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**D1P**  
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(54) **Method of preparing fabric softening compositions**

(57) A process for preparing an aqueous liquid fabric softening composition, containing (A) a fatty acid monoalkanolamide, such as tallow monoethanolamide, and (B) a cationic fabric softener, comprises:

- (i) treating the fatty acid monoalkanolamide by contact with water;
- (ii) contacting the treated fatty acid monoalkanolamide with the cationic fabric softener and heating to give a molten mixture;
- (iii) adding the molten mixture to water and mixing to form a dispersion of the molten mixture in droplet form in the water.

The processing temperature for products prepared according to this process is reduced. Furthermore, products prepared by this process have improved viscosity and storage stability.

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METHOD FOR PREPARING FABRIC CONDITIONING COMPOSITIONS

This invention relates to a process for preparing fabric conditioning compositions, in particular aqueous liquid fabric conditioning compositions containing a cationic fabric softener and a fatty acid alkanolamide.

Aqueous fabric conditioning compositions which contain a water-insoluble cationic fabric softener together with a fatty acid alkanolamide are disclosed in British Patent GB 1 104 441 (Unilever case C.149).

One of the problems associated with the processing of such formulations is the high melting point of the fatty acid alkanolamide. We have now discovered that this problem can be overcome if the fatty acid alkanolamide is treated before it is contacted with the cationic material.

We have also surprisingly found, that where the fabric conditioning compositions are prepared using the treated fatty acid alkanolamide the product has improved viscosity and low temperature storage stability when  
5 compared with compositions prepared using the untreated raw material.

Thus, according to the invention there is provided a process for preparing aqueous liquid fabric conditioning  
10 compositions containing a cationic fabric softener material and a fatty acid monoalkanolamide, derived from a fatty acid having at least 14 carbon atoms, the process comprising the steps of:

15 i ) treating the fatty acid monoalkanolamide by contact with water at a temperature above the melting point of the fatty acid mono alkanolamide;

20 ii ) contacting the treated fatty acid monoalkanolamide with the cationic fabric softener at a temperature above the melting point of the cationic fabric softener thereby to form a molten mixture thereof;

25 iii) adding the molten mixture to water, at an elevated temperature; and mixing to form a dispersion of the molten mixture in droplet form in the water.

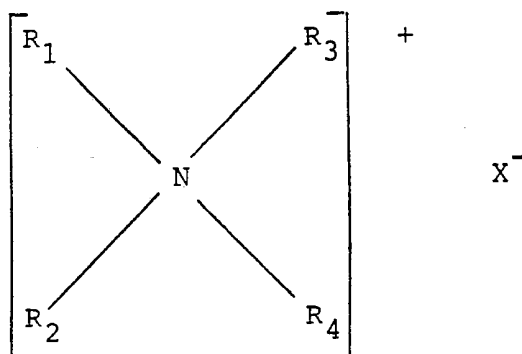
One of the advantages of the process according to the  
30 invention is that the melting point of the fatty acid monoalkanolamide is lowered after it has been treated. Thus, the energy input required for the manufacture of rinse conditioning compositions containing fatty acid monoalkanolamides can be reduced. In addition, the  
35 danger of blockages caused by the fatty acid

monoalkanolamide solidifying during processing in, for example, valves is reduced.

The cationic fabric softener is a water-insoluble cationic fabric softener. The water-insoluble cationic fabric softener can be any fabric-substantive cationic compound which has a solubility in water at pH 2.5 and 20°C of less than 10 g/l. Highly preferred materials are quaternary ammonium salts having two C<sub>12</sub>-C<sub>24</sub> alkyl or alkenyl chains, optionally substituted or interrupted by functional groups such as -OH, -O-, -CONH, -COO-, etc.

Well known species of substantially water-insoluble quaternary ammonium compounds have the formula

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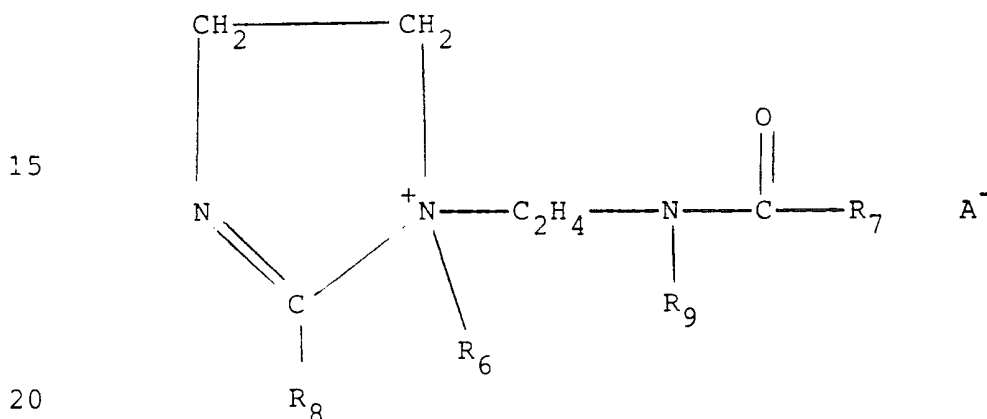
25 wherein R<sub>1</sub> and R<sub>2</sub> represent hydrocarbyl groups containing from about 12 to about 24 carbon atoms; R<sub>3</sub> and R<sub>4</sub> represent hydrocarbyl groups containing from 1 to about 4 carbon atoms; and X is an anion, preferably selected from halide, methyl sulfate and ethyl sulfate radicals.

30 Representative examples of these quaternary softeners include ditallow dimethyl ammonium chloride; ditallow dimethyl ammonium methyl sulfate; dihexadecyl dimethyl ammonium chloride; di(hydrogenated tallow alkyl) dimethyl ammonium chloride; dioctadecyl dimethyl ammonium chloride; dieicosyl dimethyl ammonium chloride; didocosyl dimethyl ammonium chloride; di(hydrogenated tallow) dimethyl ammonium methyl sulfate;

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dihexadecyl diethyl ammonium chloride; di(coconut alkyl) dimethyl ammonium chloride. Ditalow dimethyl ammonium chloride, di(hydrogenated tallow alkyl) dimethyl ammonium chloride, di(coconut alkyl) dimethyl ammonium chloride and  
5 di(coconut alkyl) dimethyl ammonium methosulfate are preferred.

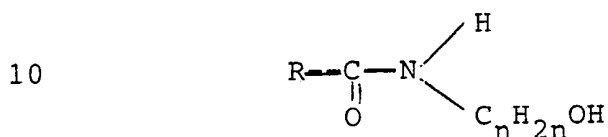
Another class of preferred water-insoluble cationic materials are the alkyimidazolinium salts believed to  
10 have the formula:



wherein  $R_6$  is an alkyl or hydroxyalkyl group containing from 1 to 4, preferably 1 or 2 carbon atoms,  $R_7$  is an alkyl or alkenyl group containing from 8 to 25 carbon  
25 atoms,  $R_8$  is an alkyl or alkenyl group containing from 8 to 25 carbon atoms, and  $R_9$  is hydrogen or an alkyl group containing from 1 to 4 carbon atoms and  $A^-$  is an anion, preferably a halide, methosulfate or ethosulfate. Preferred imidazolinium salts include 1-methyl-1-  
30 (tallowylamido-) ethyl -2-tallowyl- 4,5-dihydro imidazolinium methosulfate and 1-methyl-1- (palmitoylamido)ethyl -2-octadecyl-4,5- dihydro- imidazolinium chloride. Other useful imidazolinium materials are 2-heptadecyl-1-methyl-1- (2-stearylamido)-  
35 ethyl-imidazolinium chloride and 2-lauryl-1-hydroxyethyl- 1-oleyl-imidazolinium chloride. Also suitable herein are

the imidazolinium fabric softening components of US Patent No 4 127 489, incorporated by reference.

5 The fatty acid monoalkanolamides useful in the present invention are those derived from fatty acid amides whose alkyl radical contains at least 14 carbon atoms. They preferably have the general formula -



wherein n is 2 or 3. A suitable preferred material is tallow monoethanolamide.

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The weight ratio of the cationic softener to the fatty acid monoalkanolamide in the compositions prepared according to the invention may lie from about 1:1 to about 10:1, preferably from about 1.5:1 to about 5:1.

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The first step in the process according to the invention is the treatment of the fatty acid monoalkanolamide by contact with water.

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In one embodiment the fatty acid monoalkanolamide is melted in the presence of water.

30 In another embodiment the fatty acid monoalkanolamide can be melted and subsequently poured into hot water. The water is at a temperature above which the fatty acid monoalkanolamide is molten, such as above 75°C.

35 In this first step of the process the ratio of the fatty acid monoalkanolamide to water is preferably less than 10:1.

The exact nature of the treated fatty acid monoalkanolamide is not at present fully understood. Without being bound by theory, it is thought that it may comprise a hydrate or some other form of complex with the  
5 water.

In the presence of excess water a lipophilic phase containing the treated fatty acid monoalkanolamide and an aqueous phase are formed. The aqueous phase may be  
10 separated from this mixture.

The next step in the process according to the invention is the formation of a molten mixture of the cationic softener and the treated fatty acid  
15 monoalkanolamide, at a temperature above the melting point of the cationic fabric softener, such as above 70°C. When this molten mixture is added to water at an elevated temperature, ie a temperature above room temperature such as above 40°C, preferably above 60°C, and thoroughly  
20 mixed, a dispersion of the active materials in droplet form is created.

The compositions prepared according to the invention can also contain lanolin or a lanolin-like material as  
25 defined by GB Patent No. 2 114 619 (Unilever case C.1324) incorporated herein by reference. When the compositions contain lanolin this is added with the molten mixture containing the cationic softener and the processed fatty acid monoalkanolamide to the water at an elevated  
30 temperature. The weight ratio of the cationic softener and fatty acid monoalkanolamide to the lanolin or lanolin-like material may lie from about 1:1 to about 20:1, preferably from about 5:1 to about 15:1.

35 The compositions prepared according to the invention can also include electrolytes. The preferred level of

electrolyte in the compositions is from about 0.01% to about 0.5%, most preferably from about 0.02% to about 0.2%, measured as the anhydrous salt. Examples of suitable materials include the ionic salts of sodium, potassium, lithium, magnesium, calcium or aluminium, such as lithium chloride, sodium chloride, sodium methosulphate, sodium benzoate, calcium chloride, magnesium chloride or aluminium chloride. Of these, sodium and potassium salts are less preferred. When the composition contains electrolyte it may be added after the dispersion of the active material in droplet form has been created. The electrolyte is preferably added in the form of a concentrated solution, such as 1-10% by weight.

The compositions prepared according to the invention can also contain one or more optional ingredients selected from non-aqueous solvents such as C<sub>1</sub>-C<sub>4</sub> alkanols and polyhydric alcohols, pH buffering agents such as weak acids eg phosphoric, benzoic or citric acids (the pH of the compositions is preferably less than 6.0), antigelling agents, perfumes, perfume carriers, fluorescers, colourants, hydrotropes, antifoaming agents, antiredeposition agents, enzymes, optical brightening agents, opacifiers, stabilisers such as guar gum and polyethylene glycol, anti-shrinking agents, anti-wrinkle agents, fabric crisping agents, spotting agents, soil-release agents, germicides, fungicides, anti-oxidants, anti-corrosion agents, preservatives, dyes, bleaches and bleach precursors, drape imparting agents and antistatic agents.

These optional ingredients can be added to the water before forming the dispersion of the active material i.e. the cationic fabric softener and the fatty acid monoalkanolamide, or after adding the electrolyte, as appropriate.



The invention will now be illustrated by the following non-limiting examples, in which percentages are by weight based on the weight of the end product.

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EXAMPLES

Fabric conditioning compositions according to the formulations listed below were prepared by the following method.

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100g of a commercially available tallow mono ethanolamide (TMEA) (Priamid 1710) was melted at 80°C. The molten material was then poured onto a similar volume of hot water. The mixture was allowed to cool and the upper paste (comprising TMEA and approximately 10% water) was scraped off. This treated TMEA was used in the preparation of composition A.

Composition A was prepared by melting a mixture of the treated TMEA, Arquad 2HT (a commercially available form of dihardened tallow dimethyl ammonium chloride containing 76% of active material) and optionally lanolin at 60°C. The molten mixture was added to 80g of hot water (maintained at 65-75°C in a water bath). After thorough mixing with a high speed constant torque stirrer, a dispersion formed. Calcium chloride was added to the dispersion in the form of a 10% by weight solution. After further mixing the dispersion was allowed to cool and perfume was added.

30

By way of comparison composition B was prepared by melting a mixture of untreated TMEA, Arquad 2HT, and optionally lanolin. The molten mixture was added to 80g of hot water (maintained at a temperature above 80°C in a water bath. A higher temperature was necessary to ensure complete mixing of the molten components). The mixture

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was mixed as above and a dispersion was formed. Thereafter calcium chloride was added. After further mixing the dispersion was allowed to cool and perfume was added.

5

The products were stored at 0°C. At specific time intervals, indicated below, the viscosity of each product was assessed visually. The following linear scale was used.

10

<u>Viscosity number</u>	<u>State of composition</u>
0	liquid resembles water
2	pourable liquid
4	viscous liquid, but just pourable
6	solid

15

Example 1

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Using the process described above composition A was prepared with 5.56g of treated TMEA, (this includes about 10% by weight of water), 13.15g Arquad 2HT, 0.50g of the calcium chloride solution and 0.75% perfume.

25

Composition B was prepared with 5.00g of untreated TMEA, 13.15g Arquad 2HT, 0.50g of the calcium chloride solution and 0.75% perfume.

30

The viscosity of compositions A and B were assessed and the following results obtained.

	<u>Time/Weeks</u>	<u>Viscosity Number</u>	
		<u>A</u>	<u>B</u>
	0	½	1
35	1	½	2
	7	½	4

These results demonstrate that the product viscosity and low temperature storage stability is improved when the composition is prepared using the treated tallow monoethanolamide (composition A).

Example 2

Using the process described above composition A was prepared with 4.44g of treated TMEA (this includes about 10% by weight of water), 13.15g Arquad 2HT, 1.00g lanolin (Coronet ex Croda), and 0.50g of the calcium chloride solution and 0.75% perfume.

Composition B was prepared with 4.00g of TMEA, 13.15g Arquad 2HT, 1.00g lanolin, 0.50g of a calcium chloride solution and 0.75% perfume.

The viscosity of compositions A and B were assessed and the following results obtained.

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<u>Time/Weeks</u>	<u>Viscosity Number</u>	
	<u>A</u>	<u>B</u>
0	$\frac{1}{2}$	1
5	$\frac{1}{2}$	6

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These results also demonstrate that the product viscosity and low temperature storage stability is improved is when the composition is prepared using the treated tallow monoethanolamide (composition A).

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CLAIMS

1. A process for preparing aqueous liquid fabric conditioning compositions containing a cationic fabric softener material and a fatty acid monoalkanolamide, derived from a fatty acid having from at least 14 carbon atoms, the process comprising the steps of:

i ) treating the fatty acid monoalkanolamide by contact with water at a temperature above the melting point of the fatty acid monoalkanolamide;

ii ) contacting the treated fatty acid monoalkanolamide with the cationic fabric softener at a temperature above the melting point of the cationic fabric softener thereby to form a molten mixture thereof;

iii) adding the molten mixture to water, at an elevated temperature; and mixing to form a dispersion of the molten mixture in droplet form in water.

2. A process according to Claim 1 wherein the method of treating the fatty acid monoalkanolamide is selected from

i ) melting the fatty acid monoalkanolamide in the presence of water; and

ii ) pouring molten fatty acid monoalkanolamide into hot water.

3. A process according to Claim 2 wherein the ratio of the fatty acid monoalkanolamide to the water in step (i) is less than 10:1.

4. A process according to Claim 1 wherein the weight ratio of the cationic fabric softener to the fatty acid monoalkanolamide in the composition is within the range from 1:1 to 10:1.

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5. A process according to Claim 1 wherein an electrolyte is added in step (iii).

6. A process as claimed in Claim 1, carried out  
10 substantially as described in any one of the Examples herein.