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(54) **PEARLESCENT PREPARATIONS CONTAINING QUATERNIZED TRIETHANOLAMINE FATTY ACID ESTERS, PROCESSES FOR PREPARING THE SAME, AND METHODS OF USE THEREFOR**

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(57) **ABSTRACT**

The invention relates to pearlescent water-containing preparations which can be obtained by (a) reacting triethanolamine and C_{12-22} fatty acids in a molar ratio of 1:1.3 to 1:1.4, (b) subsequently quaternizing the esterification products in known manner and (c) dispersing the quaternization products in water.

PEARLESCENT PREPARATIONS CONTAINING QUATERNIZED TRIETHANOLAMINE FATTY ACID ESTERS, PROCESSES FOR PREPARING THE SAME, AND METHODS OF USE THEREFOR

BACKGROUND OF THE INVENTION

[0001] Quaternized fatty acid triethanolamine ester salts, which belong to the group of esterquats, are cationic surfactants which are readily taken up by synthetic and natural fibers and which provide them with substantivity and a pleasant soft feel. Accordingly, aqueous dispersions of these compounds are used as fabric softeners and as hair conditioners. They are generally produced from fatty acids which are partly esterified with triethanolamine in the presence of hypophosphorous acid and then quaternized with alkyl halides or dialkyl sulfates, preferably methyl chloride or dimethyl sulfate, in a solution of lower alcohols or polyols. A molar ratio of triethanolamine to fatty acid of 1:1.2 to 1:2.2 is generally described in the literature, a ratio of 1:1.5 to 1:1.9 being preferred for performance reasons.

[0002] From the aesthetic perspective, the market is interested not only in offering transparent formulations of these esterquats, but also—and in particular—in providing pearlescent compositions. To this end, there has been no shortage of attempts to add corresponding wax components to the dispersions, but to no real avail, because the waxes cannot be stably incorporated in the formulation for long periods.

[0003] Accordingly, the problem addressed by the present invention was to provide new water-containing preparations based on quaternized fatty acid triethanolamine ester salts which would have pearlescent properties without the addition of waxes and which would match known products in their performance properties.

SUMMARY OF THE INVENTION

[0004] The present invention relates to pearlescent water-containing preparations which can be obtained by: (a) reacting triethanolamine and C_{12-22} fatty acids in a molar ratio of 1:1.3 to 1:1.4, (b) subsequently quaternizing the esterification products in known manner and (c) dispersing the quaternization products in water.

[0005] It has surprisingly been found that esterquats which have pearlescent properties in the form of aqueous dispersions can be produced by controlling the degree of esterification and the fatty acid, so that there is no need to use typical pearlizing waxes, for example of the glycol fatty acid ester type. The invention includes the observation that the molar ratio between triethanolamine and the fatty acids selected is the critical parameter: too low a ratio would lead to low pearlescence and, in particular, to unsatisfactory performance properties whereas, if the ratio is too high, no pearlescent effect at all is observed.

[0006] The present invention also relates to a process for the production of pearlescent water-containing preparations, in which (a) triethanolamine and C_{12-22} fatty acids are reacted in a molar ratio of 1:1.3 to 1:1.4, (b) the esterification products are then quaternized in known manner and (c) the quaternization products are dispersed in water.

[0007] As a consequence of the substoichiometric use of the fatty acids, based on the dry residue, the preparations preferably contain 1 to 20% by weight, preferably 3 to 15% by weight and more particularly 5 to 10% by weight of free C_{12-22} fatty acids.

DEATILED DESCRIPTION OF THE INVENTION

[0008] Starting Materials

[0009] So far as the alkanolamine component is concerned, the choice of starting materials is limited to triethanolamine because the results obtained with methyl diethanolamine, for example, are far less satisfactory. So far as the choice of the fatty acid component is concerned, preference attaches to fatty acids corresponding to formula (I):

$$R^1COOH \quad (I)$$

[0010] in which R^1CO is a linear or branched, saturated or unsaturated acyl group containing 16 to 18 carbon atoms. The use of hydrogenated or partly hydrogenated coconut oil or tallow fatty acid and preferably palmitic, stearic and behenic acid and mixtures thereof has proved to be particularly effective.

[0011] Esterification and Guatemization

[0012] The esterquats may be produced both from fatty acids and from the corresponding triglycerides. The condensation of the alkanolamines with the fatty acids may also be carried out in the presence of defined quantities of dicarboxylic acids such as, for example, oxalic acid, malonic acid, succinic acid, maleic acid, fumaric acid, glutaric acid, adipic acid, sorbic acid, pimelic acid, azelaic acid, sebacic acid and/or dodecanedioic acid. This results in a partly oligomeric structure of the esterquats. To achieve a particular balance between pearlescence and performance, particularly softness, it has proved to be optimal to use triethanolamine and fatty acids in a molar ratio of 1:1.32 to 1:1.38. Alkyl halides or dialkyl sulfates, more particularly methyl chloride or dimethyl sulfate, are suitable for the quaternization. Both the esterification and the quaternization may be carried out in known manner. Representatives of the many prior-art publications include DE 4308794 C1 and DE 4335782 C1 (Cognis) of which the teaching is encompassed by the present patent application. More particularly, the quaternized fatty acid triethanolamine ester salts according to the invention are produced in the presence of lower aliphatic alcohols or polyols which are added to the intermediate products before the quaternization. C_{1-4} alcohols and C_{2-10} alkylene glycols, especially isopropyl alcohol or propylene glycol, are suitable for this purpose. Their production is described, for example, in DE 19738645 C1 (Cognis). The quaternization generally gives 60 to 95% by weight and preferably 75 to 85% by weight preparations of the quaternized fatty acid triethanolamine ester salts in the lower alcohols or polyols which are then diluted with water to a solids concentration of 5 to 40% by weight and preferably 10 to 30% by weight and show pearlescence even in this concentration range.

[0013] Commercial Applications

[0014] The present invention also relates to the use of the new pearlescent water-containing preparations for the pro-

duction of softeners for synthetic and/or natural fibers in which they may be present in quantities of 10 to 100% by weight, based on the final preparation. In the simplest case, the preparations themselves may be used as softeners, although they may also be further diluted with water and/or other typical auxiliaries and additives may be incorporated. This is particularly the case when the preparations are to be used as hair conditioners. So far as the additives in question are concerned, reference is again made to the above-cited DE 19738645 C1.

EXAMPLES

[0015] 596 to 943 g (2.88 to 4.56 mol) stearic acid (StA) and 1.2 to 1.8 g hypophosphoric acid (50% by weight) were introduced into a 1.5-liter three-necked flask equipped with a stirrer, dropping funnel and distillation head and heated to 70° C. 363 g (2.4 mol) triethanolamine (TEA) were added dropwise in portions under a reduced pressure of 30 mbar, the temperature being increased to 160° C. After the addition, the reaction mixture was stirred for another 2 h at 2 mbar until no more water of reaction was separated and the acid value had reached a value below 5 mg KOH/g. 400 g (0.86 mol) of the ester produced were then transferred to a second three-necked flask and dissolved in 126 g propylene glycol at 50° C. 104 g (0.83 mol) dimethyl sulfate were then added dropwise in portions and the mixture was stirred for 4 h at 65° C. A solution containing 80% by weight esterquat and 20% by weight propylene glycol, which was low in viscosity at 20° C., was obtained. Water was then added to the solution in such a quantity that a dispersion with a solids content of 15% by weight was obtained.

[0016] The pearlescent properties of the formulations were subjectively evaluated on a scale of 1 (=sparkling pearlescence) to 4 (=no effect present). To determine performance, 1 kg terry towelling was washed at 60° C. in a standard domestic washing machine of the Miele type and a measuring capful of the softener dispersions was added to the liquor. After drying, softness was determined by a panel of 5 experienced testers on a scale of 1 (=very soft) to 4 (=hard) and then averaged. The results are set out in Table 1. Examples 1 to 3 correspond to the invention, Examples C1 to C3 are intended for comparison.

TABLE 1

				Pearlesence and softness	
Example	TEA (g)	StA (g)	TEA:CFA molar ratio	Pearlescence	Softness
C1	363	596	1:1.20	2	4
1	363	645	1:1.30	1	3
2	363	671	1:1.35	1	2
3	363	695	1:1.40	1	2
C2	363	745	1:1.50	3	2
C3	363	795	1:1.60	4	1
C4	363	943	1:1.90	4	1

[0017] It can be seen that, although acceptable pearlescence is still obtained where the molar ratio is below the range according to the invention of 1:1.3 (TEA:CFA), softness is non-existent whereas, above the limit of 1:1.4 according to the invention, pearlescence is non-existent but the performance properties are very good.

[0018] It will be appreciated by those skilled in the art that changes could be made to the embodiments described above without departing from the broad inventive concept thereof. It is understood, therefore, that this invention is not limited to the particular embodiments disclosed, but it is intended to cover modifications within the spirit and scope of the present invention as defined by the appended claims.

What is claimed is:

1. A pearlescent preparation prepared by a process comprising:

(a) reacting triethanolamine and a C_{12-22} fatty acid in a molar ratio of from 1:1.3 to 1:1.4 to form an esterification product;

(b) quaternizing the esterification product; and

(c) dispersing the quaternized esterification product in water.

2. The pearlescent preparation according to claim 1, wherein the preparation comprises an amount of free C_{12-22} fatty acid of from 1 to 20% by weight based on the dry weight of the esterification product.

3. The pearlescent preparation according to claim 1, wherein the triethanolamine and the C_{12-22} fatty acid are reacted in a molar ratio of from 1:1.32 to 1:1.38.

4. The pearlescent preparation according to claim 1, wherein the C_{12-22} fatty acid comprises a fatty acid corresponding to the general formula I:

$$R^1COOH \quad (I)$$

wherein R^1CO represents a linear or branched, saturated or unsaturated acyl group having from 16 to 18 carbon atoms.

5. The pearlescent preparation according to claim 3, wherein the C_{12-22} fatty acid comprises a fatty acid corresponding to the general formula I:

$$R^1COOH \quad (I)$$

wherein R^1CO represents a linear or branched, saturated or unsaturated acyl group having from 16 to 18 carbon atoms.

6. The pearlescent preparation according to claim 2, wherein the triethanolamine and the C_{12-22} fatty acid are reacted in a molar ratio of from 1:1.32 to 1:1.38.

7. The pearlescent preparation according to claim 2, wherein the C_{12-22} fatty acid comprises a fatty acid corresponding to the general formula I:

$$R^1COOH \quad (I)$$

wherein R^1CO represents a linear or branched, saturated or unsaturated acyl group having from 16 to 18 carbon atoms.

8. The pearlescent preparation according to claim 6, wherein the C_{12-22} fatty acid comprises a fatty acid corresponding to the general formula I:

$$R^1COOH \quad (I)$$

wherein R^1CO represents a linear or branched, saturated or unsaturated acyl group having from 16 to 18 carbon atoms.

9. A process for preparing pearlescent preparations, said process comprising:

(a) reacting triethanolamine and a C_{12-22} fatty acid in a molar ratio of from 1:1.3 to 1:1.4 to form an esterification product;

(b) quaternizing the esterification product; and

(c) dispersing the quaternized esterification product in water.

10. The process according to claim 9, wherein the triethanolamine and the C_{12-22} fatty acid are reacted in a molar ratio of from 1:1.32 to 1:1.38.

11. The process according to claim 9, wherein the C_{12-22} fatty acid comprises a fatty acid corresponding to the general formula I:

$$R^1COOH \quad (I)$$

wherein R^1CO represents a linear or branched, saturated or unsaturated acyl group having from 16 to 18 carbon atoms.

12. The process according to claim 10, wherein the C_{12-22} fatty acid comprises a fatty acid corresponding to the general formula I:

$$R^1COOH \quad (I)$$

wherein R^1CO represents a linear or branched, saturated or unsaturated acyl group having from 16 to 18 carbon atoms.

13. The process according to claim 9, wherein the C_{12-22} fatty acid comprises a component selected from the group consisting of hydrogenated or partly hydrogenated coconut oil fatty acids, hydrogenated or partly hydrogenated tallow fatty acids, palmitic acid, stearic acid and mixtures thereof.

14. The process according to claim 10, wherein the C_{12-22} fatty acid comprises a component selected from the group consisting of hydrogenated or partly hydrogenated coconut oil fatty acids, hydrogenated or partly hydrogenated tallow fatty acids, palmitic acid, stearic acid and mixtures thereof.

15. The process according to claim 9, wherein the esterification product is quaternized with a reagent selected from the group consisting of alkyl halides and dialkyl sulfates.

16. The process according to claim 9, wherein the quaternization is carried out in the presence of an alcohol selected from the group consisting of lower aliphatic alcohols and polyols.

17. The process according to claim 9, wherein the quaternization is carried out in the presence of an alcohol selected from the group consisting of isopropyl alcohol and propylene glycol.

18. A method of softening a fiber, said method comprising:

(a) providing a fiber to be softened;

(b) providing a pearlescent preparation prepared by a process comprising:

(i) reacting triethanolamine and a C_{12-22} fatty acid in a molar ratio of from 1:1.3 to 1:1.4 to form an esterification product;

(ii) quaternizing the esterification product; and

(iii) dispersing the quaternized esterification product in water; and

(c) contacting the fiber with the pearlescent preparation.

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