

(19)



(11)

EP 1 806 392 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:

11.07.2007 Bulletin 2007/28

(51) Int Cl.:

C11D 1/62 (2006.01)**C11D 3/02** (2006.01)**C11D 3/00** (2006.01)(21) Application number: **06013688.4**(22) Date of filing: **01.07.2006**

(84) Designated Contracting States:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IS IT LI LT LU LV MC NL PL PT RO SE SI SK TR

Designated Extension States:

AL BA HR MK YU• **Gallotti, Manlio****Sao-Paulo-SP CEP 01421-001 (BR)**• **Nunes, George Italo Pitombeira****Sao-Paulo-SP CEP 04304-011 (BR)**• **Kume, Gustavo****Sao-Paulo-SP CEP 05586-000 (BR)**• **Barge, Claudia****Sao-Paulo-SP CEP 04022-002 (BR)**(30) Priority: **06.09.2005 EP 05019277**(71) Applicant: **Clariant (Brazil) S.A.****04795-900 Sao Paulo SP (BR)**(74) Representative: **Otto, Adalbert et al****Clariant Produkte****(Deutschland) GmbH****Group Intellectual Property****Am Unisys-Park 1****D-65843 Sulzbach (DE)**

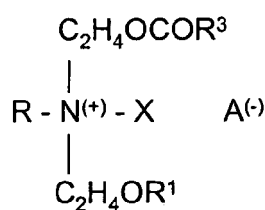
(72) Inventors:

• **Lerch, Alexander Dr.****63571 Gelnhausen (DE)**• **Scholz, Hans Juergen Dr.****63755 Alzenau (DE)**Remarks:

Amended claims in accordance with Rule 86 (2) EPC.

(54) **Stable aqueous esterquat compositions**

(57) Aqueous compositions are claimed which contain an esterquat or mixtures thereof of the formula I



(I)

wherein R is C₁-C₄-alkyl, preferably methyl, X has the same meaning as R or X is a group of the formula -C₂H₄OR², R¹ and R² are independently hydrogen or a group -COR³, R³ is C₇-C₂₁-alkyl or C₇-C₂₁-alkenyl and A is an anion, preferably chloride or methosulfate, and a metal salt, which composition is free of alkali or alkaline earth metal salt.

The addition of the metal salt, preferably zinc chloride, prevents hydrolysis of the esterquat.

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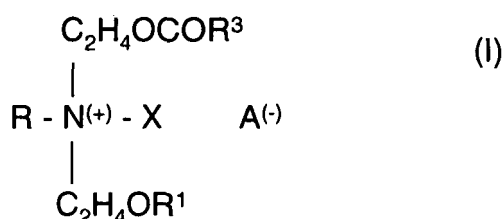
Description

[0001] Quaternized fatty acid triethanolamine (TEA) esters and quaternized fatty acid methyldiethanolamine (MDEA) esters, which are commonly called "esterquats", are widely known in detergent industry. They provide a softening effect to textile materials, especially for cotton. These compounds are made by esterification of triethanolamine (TEA) or methyldiethanolamine (MDEA) with a fatty acid and subsequent quaternisation by reacting with a short chain alkyl sulfate or alkyl chloride. In the step of esterification of TEA esterquats, one, two or three hydroxyl groups may react with the fatty acid and accordingly commercially available esterquat compositions contain quaternized mono-, di- and tri-esterquats. For the case of MDEA esterquats, only mono and di-esterquats are present.

[0002] A problem of aqueous compositions containing these Esterquats is that the Esterquats are not stable over prolonged storage since they undergo hydrolysis. In order to prevent hydrolysis it is known to add alkali metal salts (JP 10-251 972).

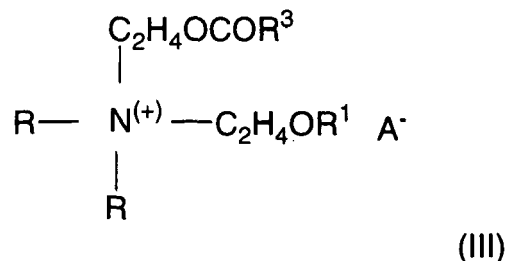
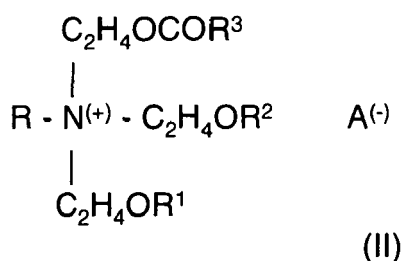
[0003] It has now been found that also other metal salts are capable of preventing hydrolysis of esterquats.

[0004] The invention accordingly provides aqueous compositions containing an esterquat or mixtures thereof of the formula I



wherein R is C₁-C₄-alkyl, preferably methyl, X has the same meaning as R or X is a group of the formula -C₂H₄OR², R¹ and R² are independently hydrogen or a group -COR³, R³ is C₇-C₂₁-alkyl or C₇-C₂₁-alkenyl and A is an anion, preferably chloride or methosulfate, and a metal salt, which composition is free of alkali or alkaline earth metal salt.

[0005] Since X in the formula (I) may be either an alkyl group R or a further hydroxyethyl group which may be esterified the claimed compositions contain either TEA esterquats or MDEA esterquats of the following formulas II and III:



[0006] The group -COR³ is preferably derived from natural occurring fatty acids such as capronic acid, caprylic acid, caprinic acid, lauric acid, myristic acid, palmitic acid, isostearic acid, stearic, oleic acid, eluidinic acid, arachinic acid, behenic acid and eruca acid. Preferred acids containing the group -COR³ are C₁₂/C₁₈ coco fatty acids, tallow fatty acid, fully or partially hydrogenated tallow fatty acid, palm fatty acid, partially or fully hydrogenated palm fatty acid or stearic acid.

[0007] As mentioned above the ester quats of formula II are present in the form of mixtures of the mono-, di- or triester of triethanolamine. Preferred mixtures are those which have 10 to 38 % by weight of the monoester (R¹ and R² being hydrogen), 46 to 63 % by weight of the diester (R¹ being hydrogen and R² -COR³) and 7 to 25 % by weight of the triester (R¹ and R² being -COR³). Especially preferred are also the following mixtures (all percentages are by weight):

Monoester: 18 - 22 %; diester 58 - 62 %; triester 18 - 22 %.

Monoester: 20 - 27 %; diester 53 - 60 %; triester 16 - 23 %.

Monoester: 29 - 37 %; diester 46 - 57 %; triester 7 - 14 %.

[0008] In the same way, the esterquats of formula III are present in the form of mixtures of mono and di-ester of methyldiethanolamine. Preferred mixtures are those which have from 1 to 70% by weight of monoester (R¹ being hydrogen), and from 30 to 99% by weight of the diester (R¹ being -COR³). Especially preferred are also the following

mixtures (all percentages by weight): Monoester 2-60%; diester 35-98%, Monoester 2-40%; diester 60-98%.

[0009] The ester quat compositions accordingly to the present invention contain in general 0.5 to 30, preferable 2 to 24 and especially 4 to 22 % by weight of the ester quats of the formula II. In order to enhance the stability of these aqueous compositions a salt is added which is not an alkali or alkaline earth metal salt. The claimed compositions are free of such salts. Preferred salts are transition metal salts, more preferably are zinc and aluminium salts such as ZnSO_4 , ZnCl_2 , AlCl_3 or $\text{Al}_2(\text{SO}_4)_3$. These salts are present in an amount preferably from 0.002 to 10.0, preferably 0.03 to 5.0, and preferably 0.04 to 3.0 % by weight.

[0010] In a preferred aspect the compositions according to the invention are also free of rare earth metal salts, metal salts of fatty acids, phthalocyanine metal complexes, phthalocyanine metal salts or chelating agents.

[0011] The compositions according to the present invention can be made by mixing the aqueous solution of the ester quats of formulas II and III final softener formulations with the salt. The salt can be added at any moment during the softeners process preparation and either in solid form or an aqueous solution. Heating and stirring is recommended for making the claimed compositions.

In order to make easier the preparation of the disclosed invention, a concentrated pre-mix containing the same molar and/or mass ratio between the ester quats and the additives can be prepared in order to be diluted up to 45 times. Other ingredients such as solvent, water or any ingredient that could be part of the final formulation may be added.

[0012] Depending on the intended use, the compositions according to the invention comprise, in addition to the mentioned compounds additives and auxiliaries which are customary and specific in each case such as for example others electrolytes, perfumes, perfume carriers, colorants, hydrotropes, antifoaming agents, other thickening agents, opacifiers, preservatives, , anti corrosion agents, co-surfactants and solvents.

[0013] In a second aspect of the invention the composition may be composed only of water, one or more esterquat of formula (I) and a metal salt, which composition is free of an alkali or alkali earth metal salt. The metal salt preferably is ZnSO_4 , ZnCl_2 , AlCl_3 or $\text{Al}_2(\text{SO}_4)_3$.

[0014] The compositions according to the invention are notable for a good storage stability and can be used as such as fabric softener or can be further diluted with water either at around room temperature or under heating .

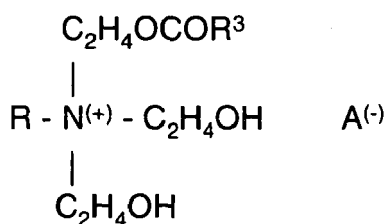
Examples

[0015] All the following compositions based on formula (II) have been prepared in the following way:

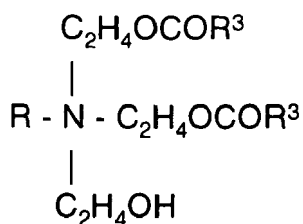
- I Water was heated to 85°C
- II addition of a varying amount of a 50 % solution of the salt as indicated and stirring for approximately 5 minutes with approximately 150 rpm
- III addition of the ester quat (5 % active content referred to the final composition)
- IV cooling under stirring within 30 minutes.

[0016] The ester quat used in all examples was a mixture of the following compounds:

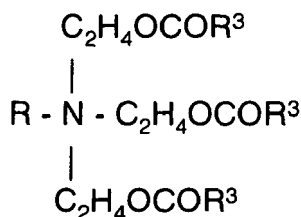
16 - 24 % of a compound of the formula



58 - 64 % of a compound of the formula



16- 24 % of a compound of the formula



Examples

[0017] For compositions based on TEA esterquats (formula II):

	ester quat	ZnCl ₂	crossing point (days)
1	5 % active matter	-	53
2	5 % active matter	0.13	63
3	5% active matter	0.61	79
	ester quat	AlCl ₃	crossing point (days)
4	5 % active matter	0.08	65
5	5 % active matter	0.40	69

[0018] For compositions based on MDEA esterquats (formula III):

- I. Water was heated to 45°C
- II. Addition of the esterquat at 50°C
- III. Cooling under stirring with approximately 150 rpm for 20 minutes
- IV. Fast cooling under stirring for 10 minutes
- V. Addition of a varying amount of a 50 % solution of the salt as indicated and stirring for approximately 5 minutes with approximately 150 rpm

	ester quat	ZnCl ₂	crossing point (days)
1	2% active matter	-	28
2	2% active matter	0.32	38

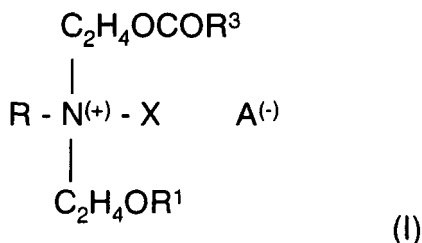
[0019] The stability profile of the formulations was monitored through storage at 45°C. The hydrolysis stability profile was analysed according to the amount of mono, di and triester analysed time to time. In the case of MDEA esterquats, it was analysed only the amount of mono and diesterquats. As hydrolysis happens the amount of monoester increases while the amount of diester decreases for both, TEA and MDEA esterquats. However, for TEA esterquats there is also a decrease on the triester amount. After a certain time the amount of monoester is higher than the amount of diester for TEA and MDEA esterquats and for TEA esterquats the triester is presented in low levels. The crossing point of the di and monoester curves was defined as the reference for comparison between the formulations. The later the crossing

point, the lower the hydrolysis rate and, consequently, the more stable the formulation.

[0020] The results given above show that the addition of ZnCl_2 or AlCl_3 shifts the crossing point further as compared with the same formulation containing no salt. This indicates the positive effect on hydrolysis stability of these salts.

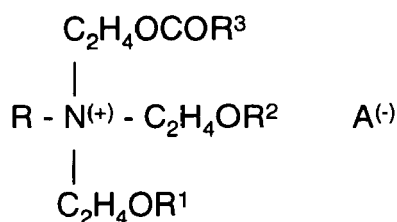
Claims

1. Aqueous compositions containing an esterquat or mixtures thereof of the formula I



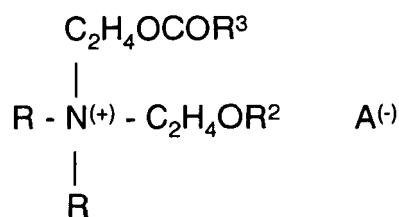
wherein R is C_1 - C_4 -alkyl, preferably methyl, X has the same meaning as R or X is a group of the formula $-\text{C}_2\text{H}_4\text{OR}^2$, R^1 and R^2 are independently hydrogen or a group $-\text{COR}^3$, R^3 is C_7 - C_{21} -alkyl or C_7 - C_{21} alkenyl and A is an anion, preferably chloride or methosulfate, and a metal salt, which composition is free of alkali or alkaline earth metal salt.

2. Aqueous compositions as claimed in claim 1 wherein the esterquat is of the formula



wherein R, R^1 , R^2 , R^3 and A are as defined in claim 1.

3. Aqueous compositions as claimed in claim 1 wherein the esterquat is of the formula



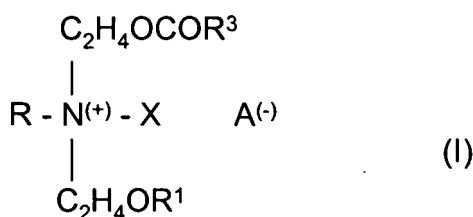
wherein R, R^1 , R^3 and A are as defined in claim 1.

4. A composition as claimed in claim 1 wherein the ester quat is of the formula II and is composed of 10 - 38 % by weight of monoester, 46 - 63 % by weight of diester and 7 - 25 % by weight of triester.
5. A composition as claimed in claim 1 wherein the ester quat is of the formula III and is composed from 1-70% by weight of the monoester and from 30-99% by weight of diester.
6. A composition as claimed in claim 1 wherein the total amount of ester quat of the formula II or of the formula III is from 0.5 to 30 % by weight.

7. A composition as claimed in claim 1 containing ZnCl_2 , AlCl_3 , ZnSO_4 or $\text{Al}_2(\text{SO}_4)_3$ as metal salt.
8. A composition as claimed in claim 1 containing 0.002 to 10 % by weight of the metal salt.
9. A composition as claimed in claim 1 containing one or more additives or auxiliaries.
10. A composition consisting of water, one or more esterquat of the formula I as defined in claim 1 and a metal salt, which composition is free of alkali or alkali earth metal salt.
11. A composition as claimed in claim 1 which is a concentrated pre-mix of the esterquat and metal salt.

Amended claims in accordance with Rule 86(2) EPC.

1. Aqueous compositions consisting of an esterquat or mixtures thereof of the formula 1



wherein R is C_1 - C_4 -alkyl, preferably methyl, X has the same meaning as R or X is a group of the formula $-\text{C}_2\text{H}_4\text{OR}^2$, R^1 and R^2 are independently hydrogen or a group $-\text{COR}^3$, R^3 is C_7 - C_{21} -alkyl or C_7 - C_{21} -alkenyl and A is an anion, preferably chloride or methosulfate, a metal salt, and optionally one or more auxiliary selected from the group consisting of electrolytes, perfumes, perfume carriers, colorants, hydrotropes, antifoaming agents, thickening agents, opacifiers, preservatives, anti corrosion agents, co-surfactants and solvents, which composition is free of alkali or alkaline earth metal salt, phthalocyanine metal complexes or phthalocyanine metal salts.



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EUROPEAN SEARCH REPORT

Application Number
EP 06 01 3688

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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 4 December 2006	Examiner Hillebrecht, Dieter
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**ANNEX TO THE EUROPEAN SEARCH REPORT
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EP 06 01 3688

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04-12-2006

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