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(54)Ingredient preventing the viscosity problem encountered in a perfumed concentrated fabric softener

Ingredient preventing viscosity problems (57)encountered in a perfumed concentrated fabric softener comprising at least an ester of the general formula

$$Y-C = 0$$
 I

,wherein R is a radical of a monohydric aliphatic alcohol or a group (CH₂)_nCOOR₁ or a group CH₂-CH(OCOR₄)-CH₂(OCOR₅) and Y is R' which is a saturated or unsaturated straight or branched C₆-C₂₄ alkyl group, a group (CH₂)_n COOR₁ or a group CH₂-C(OH) (COOR₂)-CH₂(COOR₃), wherein n is 1 to 6, and R1 -R5 are each a radical of a monohydric aliphatic alcohol.

Description

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The present invention is concerned with an ingredient preventing viscosity problems encountered in a perfumed concentrated fabric softener in liquid form. The invention is further related to a process for prevention of viscosity problems in a perfumed concentrated fabric softener, a softener produced according to the process and the use of the ingredient

Fabric softeners in liquid form to be added in the rinse step of a laundry cycle are well known. It is, since long, common practice that the fabric softeners are perfumed by fragrances incorporated in the softener composition. In general softener compositions comprise at least a dispersion of cationic surfactants in water, see, e.g. happi, March 1995, 95 - 97. Of course, also other usual components can be present, e.g. non ionic surfactants, anti-foaming agents, fluorescents, colourants, optical brightening agents and the like. For example in general a common composition of a concentrated fabric softener, which is about 3 or 4 times higher in concentration than a normal fabric softener, contains about

14 - 20% commonly used cationic surfactant,

0,75 - 1,2% fragrance,

optionally 0,5 - 1,5% commonly used nonionic surfactant,

balance to 100% water.

Here and in furtherance all percent values are weight percent values and the terms fragrance and perfume are interchangeably used within the following text.

For example the cationic surfactant can be dihardened tallow dimethyl ammonium chloride (available from Akzo as Arquad [®] 2HT), the ionic surfactant can be a tallow alcohol with 15 mole ethylene oxide (available as Genapol T [®] from Hoechst) and the fragrance can be any of the fragrances usually used in this connection, e.g. any mixture of odorant materials of natural and/or synthetic origin suitable for perfuming fabric softeners, e.g. a floral fragance (Perfume A) or a lavender fragrance (Perfume B) or a mixture of fragrances.

It now turned out that a viscosity problem has been encountered in connection with concentrated fabric softeners, especially with those of the type of the above mentioned kind. More particularly, the problem has emerged in connection with the fragrance used to perfume the concentrated liquid fabric softener. If a fragrance is present - and it is since long common practice to add perfume materials to a fabric softener - the viscosity of the concentrated liquid fabric softener becomes higher and even gelification occurs within two months of storage time, in some cases already within some days as outlined below. A product is gelified when its viscosity is 500mPa or higher. On the other hand experiments with the same basic compositions but without a fragrance revealed no upgrading in viscosity. Thus, this situation represents a serious drawback in modern concentrated fabric softener composition distribution and handling by the end user, respectively.

Hence, the object of the invention is to find an ingredient to be added to the fragrance used for perfuming a concentrated fabric softener or directly to the perfumed concentrated fabric softener in liquid form in order to avoid or at least to reduce the instability and to stabilize the viscosity, respectively, to such an extent that the softener can be handled without restrictions, i.e. the concentrated fabric softener in liquid form should be stable with respect to viscosity within a shelf life time of about two months at 37°C after the production date. It was found that the two months stability at 37°C is equivalent to about four months at room temperature (20-25°C).

Further, as a positive characteristic of the ingredient in case a lipase (see below) is contacted with the active component(s) of the ingredient added to the perfume in order to overcome the viscosity problem the active component(s) either should not be cleaved by the lipase or if the lipase is able to cleave the active component(s) the resultant reaction products should not negatively influence the smell of the perfume added to the concentrated fabric softener, i.e. the reaction products should not have a deleterious odoriferous effect after enzymatic cleavage.

It has now surprisingly been found that when adding an ingredient to the fragrance used for perfuming a concentrated fabric softener or directly to a perfumed concentrated fabric softener in liquid form comprising at least one ester of the general formula

$$Y-C \stackrel{\bigcirc}{<}_{OR} I$$

,wherein R is a radical of a monohydric aliphatic alcohol or a group $(CH_2)_nCOOR_1$ or a group CH_2 -CH(OCOR₄)- $CH_2(OCOR_5)$ and Y is R' which is a saturated or unsaturated straight or branched C_6 - C_{24} alkyl group, preferably a C_7 - C_{24} alkyl group, a group $(CH_2)_nCOOR_1$ or a group CH_2 -C(OH) ($COOR_2$)- $CH_2(COOR_3)$, wherein n is 1 to 6, and R1 - R5 are each a radical of a monohydric aliphatic alcohol, the afore mentioned instability and viscosity problem, respectively, is prevented. Preferably R' is saturated or unsaturated straight or branched C_{12} - C_{16} alkyl. Further, preferably the ester I is a diester (Ia) or a triester (Ib, Ic) of one of the general formulae:

, wherein R, R2, R3 and R', R4, R5, respectively, are identical.

Most preferably the ester I is at least one of the group consisting of propylene glycol dicaprylate/dicaprate, trioctyl citrate, dioctyl adipate, triheptanoin and octyl octanoate.

Preferably the concentrated fabric softener comprises the ester I in an amount of about 0,05 to 5%, preferably of about 0,15 to 0,5%.

If a lipase is contacted with or is part of the composition of the concentrated fabric softener preferably the ester I is at least one of the group consisting of propylene glycol dicaprylate/dicaprate, triheptanoin and trioctyl citrate, because after enzymatic cleavage the reaction products of propylene glycol dicaprylate/dicaprate and triheptanoin are non-odor-iferous and trioctyl citrate is not cleaved at all.

Propylene glycol dicaprylate/dicaprate is a mixture of the propylene glycol diesters of caprylic and capric acids. The other above mentioned specific esters conform to the following formulas.

trioctyl citrate:

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(tri-2-ethylhexyl citrate)

dioctyl adipate:

(ethyl-2-hexyl adipate)

triheptanoin:

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O CH₂ - O - C - (CH₂)₅ CH₃ O H - C - O - C - (CH₂)₅ CH₃ O CH₂ - O - C - (CH₂)₅ CH₃

(glycerol triheptanoate)

octyl octanoate:

$$\begin{array}{c} O \\ \parallel \\ CH_3 - (CH_2)_3 - CH - C - O - CH_2 - CH - (CH_2)_3 \ CH_3 \\ \mid \qquad \qquad \mid \qquad \qquad \mid \\ C_2H_5 \qquad \qquad C_2H_5 \end{array}$$

(2-ethylhexyl-2-ethylhexanoate)

Preferably the ingredient is added in an amount of up to 30%, most preferably of about 16%, of the fragrance used to perfume the concentrated fabric softener.

All afore mentioned results have been derived from tests performed at 37°C using each time a batch of 100g of a concentrated fabric softener in liquid form comprising:

15% Rewoquat WE 18,

2% Genapol T 150, (composition I)

0,75% Perfume A,

0,15% ingredient (ester I),

balance to 100% water.

Rewoquat WE 18, supplied by Rewo company, is di-(tallow carboxyethyl)hydroxy ethyl methylammonium methosulfate.

The measurement of viscosity were performed using a Hacke VT 501 viscosimeter with NV spindle at a speed of 64 rounds per minute.

As described previously the effect at 37°C is double that at room-temperature. The test temperature of 37°C is preferred because it reduces the testing time. In all further described experiments therefore the test temperature is 37°C.

Measurements of the viscosity of the fabric softener with the above composition I but containing 0,9% of Perfume A and no ingredient (composition II) compared with a fabric softener with the same composition except that the fragrance was substituted by the same amount of water (composition III) yield the following results.

Table I

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storage time at 37°C						
	production date	after 1 week	after 2 weeks	after 2 months		
composition II	15 mPa	200 mPa	>500mPa	solid		
composition III	15 mPa	15 mPa	15 mPa	15 mPa		
(1 mPa = 1cps)						

This shows that the unperfumed composition III is stable, i.e. the viscosity does not increase whereas the perfumed composition under the same storage conditions is instable and becomes a gel within two weeks (>500mPa) and even a solid after two months.

Further, tests have been made under the same conditions as above with composition II but using other fragrances. The results are presented in Table II.

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Table II

fragrance	storage time at 37°C		
	production date	after 1 week	after 2 weeks
Perfume A	15 mPa	200 mPa	>500 mPa
Perfume B	15 mPa	152 mPa	solid

The results show that some key components of the perfumes may be responsible for the viscosity increase. Therefore, to reduce the instability of the perfumed concentrated softener some components of the perfume have to be reduced and/or rebalanced by other odoriferous ingredients.

According to the invention the solution of the problem can be solved simply by adding an ingredient comprising at least one compound of formula I to the perfume used to perfume the concentrated fabric softener or to the already perfumed concentrated fabric softener.

With the knowledge of all these results a further test series was performed with numerous esters. This gave the best results leading to the ingredient as claimed. The test series was performed in the same way as above with the same concentrated fabric softener by adding 0,15% of the ingredient. Results are presented in the following Table III.

Table III

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renormance					
1 very efficient	2 efficient	3 still reasonable effi- cient			
propylene glycol dicaprylate /dicaprate	triheptanoin	isopropyl myristate			
trioctyl citrate	octyl octonoate	isopropyl palmitate			
dioctyl adipate					

The performance is divided roughly in three categories with respect to the viscosity values after 2 months, viz.

very efficient	≤ 20 mPa,	
efficient	21 ≤ 40 mPa,	
still reasonable efficient	41 ≤ 150 mPa.	

So it turned out, as already said before, that an ingredient comprising an ester of the general formula

$$Y-C = 0$$

,wherein R is a radical of a monohydric aliphatic alcohol or a group $(CH_2)_nCOOR_1$ or a group CH_2 -CH(OCOR₄)- CH_2 (OCOR₅) and Y is R' which is a saturated or unsaturated straight or branched C_6 - C_{24} alkyl group, preferably a C_7 - C_{24} alkyl group, a group $(CH_2)_nCOOR_1$ or a group CH_2 -C(OH) $(COOR_2)$ - CH_2 (COOR₃), wherein n is 1 to 6, and R1 - R5 are each a radical of a monohydric aliphatic alcohol, overcomes the afore mentioned problem. Prefered R' is a C_{12} - C_{16} alkyl.

The ingredient is also effective if it contains more than one ester, especially more than one of those mentioned in column 1 and /or 2 of Table III.

In addition, the interaction between these esters and a lipase was tested because lipases may be used in a washing cycle, especially to remove greasy stains.

The lipase works during the drying cycle, i.e. after the wet fabric from the washing cycle, in which a lipase containing powder detergent is used, has been handled in the following rinse step with the fabric softener. So, in case of the use of a fabric softener containing the ingredient as described before the lipase contained in the water remaining in the fabric, even if the fabric has been centrifuged between the washing cycle and rinse step, may attack the esters in the fabric softener, especially the ester of the fragrance. The ester(s) of the general formula I with the restrictions as specified above comprised in the ingredient preventing the viscosity problem encountered in a perfumed concentrated fabric softener might as well be attacked by the lipase to yield an alcohol. As said before this alcohol should not negatively influence the smell of the fabric softener. The esters can be cut by the lipase by the following reaction to yield an alcohol according to the general reaction:

$$R_{y}-C$$

$$O-R_{x}$$
Lipase
$$R_{x}-OH+R_{y}COOH$$
alcohol

Therefore, the interactions of a lipase with the ester(s) of the general formula I with the restrictions as specified have been tested with the help of the linitest method.

A piece of fabric has been washed during 30 minutes at a temperature of 40°C with 2g of a commercial powder detergent containing a lipase and was then rinsed with a fabric softener containing an ingredient to be tested in an amount of 0,3%. The olfactive evaluation has been done as well on the damp as an the dry fabric to evaluate the smell of the released alcohol. The results are presented in Table IV.

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Table IV

ester	released alcohol	olfactive aspect of released alcohol
dioctyl adipate	ethyl-2 hexanol	odoriferous
octyl octanoate	ethyl-2 hexanol	odoriferous
trioctyl citrate	none	
propylene glycol dicaprylate/dicaprate	propylene glycol	non odoriferous
triheptanoin	glycerol	non odoriferous

The reactions are as follows.

dioctyl adipate:

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$$\begin{array}{c|c} O & O \\ \parallel & \parallel & \\ C_8H_{17}O\text{-}C(CH_2)_4\text{-}C\text{-}OC_8H_{17} & \xrightarrow{\text{Lipase}} \\ & & C_8H_{17}OH + HOOC\text{-}(CH_2)_4COOH \\ & & \text{ethyl-2 hexanol} \end{array}$$

octyl octanoate:

The other three tested esters are not cleaved or are enzymatically cleaved but are producing non odoriferous alcohols according to the following reactions:

propylene glycol dicaprylate/dicaprate:

CH₃-(CH₂)₆-C-O-CH₂-CH-O-C-(CH₂)₆CH₃

$$CH_3$$

triheptanoin:

$$\begin{array}{c}
O \\
\parallel \\
CH_2 - O - C - (CH_2)_5 CH_3 \\
O \\
\parallel \\
H - C - O - C - (CH_2)_5 CH_3
\end{array}$$

$$\begin{array}{c}
CH_2OH \\
CHOH + 3CH_3(CH_2)_5COOH \\
CH_2 - O - C - (CH_2)_5 CH_3
\end{array}$$

$$\begin{array}{c}
CH_2OH \\
CHOH + 3CH_3(CH_2)_5COOH \\
CH_2OH
\end{array}$$

$$\begin{array}{c}
CH_2OH \\
CH_2OH
\end{array}$$

$$\begin{array}{c}
CH_2OH
\end{array}$$

$$\begin{array}{c}
CH_2OH
\end{array}$$

Due to the enzymatic cleavage dioctyl adipate and octyl octanoate are each producing ethyl-2 hexanol, an odoriferous alcohol having a green metallic smell.

Propylene glycol dicaprylate/dicaprate, trioctyl citrate and triheptanoin do not affect the strength and the character of the fabric softener when a lipase is present. Added to the fragrance of a concentrated fabric softener the olfactive behavior of these esters is neutral and olfactively acceptable for an expert panel. So, preferably R in the general formula I is a radical of a non-odoriferous alcohol of formula ROH.

A process for prevention of viscosity problems in a perfumed concentrated fabric softener is performed in that the ingredient as described before is added to said softener. The addition of the ingredient may be done during the production of the softener or after the softener has already been produced. Addition of the ingredient to the fragrance which is used to perfume the concentrated fabric softener is the prefered method. As the afore described experiments signify it is advisable to incorporate the ingredient into the complete softener soon after the production date or, what is more recommended, immediately after the production.

Claims

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1. Ingredient preventing viscosity problems encountered in a perfumed concentrated fabric softener comprising at least one ester of the general formula

$$Y-C = O$$
OR

- ,wherein R is a radical of a monohydric aliphatic alcohol or a group $(CH_2)_nCOOR_1$ or a group $CH_2-CH(OCOR_4)-CH_2(OCOR_5)$ and Y is R' which is a saturated or unsaturated straight or branched C_6-C_{24} alkyl group, preferably a C_7-C_{24} alkyl group, a group $(CH_2)_nCOOR_1$ or a group $CH_2-C(OH)$ ($COOR_2$)- CH_2 ($COOR_3$), wherein n is 1 to 6, and R1 R5 are each a radical of a monohydric aliphatic alcohol.
- 50 2. Ingredient of claim 1 characterized in that the ester is a monoester, a diester or a triester.
 - 3. Ingredient of claim 1 or 2 characterized in that the diester has the general formula

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, especially

$$\begin{array}{ccc} O & CH_3 & O \\ \parallel & \parallel & \parallel \\ CH_3\text{-}(CH_2)_6 & -\text{C-O-CH}_2\text{-CH-O-C-}(CH_2)_6\text{CH}_3 \end{array}$$

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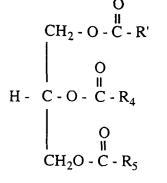
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4. Ingredient of claim 1 or 2 characterized in that the triester has the general formula

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H₂ - C - O - R

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Ic

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,wherein R, R2, R3, and R', R4, R5, respectively, are identical.

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5. Ingredient of claim 1 characterized in that the ester is at least one of the group consisting of propylene glycol dicaprylate/dicaprate, trioctyl citrate, dioctyl adipate, triheptanoin and octyl octanoate.

or

- 6. Ingredient of claim 5 characterized in that the ester is at least one of the group consisting of propylene glycol dic-40 aprylate/dicaprate, trioctyl citrate, triheptanoin.
 - 7. Ingredient of claim 6 characterized in that the ester is propylene glycol dicaprylate/dicaprate.
- 8. Process for prevention of viscosity problems in a perfumed concentrated fabric softener comprising adding the ingredient of any one of the claims 1 to 7 to said softener.
 - 9. Softener having a viscosity stability of at least 2 months storage time at 37°C and having incorporated the ingredient of any one of the claims 1 to 7.
- **10.** Use of the ingredient of any one of the claims 1 to 7 for prevention of viscosity problems in a perfumed concentrated fabric softener.