mixture of palmitic acid and stearic acid, 53.2% of oleic acid and other minor amounts of fatty acid substances. Although esterquats derived from fully saturated
30 palm-based methyl esters exhibit better softening performance compared to esterquats

from non-hydrogenated palm-based methyl esters, the rewetting ability is reduced due to the hydrophobicity effect caused by the fully saturated fatty acid chain lengths in the feedstock of the oils or fats. Degree of unsaturation will affect the rewetting power and this effect can be observed from a plot of graph, where enhancement of the
5 rewetting power was observed with the incorporation of unsaturated fatty acids in the esterquats production.

According to the preferred embodiment of the present invention, the esterquats derived from methyl ester having certain amount of palmitic acid, stearic acid and
10 oleic acid exhibit comparable softening performance to palm stearin fatty acid-based and tallow-based commercial esterquats without reducing the rewetting property.

The present disclosure includes as contained in the appended claims, as well as that of the foregoing description. Although this invention has been described in its preferred
15 form with a degree of particularity, it is understood that the present disclosure of the preferred form has been made only by way of example and that numerous changes in the details of construction and the combination and arrangements of parts may be resorted to without departing from the scope of the invention.

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EXAMPLE

The fatty acids compositions of methyl ester for esterquats production suitable for fabric softener formulation and the esterquats derived from methyl ester having these
25 fatty acid compositions are shown in Table 1 and Table 2.

Table 1: Various Fatty Acid Compositions of Methyl Ester For Esterquats Production

	Methyl Ester (A)	Methyl Ester (B)	Methyl Ester (C)
C12:0	0.3	0.5	0.7
C14:0	0.7	1.0	1.3
C16:0	58.0	45.3	32.3
C16: 1	0.1	0.1	0.1
C18:0	34.9	22.9	10.7
C18: 1	5.0	26.3	48.0
C18: 2	0.6	3.4	6.4
C18: 3	-	0.1	0.1
C20: 0	0.3	0.3	0.3

Table 2: Properties of Esterquats Derived From Methyl Ester of Various Fatty Acid Compositions

	Esterquats (A)	Esterquats (B)	Esterquats (C)
Appearance	Paste	Paste	Paste
Active Matter (%)	60	51.9	60.1
Colour Lovibond	4.9R	3.8R	5.4R
5% pH Solution	3.17	3.31	3.46
Total Solid Content (%)	90.84	90.22	90.78

5 Example 1

Production of Palm-Based Quaternized Triethanolamine Ester Using Methyl Ester Having Compositions of 68% Palmitic and Stearic and 26% Oleic As Starting Material.

- 10 a. **Transesterification.** 3 kg of methyl ester having compositions of 68% palmitic acid and stearic acid with 26% of oleic acid, 0.890 kg of triethanolamine and 0.15 kg sodium methoxide in the form of 1% by weight aqueous solution are introduced into a reactor equipped with mechanical stirrer. The reaction mixture is heated to 100°C within 30 minutes with a
- 15 vacuum pressure of 200 mbar. The reaction mixture is stirred at a stirring rate

of 540 rpm. These conditions are maintained for 1 to 1.5 hours or until the amine value of the reaction mixture reaches maximum constant value. The palm stearin triethanolamine ester is then cooled to 60°C with continuous stirring and vacuum pressure maintained at 200 mbar.

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b. **Quaternization.** Subsequently, the above reaction mixture of 3.54 kg is mixed with isopropanol and heated to 45°C under an atmospheric pressure. Dimethyl sulfate of 0.715 kg is then added dropwise into the reaction mixture. The addition is performed at a temperature between 45°C to 50°C with 77 rpm stirring rate. The reaction mixture was further heated to 60°C with mixing intensity of 540 rpm upon completion of dimethyl sulfate addition. These conditions are maintained until the amine value reached below 5 mgKOH/g sample. In order to destroy traces of unreacted dimethyl sulfate, 0.022 kg of glycine is then added to the reaction mixture, followed by stirring for another 1 hour at 60°C.

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Example 2

Examples of Application

20 The towels treated with esterquats derived from methyl ester having fatty acid compositions A, B and C as in Table 1, were evaluated based on their softening performance according to ASTM D 5237-92, AATCC EP5 and in house, and rewetting performance according to ASTM D 5237-92.

25 The softening evaluation is conducted by 20 trained panelists for the towels treated with esterquats derived from methyl ester having fatty acid compositions A, B and C as in Table 1 and the test results are shown in Table 3. The result indicates that the softening performances of the esterquats derived from methyl ester having fatty acid compositions A, B and C are comparable with the esterquats derived from palm-
30 stearin fatty acid having 66.2% palmitic and stearic and 25.8% oleic. Similarly, the

softening performances of the esterquats derived from methyl ester with fatty acid compositions A, B and C are comparable with the esterquats derived from tallow having 48.4% palmitic and stearic and 42.8% oleic.

- 5 Rewettability of the treated towels was determined by the rise level test in which the level to which the dye solution rises in mm was evaluated after 10 minutes. The test results are tabulated in Table 4.

Table 3: Softening Evaluation			
Samples	Panel Preference		Significant at 95% (RSD > 15)
Esterquats (A) vs Esterquats (PSFA)	11	9	No
Esterquats (B) vs Esterquats (PSFA)	15	5	No
Esterquats (C) vs Esterquats (PSFA)	10	10	No
Esterquats (A) vs Esterquats (Tallow)	14	6	No
Esterquats (B) vs Esterquats (Tallow)	9	11	No
Esterquats (C) vs Esterquats (Tallow)	12	8	No

Table 4: Rewettability Test	
Samples	Distance climbed by dye (mm)/10 mins
Esterquats (A)	43.7
Esterquats (B)	58.7
Esterquats (C)	59.0
Esterquats (PSFA)	49.3
Esterquats (Tallow)	75.3

CLAIMS

1. A composition of methyl esters for esterquats production, characterized by 40% to 95% of saturated fatty acid methyl esters and 5% to 60% of unsaturated fatty acid methyl esters, transesterified with triethanolamine and quaternized with dimethyl sulfate.
2. A composition according to claim 1, wherein the methyl esters are derived from vegetable oils or animal fats.
3. A composition according to claim 1 or claim 2, wherein the methyl esters are derived from palm oil, soybean oil, corn oil, cottonseed oil, peanut oil, olive oil, canola oil, sunflower oil, coconut oil, palm stearin, palm kernel oil, lard, tallow or derivatives thereof or a combination of two or more thereof.
4. A composition according to claims 1 to 3, wherein the saturated fatty acid methyl ester is palmitic acid, stearic acid or a combination thereof.
5. A composition according to claims 1 to 4, wherein the unsaturated fatty acid methyl esters is oleic acid.
6. A fabric softener comprising a composition of methyl ester according to any of the preceding claims.