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⑦① Applicant: **S.A. Camp Fàbrica de Jabones, Fray**
Carbo, 24, Granollers Barcelona (ES)

⑦② Inventor: **Barba, Joaquim M., San Elias 11, 19,**
Barcelona 6 (ES)
Inventor: **Càliz, Joan, Puig d'en Colomer 56, 58, Banyoles**
Girona (ES)

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⑦④ Representative: **Klöpsch, Gerald, Dr.-Ing., An Gross St.**
Martin 6, D-5000 Köln 1 (DE)

⑤④ **Stable concentrated aqueous dispersions of water-insoluble cationic compounds and preparation thereof.**

⑤⑦ Fabric softening compositions, based in concentrated aqueous dispersions of water-insoluble cationic compounds, with adequate viscosity and dispersibility in water and an improved stability which time of storage and temperature variations.

The essential components are the following:

A. - 10 to 20% of water-insoluble cationic softener

B. - 0.1 to 3% of an alkoxyated tertiary amine with 20 to 100 mols of alkylene oxide per mol of amine.

C. - 0.5 to 5% of a system constituted by two non-ionic surfactants pertaining to the type of alkoxyated fatty alcohols or alkoxyated alkyl phenols, in such a way that one of them exhibits a high degree of alkoxylation and the other one a low degree of alkoxylation, with a weight ratio between them ranging from 10 : 1 to 1 : 10.

Method to obtain these said compositions.

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DR.-ING. GERALD KLOPSCH
PATENTANWALT

An Groß St. Martin 6
D 5000 KÖLN 1
Telefon: (02 21) 23 83 48
Telegramme: Marspatent
Telex-Nr.: 8 882 336
Kl/hg, 3.6.1983

S.A.CAMP, Fabrica de Jabones
Fray Carbo, 24 Granollers
Barcelona - Spain

STABLE CONCENTRATED AQUEOUS DISPERSIONS OF WATER-
INSOLUBLE CATIONIC COMPOUNDS AND PREPARATION THEREOF

Technical field

This present invention deals with concentrated aqueous
dispersions or emulsions of insoluble cationic compounds,
used as fabric softeners, with an improved stability with
time, and in regards to temperature variations in
relation to the viscosity and dispersibility in water of
5 the said dispersions. It also deals with the adequate
preparation methods for the obtention of the said
dispersions.

State of the former technique

The appearance, in the last years, of more concentrated liquid fabric softener compositions is undoubtedly due to economical motivations, and to a greater convenience to the consumer. This fact has originated the emergence of serious technical problems related with the increase in active matter, fundamentally insoluble cationic compounds, from concentrations ranging between a 3% to a 7% in the traditional diluted compositions, to proportions higher than 10% in the currently called concentrated softeners. The primary problems have to do with the physical properties of the concentrated compositions, essentially with their viscosity and dispersibility in water and with the variation, in negative sense, of the said properties with temperature and time of storage, as well as with relatively abrupt changes of temperature.

It has been known for a time, for instance in US-Patent No. 3,681,241, that the addition of small quantities of such electrolytes as salts of divalent metals causes a considerable decrease in viscosity. In German Patent No. 29 11 198 there has also been a description of the use of aluminium salts, producing an anti-gelling effect. But these solutions are only partly effective, as the quantity of electrolyte that can possibly be added has to be very small, otherwise there could be problems caused by the formation of clots and rupture of phases. Another attempt to solve this problem consists in substituting soluble cationic products instead of the insoluble cationic products, as described, for instance, in the European Patent Application No.00 406, but these compositions do not give the required softening performance, as it is necessary for the cationic compound to become bound to the surface of the fabric for the

softening effect to occur, and these compounds, because of their solubility in water, are dragged away in a great proportion during the rinsing process. The European Patent Applications Nos. 13 780 and 18 039 describe the use of non-cyclic hydrocarbons, fatty acids or their esters, and fatty alcohols as additives to decrease viscosity and to enhance the stability of concentrated softening compositions, but the effectivity of these compounds decreases noticeably with low storage temperatures, so the solution provided by them is only a partial one. The addition of non-ionic alkoxylated surfactants is known for diluted compositions, and has been applied in concentrated compositions, for instance in the German Patent No. 29 11 198, in the French Patent No. 2 482 636 and in the US-Patent No. 3,947,076, although its utilization has not been sufficiently specified as far as the types of products are concerned, nor as to the effect produced therewith, and, nevertheless, their stabilizing action on viscosity has little effectivity at high temperatures, so it also has to be considered as only a partial answer.

The utilization of alkoxylated amines in softener composition has been known now for some time, as shown in the following documents: US-Patent No. 3,850,818, German Patent Application No. 16 19 043, US-Patent No. 4,076,632, US-Patent No. 4,157,307, European Patent Application No. 21 476, US-Patent No. 4,320,013. The part played by the alkoxylated amines in the compositions described by the documents above cited are both of fabric softening and of enhancing the stability of the dispersion, but all of them are referred to diluted softener compositions, and, moreover, the stabilizing action of the alkoxylated amine has to be concurrent with the joint action of other additives, like urea or alcohols. In

European Patent Application No. 56 695 is introduced for the first time the concept of the action of alkoxyated amines as factors to achieve a proper viscosity, as well as a satisfactory stability, in concentrated softener compositions, provided that it is associated to the addition of low levels of electrolyte. Nevertheless, there is a serious limitation, as it is stated that the number of mols of oxyalkyl which conform the two alkoxyated substituents of the amine cannot ever be higher than 14 and that preferably it has to be between 2 and 8, as otherwise it should be necessary, to maintain the adequate viscosity, to significantly increase the presence of electrolyte, which would be deleterious to product stability. This limitation reduces the range of what is possible in the technique of obtention of stable concentrated softener compositions, and does not allow the utilization of alkoxyated amines with a high degree of alkoxylation which, moreover, are significantly lower in cost than those of a lower degree of alkoxylation.

Vindication of the invention

The invention herein solves these problems, as in a surprising way unexpected results are achieved in good characteristics of viscosity and a good dispersibility in water of concentrated aqueous dispersions of insoluble cationic compounds, as well as an extended stabilization thereof in ranges of temperatures between 0° and 50°C, by means of the addition of amines with a high degree of alkoxylation in union with a precise and definite system of two non-ionic components. Also, surprisingly, the dispersions described in the invention herein exhibit an excellent adaptability in the maintenance of their physical properties versus abrupt and wide changes of

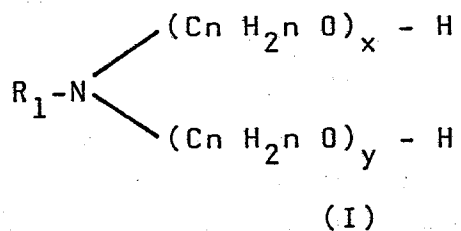
temperature. Also inherent difficulties to the process of obention of aqueous concentrated dispersions of cationic softeners are solved.

5 The fundamental object of the invention herein, then, is to achieve concentrated aqueous dispersions of insoluble cationic softeners with adequate viscosity and disper- sibility and an improved stability in storage and in abrupt temperature changes. Another aim of the invention is the achievement of more economically profitable
10 concentrated softener compositions. Another aim of the invention herein is to develop a process to obtain the compositions hereof, easy and dependable in its results.

Description of the invention

The invention herein relates to fabric softener compo-
15 sitions which are basically constituted by the following components:

1. From 10% to 20% by weight of a cationic water-insol-
uble softener compound, or of a mixture of the such.
2. From 0,1% to 3% by weight of an alkoxyated tertiary
20 amine, or of its correspondent ammonium salt, having the general formula:



wherein R_1 represents an alkyl or alkenyl group, between 10 to 20 carbon atoms, n is an integer between 1 and 4; x and y represent numbers between 10 and 50, so that the sum thereof be between 20 and 100 ($x + y = 20$ to 100).

5

3. 0,5% to 5% by weight of a mixture of two non-ionic surfactants pertaining to the groups:

10

A) C_{10} to C_{20} alkoxyated fatty alcohols or C_7 to C_{10} alkoxyated alkylphenols with a number of mols of alkylene oxide between 25 and 75 per mol of alcohol or phenol.

15

B) C_{10} to C_{20} alkoxyated fatty alcohols or C_7 to C_{10} alkoxyated alkylphenols with a number of alkylene oxide mols between 1 and 5, per mol of alcohol or phenol in such a way that the ration between A and B ranges between 10:1 and 1:10.

The above mentioned compounds are dispersed or dissolved in water with a pH ranging between 3 and 5.

20

It is highly preferred, in the composition of the invention herein, to add small quantities of an electrolyte, preferably salts of strong acids and divalent metals, the effect of which is very beneficial on the viscosity parameter. The most adequate proportions of the said electrolyte range between 0.02% and 0.15%. Below the inferior limit, the electrolyte action is very unsubstantial, and above the upper limit, there is risk that undesirable phenomena may appear, undermining the stability of the compositions, due to problems of phase disruption.

25

The insoluble cationic compounds of the invention herein belong to the type usually used in aqueous dispersions with softening activities on textile fabrics, preferably those belonging to the following groups:

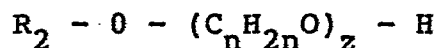
- 5 1 - Salts of quaternary ammonium with two long chain alk(en)yl groups, C₁₀ to C₂₀.
 2 - Imidazolinium quaternary salts, with two long chain alk(en)yl groups C₁₀ to C₂₀,

10 and are incorporated into the compositions in a proportion ranging between a 10% to a 20% by weight of the aforesaid composition, and in a preferred proportion of 11% to a 16% to achieve an optimal ratio of softener activity/viscosity.

15 The alkoxyated tertiary amines with a number of mols of alkylene oxide higher than 20 per mol of amine, are highly important to achieve a viscosity between 50 and 900 centipoises with a good stability, during long storage terms. In contradistinction to what was anticipated in the previous technique as expressed in the
20 European Patent No. 56 695, it has been possible to adjust the formula in a surprising way so as to make it feasible that highly alkoxyated amines perform the said function with the presence of small quantities of electrolyte in a range of 500 to 1000 ppm. Notwith-
25 standing the fact that the said amines can be used in proportions ranging from 0.1% to 3% by weight, the preferred proportions are those from 0.5% to 1.5%, with a view to achieve a good balance between the desired effect of viscosity reduction and the incidence in the final
30 cost of the composition.

The system of non-ionic surfactants herein shows itself to be effective to achieve compatibility of the alkoxy-
ated amine with a high degree of alkoxylation with the
formulation in presence of small amounts of electrolyte,
5 and it is also basic to confer on the dispersion its
qualities of elasticity versus temperature changes during
storage. These non-ionic surfactants are selected from
amongst the group of alkoxyated fatty alcohols or
alkoxyated alkylphenols with a general formula:

10 (II)



wherein R_2 represents an alk(en)yl group between C_8 and
 C_{20} or phenylalkyl from C_7 to C_{10} , n is an integer from 1
to 4, and z is a number between 1 and 75.

15 For a good execution of the invention, it is necessary to
use two non-ionic surfactants which are clearly differen-
tiated on their z value:

A) A non-ionic surfactant according to the general
formula II, in which z ranges between 25 and 75.

20 B) A non-ionic surfactant according to the general
formula II, in which z ranges between 1 and 5.

Although the proportion between A and B can have a range
between 10:1 to 1:10, the preferable proportion is
between a range of 3:1 to 1:3.

25 The process of obtention has also a great importance to
obtain good results in viscosity, dispersibility in
water, and stability. The temperature during the said
process has to be maintained with decreasing rhythm

between 60°C and 30°C, but the most important factor is the order and rate of addition of the different components. So, for example, it is convenient to add to the water, in the first place, a molten mixture of the
5 alkoxyated amine together with the system of non-ionic surfactants, and to adjust the pH by means of a strong acid between 3 and 5. The addition of the insoluble cationic compound is performed in a fractionated way, alternating with additions, also in a fractionated way, of
10 the electrolyte. Small variations in the order of addition are the cause of dispersions with a very high initial viscosity or with a bad performance versus temperature variations.

The characteristics of the dispersions so obtained are as
15 follows:

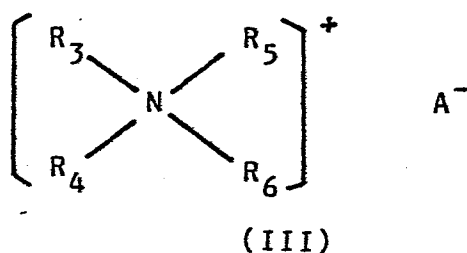
- pH = 3 to 5
- Specific Gravity = 0.95 to 1.05 g/cc
- Initial Viscosity = 50 to 400 cps
- Viscosity after 8 weeks at room temperature =
20 50 to 400 cps.
- Viscosity after 2 weeks at 0°C to 5°C:
Less than 900 cps.
- Viscosity after 2 weeks at 45°C:
Less than 900 cps.
- 25 - Dispersibility in water: Excellent,

being these characteristics very appropriated from the viewpoint of its storage and of its use as a fabric liquid softener composition.

The detailed description of the different components of
30 the invention herein are described below.

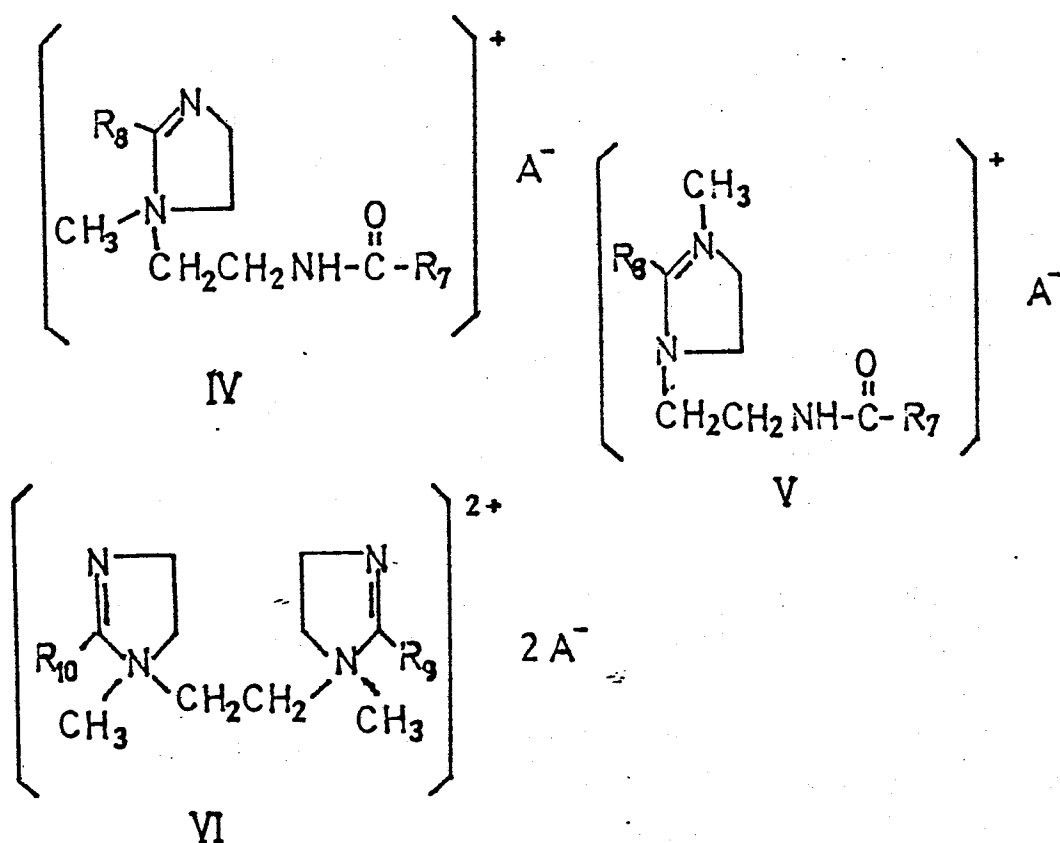
The insoluble cationic compounds are the active softening matter, and can be selected from amongst the following groups:

1. Quaternary acyclic ammonium salts represented by the
5 general formula:



wherein R_3 and R_4 represent alkyl or alkenyl groups of
10 from 10 to 20 carbon atoms, R_5 and R_6 represent short
alkyl groups from 1 to 4 carbon atoms, and A^- represents
the salt counteranion, as, for example, halides
or methyl sulphate. These are very widely known
products and, as examples, the following could be
15 quoted: ditallow dimethyl ammonium chloride; dicoco
dimethyl ammonium chloride; distearyl dimethyl
ammonium chloride; di(hydrogenated-tallow) dimethyl
ammonium methyl sulfate; di(hydrogenated tallow)
dimethyl ammonium chloride; ditallow dimethyl ammonium
20 methyl sulfate. They are easily available in the
market under different trademarks, like Adogen^R,
Varisoft^R (Sherex Chemicals), Quartamin^R (Kao Corp.),
Arquad^R (Akzo), Präpagen^R (Hoechst, etc... Of these,
di(hydrogenated tallow) dimethyl ammonium chloride is
25 preferred.

2. Quaternary imidazolinium salts represented by the
general formulae:



where R_7 , R_8 , R_9 and R_{10} represent saturated or unsaturated alkyl groups, with 10 to 20 carbon atoms, and A^- represents the salt counteranion of a monoprotic acid, such as, for example, halide or methyl sulfate. This type of products is also well known, and as examples the following are quoted:

Methyl-1-(hydrogenated tallow) amido ethyl-2-(hydrogenated tallow) imidazolinium methyl sulfate; methyl-1-tallow amido ethyl-2 tallow imidazolinium methyl sulfate; methyl-1-oleyl amido ethyl-2-oleyl imidazolinium chloride; 1-ethylene bis(2-tallow, 1-methyl imidazolinium methyl sulfate). Preferred compounds are methyl-1-(hydrogenated tallow) amido ethyl-2-(hydrogenated tallow) imidazolinium methyl sulfate, marketed by Sherex Chemical Company under the designation Varisoft^R 445, and 3-methyl-

ethyl-1-(hydrogenated tallow) amido ethyl-2-(hydrogenated tallow) imidazolinium methyl sulfate, marketed by Rewo Chemische under the designation Rewoquat^R W7500H.

5 The alkoxylated amines have the general formula I, wherein R₁ represents an alkyl group, saturated or non-saturated, from 10 to 20 carbon atoms, as those corresponding to coconut, oleic, tallow, hydrogenated tallow, and hydrogenated tallow is preferred; n is an integer between 1 and 4, value 2 being preferred, which
10 corresponds to ethoxylated amines. The values of x and y range between 10 and 50 in such a way that the total sum thereof ranges between 20 and 100, this is to say, the number of mols of ethylene oxide per amine mol must range between 20 and 100, although the preferred values of x +
15 y range between 20 and 50. This type of products is available in the market under various commercial designations as, for example, Etilenox^R (Pulcra), Ethomeen^R (Akzo), Genamin^R (Hoechst). Highly preferred are the amines of hydrogenated tallow with 20 mols of ethylene
20 oxide such as Etilenox^R KM-30 of Pulcra, S.A. and Genamin^R S-250 of Hoechst A.G., and with 50 mols of ethylene oxide as, for example, Ethomeen^R HT60 of AKZO Chemie.

Non-ionic surfactants have the general formula II,
25 wherein R₂ represents an alkyl group of between 8 and 20 carbon atoms like, for example, those corresponding to fatty alcohols like lauric, miristic, palmitic or stearic alcohols, or the mixtures thereof, or an alkyl phenyl
30 group with 7 to 10 carbons, as those corresponding to the alkyl phenols with 8 and 9 carbon atoms. The preferred groups are those corresponding to the lauric alcohol and nonylphenol. The value of n can be between 1 and 4, being the preferred value that of 2, and, therefore, the

ethoxylated products. According to the invention herein, a binary system of non-ionic oxyethylenated surfactants is necessary in function of the Z value:

5 A) A non-ionic surfactant according to general formula II and with a Z value between 25 and 75, the preferred values being between 25 and 50.

B) A non-ionic surfactant according to the general formula II with a Z value between 1 and 5, preferably between 1 and 3.

10 in a weight ratio in the range from 10:1 to 1:10, the preferred weight ratio being that of between 3:1 and 1:3. These products are well available in the market and can be found under the following commercial designations: Dobanol^R (ICI), Dowfax^R (Dow Chemical), Emulgen^R (Kao Corp.), Catinex^R (Pulcra, S.A.), etc...Especially
15 preferred is the binary system nonylphenol with 30 mols of ethylene oxide together with nonylphenol with 1.5 mols of ethylene oxide, with a weight ratio ranging from 3:1 to 1:3.

20 Besides the essential components, a series of optional additives can be incorporated to the compositions of the invention herein which are conventionally used in the field of fabric softeners, like dyes, perfumes, supplementary viscosity modifiers, emulgents, optical brighteners,
25 antioxidants, germicides, fungicides, moisteners, etc...

It is especially indicated, for a better viscosity control, the use of electrolytes, as salts of divalent metals with strong acids, in weight ratios ranging between 200 and 1.500 ppm. Especially preferred electrolytes are calcium chloride and magnesium sulfate.

Other optional additives, which are indicated as auxiliary in viscosity control, are lineal or ramified short chain alcohols from 1 to 4 carbon atoms. Isopropilic alcohols, particularly, may be had in a proportion, between a 1% and a 4%, but not in a bigger proportion that may cause lack of stability.

Optional components indicated to improve the water absorbancy of the fabrics treated with the softeners are the emulsified silicones, such as DC-347, DC-346 and DC-HV490, marketed by Dow Corning. Small amounts, of an order of a 0.01% to a 0.3% in active matter, are employed.

Optical brighteners can also be employed, like, for example, derivatives of estilbene and distirylbiphenyl, marketed by Ciba-Geigy as Tinopal^R.

It is convenient here to remark that, in what concerns the more detailed aspects of the process of obtention given by the invention herein, that the said process exhibits two very distinct phases:

- 25 A - The addition of a molten mixture of the non-ionic components and the alkoxyated amine on water, with stirring, at a temperature between 50° and 60°C, and the addition of a strong acid up to a pH between 3 and 5.

5 B - Addition of the molten cationic component in two
fractions of the 70% and 30%, respectively, of the
total amount, alternating with the addition of
electrolyte, also in two fractions corresponding
to a 30% and a 70% of the total quantity of the
same, in such a way that after the first addition,
of the 70% of the cationic compound, follows the
30% of the electrolyte, and that, following a time
of stirring and mixing, the second fraction of a
10 30% of the cationic component is added and, upon
this, the remaining 70% of electrolyte. All
this part of the process takes place with stirring
and at a temperature between 45° and 55°C.

15 Once the dispersion has taken place in the manner
hereinabove, it is left to cool to 30° to 40°C, adjusting
the pH again between 3 and 5, and the remaining optional
ingredients can now be added.

20 It is important to remark that the order of addition
hereinbefore is essential to achieve dispersions with the
desired viscosity and stability.

25 For a better understanding of the description of the
invention given herein, the following examples are given,
with the statement that they must not be construed as
restrictions to the aforesaid invention:

Example I

30 1.5 parts of etoxylated hydrogenated tallow amine with 25
mols of ethylene oxide per mol of amine are mixed, at a
temperature of 55°C, together with 1 part of etoxylated
nonylphenol with 30 mols of ethylene oxide per mol of
nonylphenol and 1 part of etoxylated nonylphenol with 1.5

mols of ethylene oxide per mol of nonylphenol. The said molten mixture is added upon 70 parts of deionized water, at a temperature of 50° to 60°C, and with strong stirring. Hydrochloric acid 1 N is added until a pH of 3 to 4 is reached. Upon this, and keeping a temperature between 50° and 55°C, and a strong stirring, 11.7 parts of molten di(hydrogenated tallow) dimethyl ammonium chloride are added, with an active matter content of a 75%, and after this 0.03 parts of calcium chloride are added, in 10% aqueous solution. The mixture is stirred for 5 minutes, and then 5.0 more parts of molten cationic component are added and 0.07 parts of calcium chloride. The dispersion is cooled to 40°C in 10 minutes, maintaining the stirring on, and 0.6 parts of DC-HV490 are added (a 35% silicone emulsion marketed by Dow Corning). It is left to cool to 30° to 35°C, and other optional ingredients are added, as desired, such as perfume, dyes, etc... and deionized water is added to make up 100 parts. The dispersion is cooled to 25°C, and pH is brought to between 3.5 and 4.5 and, after this, the product is bottled.

With the dispersion prepared as hereinbefore, the following tests and measurements are performed:

Initial Viscosity. - The dispersion is left to settle until the day following its preparation, and its viscosity is measured at 20°C with a Brookfield LV Viscosimeter. Initial viscosity for the solution thus prepared is 200 centipoises.

Stability at room temperature. - The dispersion, well packed and closed, is stored at a temperature of 25°C $\pm$ 5°C for two months, and at the end of this period a

viscosity measurement is performed at 20°C. The viscosity value observed for the dispersion thus prepared is 210 centipoises.

5 Stability at high temperatures. - A sufficient amount of sample of the dispersion is hermetically sealed in the same vessel where the viscosity value is to be determined, and it is kept in a stove at 45°C during five days. Then, the sample is removed from the stove, left for another day at room temperature, and its viscosity is
10 measured at 20°C. The sample is again hermetically sealed and it is again placed in the stove at 45°C, and it is again removed at the end of that period, it is left to stabilize during another day at room temperature and its viscosity is measured at 20°C. The values that have been
15 found are:

Viscosity after 5 days = 380 centipoises

Viscosity after 10 days = 850 centipoises.

20 Stability at low temperatures. - A sample of the dispersion is hermetically sealed in the same vessel where the viscosity is to be tested, and it is placed in a refrigerator at $3^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The sample is removed after five days, it is left for another day at room temperature and its viscosity is tested at 20°C.

25 The sample is again placed in the refrigerator, for another four days, and then it is removed, left to stabilize for one day at room temperature, and then the viscosity is tested at 20°C. The values of viscosity that have been found are:

Viscosity at 5 days = 425 centipoises

30 Viscosity at 10 days = 800 centipoises

Elasticity versus temperature. - A sample of the dispersion is taken, and placed in an appropriate vessel, it is placed in a bath provided with a temperature regulator, and the temperature is adjusted at 20°C and the viscosity is then measured. It is heated to 70°C in 30 minutes, and the viscosity is again measured. It is cooled again to 20°C in 30 minutes, and the viscosity is tested. The values found are:

Viscosity at 20°C = 200 centipoises
Viscosity at 70°C = 490 centipoises
Viscosity at 20°C = 325 centipoises

Dispersibility in water. - This test is approximate and orientative. A graduate cylinder is filled with a liter of water at 20°C $\pm$ 5°C of temperature, and with a hardness of 20°HF. 30 grams of the dispersion, which has been kept two months stored at a temperature of 20°C $\pm$ 5°C, are poured on the surface from a height of 8 cm. That part of the dispersion which results not dispersed in the water remains in a ring shape in the higher or lower part of the graduated cylinder, according to its density, and an arbitrary scale is assigned to it, from 0 to 10. 0 means total dispersion and 10 no dispersion. The volume of water to which the dispersion has not arrived is measured, so that the optimal volume is that of 0 ml, and the worst one is that of 1000 ml.

By means of these data, an approximated and arbitrary scale is made, as follows:

	Grade of Dispersion	Grade of Diffusion	Dispersibility	Conventional Sign
	8 - 10	1000 - 800	Poor	-
	6 - 7	800 - 600	Scarce	+
5	4 - 5	600 - 400	Acceptable	++
	2 - 3	400 - 200	Good	+++
	0 - 1	200 - 0	Excellent	++++

10 Applying these criteria to the dispersion obtained by the invention herein, its dispersibility in water is excellent.

Example II

Performing the same operations as described under example I, a dispersion is prepared with the following ingredients:

15	Di(hydrogenated tallow) dimethyl ammonium chloride (100%)	12 parts
	Hydrogenated tallow amine with 25 mols of Ethylene oxide	1 part
20	Nonyl phenol with 30 mols of Ethylene oxide	1 part
	Nonyl phenol with 1,5 mols of Ethylene oxide	1 part
	Calcium Chloride	0.1 parts
	Optional components and water	to 100 parts

25 The dispersion exhibits the following characteristics, determined according to the criteria established in example I:

	Initial Viscosity	130 centipoises
	Viscosity after 2 months at room temperature	130 centipoises
	Viscosity after 5 days at 45°C	250 centipoises
5	Viscosity after 10 days at 45°C	410 centipoises
	Viscosity after 5 days at 3°C $\pm$ 2	125 centipoises
	Viscosity after 10 days at 3°C $\pm$ 2	260 centipoises
10	Viscosity after the elasticity cycle 20°C - 70°C - 20°C	300 centipoises
	Dispersibility in water	++++ (Excellent)

Example III

15 Performing the same operations as described herein under
example I, a dispersion is prepared with the following
ingredients:

	Di(hydrogenated tallow) dimethyl ammonium chloride (100%)	13 parts
	hydrogenated tallow amine with 50 mols 20 of Ethylene oxide	1 part
	Lauric alcohol with 50 mols of Ethylene oxide	0.5 parts
	Lauric alcohol with 2.7 mols of Ethylene oxide	1.5 parts
25	Calcium Chloride	0.1 parts
	Optional components and water	to 100 parts

The dispersion exhibits the following characteristics,
determined according to the criteria established in
example I:

	Initial Viscosity	165 centipoises
	Viscosity after 2 months at room temperature	190 centipoises
	Viscosity after 5 days at 45°C	350 centipoises
5	Viscosity after 10 days at 45°C	650 centipoises
	Viscosity after 5 days at 3°C $\pm$ 2	350 centipoises
	Viscosity after 10 days at 3°C $\pm$ 2	780 centipoises
10	Viscosity after the elasticity cycle 20°C - 70°C - 20°C	225 centipoises
	Dispersibility in water	++++(Excellent)

Example IV

- Performing the same operation as described herein under
 15 example I, a dispersion is prepared with the following ingredients:

	Di(hydrogenated tallow) dimethyl ammonium chloride (100%)	10 parts
20	3-methyl-1-(hydrogenated tallow) amido ethyl-2-(hydrogenated tallow) imidazol- inium methyl sulfate (100%)	5 parts
	Hydrogenated tallow amine with 25 mols of Ethylene oxide	1 part
	Lauric alcohol with 50 mols of	
25	Ethylene oxide	1 part
	Lauric alcohol with 2.7 mols of Ethylene oxide	1 part
	Calcium chloride	0.08 parts
	Optional components and water	to 100 parts

The dispersion exhibits the following characteristics, determined according to the criteria established in example I:

	Initial Viscosity	230 centipoises
5	Viscosity after 2 months at room temperature	280 centipoises
	Viscosity after 5 days at 45°C	450 centipoises
	Viscosity after 10 days at 45°C	850 centipoises
	Viscosity after 5 days at 3°C $\pm$ 2	240 centipoises
10	Viscosity after 10 days at 3°C $\pm$ 2	280 centipoises
	Viscosity after the elasticity cycle 20°C - 70°C - 20°C	500 centipoises
15	Dispersibility in water	+++ (Good)

The dispersions described in the examples I to IV exhibit excellent viscosity, dispersibility and stability characteristics during storage and in extreme temperature conditions for their use as fabric concentrated softeners.

Examples V to XIV

It is the purpose in the following examples to show the importance of the amine with a high degree of alkoxylation, and of the system of non-ionic surfactants in the viscosity, dispersibility in water, and stability of the dispersions. To the end of the establishment of comparative criteria, some basic aspects, such as the contents in active insoluble cationic matter, nature and contents of the alkoxyated amine, and total contents in non-ionic surfactant, have been harmonized.

The abbreviation to design the different ingredients are as follows:

- DHTDMAC: Di(hydrogenated tallow) dimethyl ammonium chloride
- 5 DTIMS-3M: 3-methyl-1-tallow amido ethyl-2 tallow imid-
azolinium methyl sulfate
- HTA 25: Hydrogenated tallow amine with 25 mols of
Ethylene oxide
- NP 30: Nonyl phenol with 30 mols of Ethylene oxide
- 10 NP 14: Nonyl phenol with 14 mols of Ethylene oxide
- NP 9: Nonyl phenol with 9 mols of Ethylene oxide
- NP 1,5: Nonyl phenol with 1.5 mols of Ethylene oxide
- LA 50: Lauric alcohol with 50 mols of Ethylene
oxide
- 15 LA 2,7: Lauric alcohol with 2.7 mols of Ethylene
oxide
- Tween^R60: Polyoxyethylene sorbitane monostearate (ICI)
- GLYC (OP-OE): Glycerine with 8 mols of Ethylene
oxide and 28 mols of propylene oxide
- 20 Plurafac^R40: Polyalkoxylated alcohol, from 13 to 15
carbon atoms, marketed by Uguine Kuhlmann

In Table 1 are shown the different compositions prepared following the basic ideas as described in example I, and in Table 2 their characteristics are detailed according
25 to the criteria given in example I.

On checking the data in Tables 1 and 2, it can be inferred that the addition of the highly alkoxylated amine causes an improvement in the stability of the compositions at room temperature and an improvement in
30 the dispersibility of the compositions in water. The incorporation of non-ionics improves the stability at low temperatures, but it is not effective at high tempera-

tures. All dispersions with a system of non-ionics as described in the invention herein cause an improvement of stability both at low and at high temperatures, and cause the viscosity value at room temperature to stay practically unchangeable, and give an excellent dispersibility in water.

TABLE 1

V VI VII VIII IX X XI XII XIII XIV

DHDMAC	12	12	12	12	12	12	12	12	12	12	12	12	-
DTMS-3M	-	-	-	-	-	-	-	-	-	-	12		
HTA 25	-	-	1	1	1	1	1	1	1	1	1	1	1
NP 30	-	-	-	2	1	-	-	-	-	-	-	1	-
NP 14	-	-	2	-	-	-	-	-	-	-	-	-	25
NP 1,5	-	-	-	-	-	-	1	1	-	-	-	1	-
IA 50	1	-	-	-	1	-	-	1	-	1	1	-	1
Tween 60	-	1	-	-	-	-	-	-	-	-	-	-	-
GLYC (OE+OP)	1	-	-	-	-	-	-	-	-	-	-	-	-
Plurafac 40	-	1	-	-	-	-	-	-	-	-	-	-	-
Cl ₂ Ca	0,1	0,07	0,05	0,05	0,03	0,1	0,05	0,05	0,08	0,1	0,08	0,1	0,05

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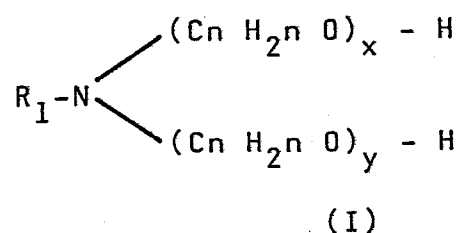
TABLE 2

	V	VI	VII	VIII	IX	X	XI	XII	XIII	XIV
Initial viscosity	320	120	155	150	410	210	170	100	160	230
2 months at 20°C ^{±5}	>1.000	650	265	220	450	450	210	120	140	350
5 days at 45°C	>10.000	>10.000	>10.000	535	>10.000	>10.000	380	300	250	435
10 days at 45°C	>10.000	>10.000	>10.000	3.300	>10.000	>10.000	590	550	410	810
5 days at 3°C ^{±2}	400	700	300	155	375	540	720	260	400	335
10 day at 3°C ^{±2}	700	>1.000	600	430	400	880	1.000	450	500	400
Elasticity 20°C-70°C-20°C	>10.000	330	375	215	715	>10.000	175	325	295	420
Dispersibility	-	+	++	+++	+	+	+++	+++	+++	+

CLAIMS

1. Fabric concentrated softener compositions with a good viscosity and dispersibility in water and an improved stability versus a long period of storage, and versus temperature changes, its essential components being as follows:

- A. 10 to 20% by weight of a water-insoluble cationic fabric softener compound, or a mixture thereof.
- B. 0.1 to 3% by weight of a tertiary alkoxyated amine or its corresponding ammonium salt, with a general formula as follows:



wherein R_1 represents an alkyl or alkenyl group, with 10 to 20 carbon atoms, n is an integer from 1 to 4, and x and y represent numbers between 10 and 50, being the total sum of both between 20 and 100. ($x + y = 20$ to 100)

C. 0.5 to 5 % by weight of a mixture of two non-ionic surfactants belonging to the following groups:

5 i.- C₈ to C₂₀ alkoxyated fatty alcohols or C₇ to C₁₀ alkoxyated alkyl phenols, with a number of mols of alkylene oxide per mol of alcohol or phenol ranging between 25 and 75.

ii.- C₈ to C₂₀ alkoxyated fatty alcohols or C₇ to C₁₀ alkoxyated alkyl phenols with a number of mols of alkylene oxide ranging between 1 and 5,

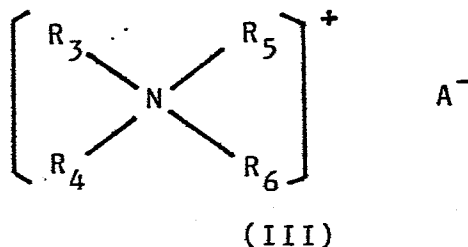
10 in such a way that the weight proportion of the said mixture of non-ionic surfactants is in the range from 10:1 to 1:10.

The said ingredients dissolved or dispersed in water at a pH ranging between 3 and 5.

15 2. Compositions according to claim 1 characterized in that the water-insoluble cationic component or components are selected from amongst the following groups:

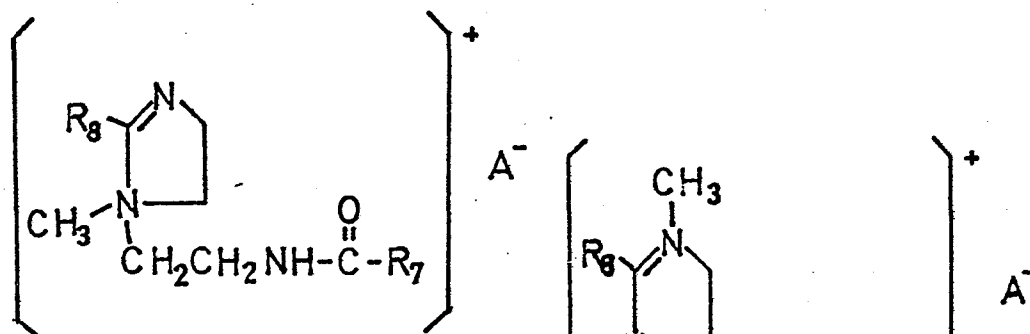
A.-Non-cyclical quaternary ammonium salts having the general formula

20

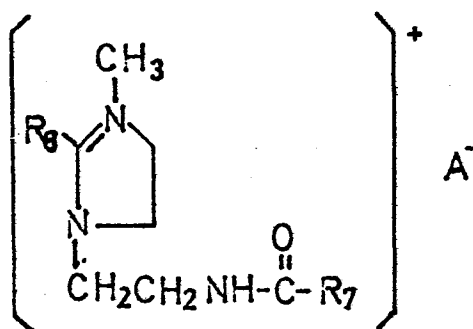


wherein R_3 and R_4 represent C_{10} to C_{20} alkyl or alkenyl groups, R_5 and R_6 represent short chain C_1 to C_4 alkyl groups and A^- represents the salt counteranion from a strong acid

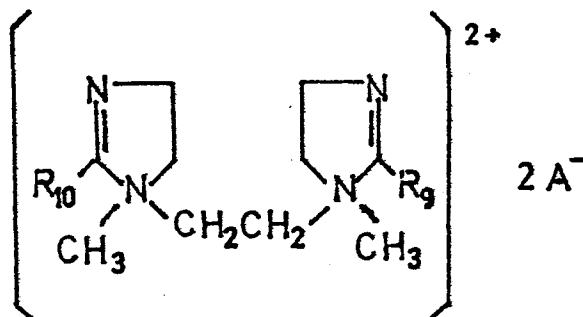
- 5 B.-Quaternary imidazolinium salts having the following general formulae:



IV



V



VI

where R_7 , R_8 , R_9 and R_{10} represent saturated or unsaturated alkyl groups, with chains from 10 to 20 carbon atoms, and A^- represents the counteranion of a strong acid.

3. Compositions according to claims 1 and 2, wherein the water-insoluble cationic component or components are: Di(hydrogenated tallow) dimethyl ammonium chloride and/or 3-methyl-1-(hydrogenated tallow) amido ethyl-2-(hydrogenated tallow) imidazolinium methyl sulfate.
4. Compositions according to claims 1 to 3, characterized in that the tertiary alkoxyated amine is: Ethoxylated (hydrogenated tallow) amine with 25 mols of ethylene oxide or ethoxylated (hydrogenated tallow) amine with 50 mols of ethylene oxide.
5. Compositions according to claims 1 to 4, characterized in that the highly alkoxyated non-ionic surfactant component is selected from amongst ethoxylated nonyl phenol with 30 mols of ethylene oxide or ethoxylated lauric alcohol with 50 mols of ethylene oxide and the non-ionic surfactant with a low level of alkoxylation is selected from amongst ethoxylated nonyl phenol with 1.5 mols of ethylene oxide or ethoxylated lauric alcohol with 2.7 mols of ethylene oxide.
6. Compositions according to any preceding claim, characterized in that an electrolyte is added to them as an agent to improve the value of viscosity, belonging to the salts of divalent metals with strong acids, with a weight ratio ranging between 0.02% and 0.15% of the total weight of the composition.
7. Compositions according to any preceding claim, characterized by having the following components:
- A.- 12 to 16% by weight of a water-insoluble cationic softener compound or a mixture thereof.

B.- 0.5 to 1.5% by weight of an alkoxyated tertiary amine having the general formula I, with 20 to 100 mols of alkylene oxide per mol of amine.

5 C.- 1 to 3% by weight of a mixture of two non-ionic surfactants pertaining to the following groups:

i.- C_8 to C_{20} alkoxyated fatty alcohols or C_7 to C_{10} alkoxyated alkyl phenols with a number of mols of alkylene oxide ranging from 25 to 75 per mol of alcohol or phenol.

10 ii.- C_8 to C_{20} alkoxyated fatty alcohols or C_7 to C_{10} alkoxyated alkyl phenols with a number of alkylene oxide mols ranging from 1 to 5 per mol of alcohol or phenol

15 wherein the weight ratio between the two non-ionic surfactants is in the range from 3:1 to 1:3.

D.-0.03 to 0.15% by weight of an electrolyte, belonging to the type of salts of divalent metals with strong acids.

20 The said ingredients dissolved or dispersed in water at a pH ranging between 3.5 and 4.5.

8. A method of making the compositions according to any preceding claim, characterized in that in a first stage a molten mixture of the non-ionic components is added with stirring together with the alkoxyated amine on deionized
25 water, in a second stage a fractioned addition of the water-insoluble cationic compound is effected, alternating with fractioned additions of electrolyte, all this

with stirring, and the preparation of the composition being finished with the addition of optional ingredients.

9. A method according to claim 8, characterized in that it takes place in a range of temperatures between 60°C and 30°C.

10. A method according to claims 8 and 9, characterized by the addition of the water-insoluble cationic compound in two fractions, the first of them being of a 70% of the total amount and the second to the 30% of the said total amount, alternating with the addition of the electrolyte also in two fractions, the first being of a 30% of the total amount, and the second of a 70% of the total amount.



DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
D,X	EP-A-0 056 695 (PROCTER & GAMBLE CO.) * Claims 1-9 * ---	1	C 11 D 3/00 C 11 D 3/30 C 11 D 1/835 C 11 D 1/62 D 06 M 13/46
D,A	US-A-4 157 307 (M.S. JAEGER et al.) * Claims 1, 2, 5 * ---		
A	US-A-4 233 164 (J.E. DAVIS) * Claim 1 * ---		
A	EP-A-0 063 329 (E.I. DU PONT DE NEMOURS & CO.) * Claim 1 * ---		
A	EP-A-0 074 056 (HOECHST AG) * Claim 1 * ---		TECHNICAL FIELDS SEARCHED (Int. Cl. ³)
A	BE-A- 888 535 (LESIEUR-COTELLE & ASSOCIES S.A.) * Claims 1-3, 7 * -----		C 11 D 1/00 C 11 D 3/00 D 06 M 13/00
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 01-02-1984	Examiner SCHULTZE D
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	