

(9)



Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number:

**0 274 142
A2**

(2)

EUROPEAN PATENT APPLICATION

(21) Application number: **87202157.1**

(51) Int. Cl. 4: **C11D 1/52 , C11D 3/32**

(22) Date of filing: **06.11.87**

The title of the invention has been amended
(Guidelines for Examination in the EPO, A-III,
7.3).

(30) Priority: **13.11.86 GB 8627181**

(43) Date of publication of application:
13.07.88 Bulletin 88/28

(84) Designated Contracting States:
AT BE CH DE FR GB GR IT LI LU NL SE

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(54) **Detergent composition containing a polyamide softening agent.**

(57) Detergent compositions capable of providing through-the-wash softness and other fabric-care benefits are disclosed, which contain specific polyamide softening agents.

EP 0 274 142 A2

SOFTENING DETERGENT COMPOSITIONS CONTAINING SPECIFIC POLY-AMIDE SOFTENING AGENT

Technical Field

5 The present invention relates to softening detergent compositions containing a narrowly-defined poly-amide softening agent.

The compositions herein, in addition to providing good cleaning performance, exhibit excellence through-the-wash softening properties, and provide additional fabric-care benefits such as anti-static, anti-wrinkling, ease-of-ironing, color fidelity and anti-felting of wool.

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Background of the invention

Formulators of fabric treatment compositions have long sought means for simultaneously washing and softening fabrics. Among the various approaches suggested are methods employing clay softeners, or
15 amine materials, or both ingredients in combination, such as described in e.g.: German Patents 29.64.114, 28.57.16, 24.39.541, 23.34.899 and European Patents 0 026 528 and 0 028 432.

Amines have been used in combination with soaps (U.K. patent 1 514 276) fatty acids (published E.P.E. 0 133 804) or phosphate esters (published E.P.A. 0 168 889) as through-the-wash softeners.

20 The formulation of the art through-the-wash softening detergent compositions can, however, be subjected to flexibility problems which are often due to incompatibility between certain ingredients (between e.g. peroxyacid bleaching agents and softening agents).

Carboxy-amides have been used as antistatic agents in industrial textile treatment (German patent 30 43 618) and N-alkyl isostearamides as antistatic agents in laundry application (French patent 2,531,447). Japanese patent J5 8144-175-A discloses the industrial treatment of textile by cationic softening agents and
25 ethoxylated fatty acid amides.

Alkoxylated fatty amides are known as surfactants (EP 0 000 595) and as viscosity control agents (EP 0 112 719).

German Patent Application 19 59 007 discloses the use as softening agent of a monoethanolamide. German Patent Application 33 10 417 discloses the use of fatty acids diethanolamides as antistatic/non-
30 yellowing agents.

None of the above documents discloses the use of the specific amides of the present invention in softening detergent compositions.

It is an object of the present invention to provide detergent compositions capable of providing excellent cleaning, softening, and fabric-care properties.

35 It is a further object of the invention to provide softening detergent compositions which can be formulated with increased flexibility, in particular in presence of peroxyacid bleaching compounds.

Indeed, it has now been discovered that the use of a narrowly-defined class of amides as softening agents in detergent compositions provides excellent results in both cleaning and softening of fabric, as well as other "fabric care" benefits, possibly in the presence of peroxyacid bleaching agents.

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Summary of the invention

The present invention relates to detergent compositions, capable of giving through-the-wash fabric-care
45 benefits inclusive of softness, containing deterative surfactants, a nitrogen-containing fabric softening agent and, if desired deterative additives, are characterized in that the nitrogen-containing softening agent is selected from the group of :

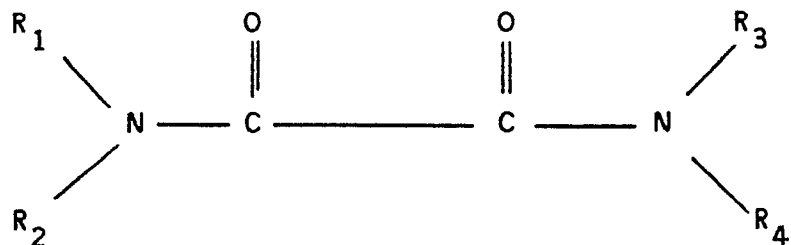
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i) diamides of the formula :

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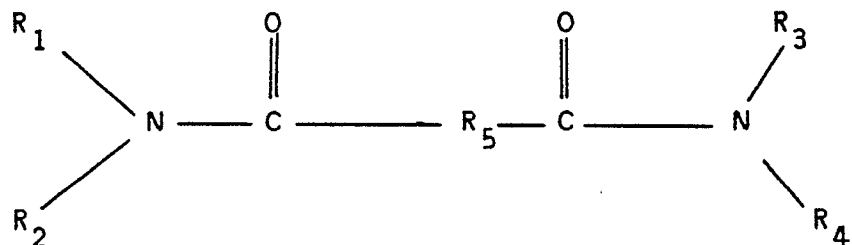


ii) diamides of the formula :

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iii) triamides of the formula :

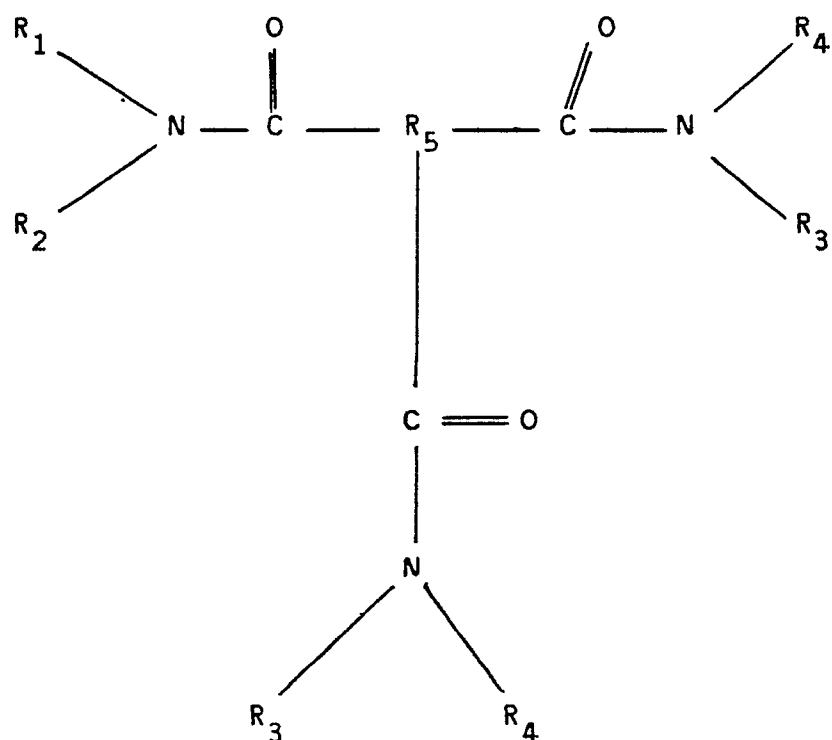
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wherein R_1 , R_2 , R_3 , R_4 , are, selected independently, C_{12-22} alk(en)yl, aryl, or alkyl-aryl groups, possibly containing hydroxy groups, ester groups, and/or ether linkages.

R_5 is selected from the group of : alk(en)yl groups containing from 1 to 10 carbon atoms ; aryl groups, substituted aryl groups, heteroaromatic groups, substituted heteroaromatic groups, polyaromatic groups. R_5

possibly containing hydroxy groups, ester groups, and/or ether linkages.

iv) sulfonamides corresponding to the amides described in i) to iii).

Detailed Description of the Invention

The softening agent -

The softening agents for use in the present invention are selected from the amides or sulfonamides of the formulas i) to v) disclosed hereinabove.

Preferably, R_1 , R_2 , R_3 , R_4 in formula i) to v) are all selected from tallow and coconut alkyl groups.

Preferred compounds have the formula ii) with R_5 being either a C_{1-5} alkyl or alkenyl group, or a phenyl or substituted phenyl group.

When R_5 is an alk(en)yl group, preferred compounds include the tetra-coconut and tetra-tallow diamides derived from the following carboxylic acids : malonic, succinic, maleic, glutaric, adipic.

An especially preferred compound of above category is tetracoconut adipamide.

When R_5 is an aromatic group, the amide group bearing R_3 and R_4 chains can be in any of the ortho, meta, or para position with regard to the amide group bearing R_1 and R_2 chains.

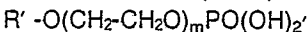
When R_5 is a phenyl or substituted phenyl group, preferred compounds are tetra-tallow terephthalic diamide and tetracoconut terephthalic diamide.

A preferred polyaromatic group for R_5 is biphenyl.

The softening agent is used at levels of from 0.1% to 15% by weight, preferably from 1% to 10% by weight, most preferably from 3% to 6% by weight.

In a highly preferred embodiment, the softening agent is pre-mixed (predispersed) with a dispersing agent, and the resulting mixture is then added to the rest of the composition.

Any conventional dispersing agent can be used for that purpose. Examples of suitable dispersing agents include nonionic surfactants resulting from the condensation of primary or secondary aliphatic alcohols e.g. tallow alcohol or alkyl phenol, with from 5-12 ethylene oxide units; and phosphate esters as described in European patent application n° 85201127.9, having the formula :



with $R' = C_{12-14}$ and $m = 1-5$, sold under the Trade Name "Servoxyl UPAZ".

In a particularly preferred embodiment, fatty acids are used as dispersing agents for the amide softener. Fatty acids having from 10 to 20 carbon atoms in the alkyl chain like lauric, myristic, palmitic, stearic, oleic acids and mixtures thereof can advantageously be used in the present context. Especially preferred is a mixture of palmitic and stearic acids.

Fatty acid dispersing agents are used most preferably in a weight ratio of 1/1 to 10/1 of amide softening agent to fatty acid.

Deterasive surfactants -

The compositions of this invention will typically contain organic surface-active agents ("surfactants") to provide the usual cleaning benefits associated with the use of such materials.

Deterasive surfactants useful herein include well-known synthetic anionic, nonionic, amphoteric and zwitterionic surfactants. Typical of these are the alkyl benzene sulfonates, alkyl-and alkylether sulfates, paraffin sulfonates, olefin sulfonates, alkoxylates (especially ethoxylated) alcohols and alkyl phenols, amine oxides, alpha-sulfonates of fatty acids and of fatty acid esters, and the like, which are well-known from the detergency art. In general, such deterasive surfactants contain an alkyl group in the C_9 - C_{18} range; the anionic deterasive surfactants can be used in the form of their sodium, potassium or triethanolammonium salts; the nonionics generally contain from about 5 to about 17 ethylene oxide groups. U.S. Patent 4.111.855 contains detailed listings of such typical deterasive surfactants. C_{11} - C_{16} alkyl benzene sulfonates, C_{12} - C_{18} paraffin-sulfonates and alkyl sulfates, and the ethoxylated alcohols and alkyl phenols are especially preferred in the compositions of the present type.

Also useful herein as the surfactant are the water-soluble soaps, e.g. the common sodium and

potassium coconut or tallow soaps well-known in the art.

The surfactant component can comprise as little as 1% of the compositions herein, but preferably the compositions will contain 5% to 40%, preferably 10% to 30%, of surfactant. Mixtures of the ethoxylated nonionics with anionics such as the alkyl benzene sulfonates, alkyl sulfates and paraffin sulfonates are preferred for through-the-wash cleansing of a broad spectrum of soils and stains from fabrics.

Deterative adjuncts -

The amide softening agent is preferably, without this being an essential requirement, used in combination with a detergent-compatible clay softener. Such clay softeners are well-known in the detergent art and are in broad commercial use, both in Europe and in the United States. Included among such clay softeners are various heat-treated kaolins and various multi-layer smectites. Preferred clay softeners are smectite softener clays that are described in German Patent Application 23 34 899 and in U.K. Patent 1.400.898, which can be referred to for details. Softener clays are used in the preferred compositions at levels of at least 1%, generally 1-20%, preferably 2-10%.

The compositions herein can contain other ingredients which aid in their cleaning performance. For example, the compositions herein can advantageously contain a bleaching agent, especially a peroxyacid bleaching agent, without any prejudice to the stability and overall performance, thanks to the compatibility of the amide softening agents of the invention with peroxyacid bleaching agents. In the context of the present invention, the term peroxyacid bleaching agent encompasses both peroxyacids per se and systems which are able to yield peroxyacids in situ.

Peroxyacids per se are meant to include the alkaline and alkaline-earth metal salts thereof. Peroxyacids and diperoxyacids are commonly used; examples are diperoxydodecanoic acid (DPDA) or peroxyphthalic acid.

Systems capable of delivering peracids in situ consist of a peroxygen bleaching agent and an activator therefor.

The peroxygen bleaching agents are those capable of yielding hydrogen peroxide in an aqueous solution; those compounds are well-known in the art, and include hydrogen peroxide, alkali-metal peroxides, organic peroxide bleaching agents such as urea peroxide, inorganic persalt bleaching agents such as alkali metal perborates, percarbonates, perphosphates, persulfates, and the like.

Preferred are sodium perborate, commercially available in the form of mono- and tetra-hydrates, sodium carbonate peroxyhydrate, sodium pyrophosphate peroxyhydrate and urea peroxyhydrate.

The liberated hydrogen peroxide reacts with the bleach activator to form the peroxyacid bleach. Classes of bleach activators include esters, imides, imidazoles, oximes, and carbonates. In those classes, preferred materials include methyl o-acetoxy benzoates; sodium-p-acetoxy benzene sulfonates such as sodium 4-nonanoxyloxybenzene sulfonate; sodium-4-octanoxyloxybenzene sulfonate, and sodium-4-decanoxyloxybenzenesulfonate; biophenol A diacetate; tetra acetyl ethylene diamine; tetra acetyl hexamethylene diamine; tetra acetyl methylene diamine.

Other highly preferred peroxygen bleach activators which are disclosed in U.S. Patents 4.483.778 and 4.539.130, are alpha-substituted alkyl or alkenyl esters, such as sodium-4(2-chlorooctanoxyloxy)benzene sulfonate, sodium 4-(3,5,5-trimethyl hexanoxyloxy)benzene sulfonate. Suitable peroxyacids are also peroxygen bleach activators such as described in published European Patent Application N° 0 166 571, i.e., compounds of the general type RXOOH and RXAL, wherein R is a hydroxycarbonyl group, X is a heteroatom, A is a carbonyl bridging group and L is a leaving group, especially oxybenzenesulfonate.

The compositions herein may contain alternatively any other type of bleaching agent, conventionally used in detergent compositions. Also, it is highly preferred that through-the-wash detergent compositions contain a detergent builder and/or metal ion sequestrant. Compounds classifiable and well-known in the art as detergent builders include the nitrilotriacetates, polycarboxylates, citrates, water-soluble phosphates such as tri-polyphosphate and sodium ortho- and pyro-phosphates, silicates, and mixtures thereof. Metal ion sequestrants include all of the above, plus materials like ethylenediaminetetraacetate, the amino-polyphosphonates and phosphates (DEQUEST) and a wide variety of other poly-functional organic acids and salts too numerous to mention in detail here. See U.S. Patent 3.579.454 for typical examples of the use of such materials in various cleaning compositions. In general, the builder/sequestrant will comprise about 0.5% to 45% of the composition. The 1-10 micron size zeolite (e.g. zeolite A) builders disclosed in German Patent 24 22 655 are especially preferred for use in low-phosphate or non-phosphate compositions.

The laundry compositions herein also preferably contain enzymes to enhance their through-the-wash cleaning performance on a variety of soils and stains, such as amylase, protease, cellulase enzymes.

Amylase and protease enzymes suitable for use in detergents are well-known in the art and in commercially available liquid and granular detergents. Commercial deterative enzymes (preferably a mixture of amylase and protease) are typically used at levels of 0.001% to 2%, and higher, in the present compositions. Other highly desirable detergent ingredients for use in the detergent compositions of the present invention are

5 quaternary ammonium compounds of the form $R_4R_5R_6R_7N^+ X^-$, wherein R_4 is alkyl having from 10 to 20, preferably from 12-18 carbon atoms, and R_5 , R_6 and R_7 are each C_1 to C_4 alkyl preferably methyl; X^- is an anion, e.g. chloride. Examples of such quaternary ammonium compounds include C_{12} - C_{14} alkyl trimethyl ammonium chloride and cocoalkyl trimethyl ammonium methosulfate. The quaternary ammonium compounds can be used at levels from 0.5% to 5%, preferably from 1% to 3%.

10 Moreover, the compositions herein can contain, in addition to ingredients already mentioned, various other optional ingredients typically used in commercial products to provide aesthetic or additional product performance benefits. Typical ingredients include pH regulants, perfumes, dyes, optical brighteners, soil suspending agents, hydrotropes and gel-control agents, freeze-thaw stabilizers, bactericides, preservatives, suds control agents, bleach stabilizing agents.

15 In a through-the-wash mode, the compositions are typically used at a concentration of at least 500 ppm, preferably 0.10% to 1.5%, in an aqueous laundry bath at pH 7-11 to launder fabrics. The laundering can be carried out over the range from 5°C to the boil, with excellent results.

20 Form and Preparation of the compositions -

The detergent compositions of this invention can be present in any suitable physical state inclusive of granular, liquid, pasty, or sheet-like form. They may be prepared in any way, as appropriate to their physical form, by mixing the components, co-agglomerating them, micro-encapsulating them, dispersing

25 them in a liquid carrier, and releasably adsorbing or coating them onto a non-particulate substrate, such as a non-woven or paper sheet.

Preferably, the compositions are in granular form.

A preferred method of preparation of said granular compositions consists in preparing a melt of the dispersing agent and the amide, dispersing the molten mixture into a stirred, aqueous crutcher mix

30 comprising the balance of the deterative ingredients, and spray-drying in standard fashion. In an equally preferred mode, the melt can be atomized onto the detergent granules. The compositions herein may also be sprayed onto particles of, e.g., sodium perborate mono or tetrahydrate, sodium sulfate, sodium carbonate, sodium silicate, sodium phosphate, or clay of the type described above.

35 Industrial Application

The following examples are typical of the preferred execution of the invention, but are not intended to limit the scope.

40 EXAMPLE 1

A mix of tetracoconut terephthalic diamide (total 5% of complete formulation after spray-on) and lauric acid (1.5% of complete formulation) are admixed, melted and stirred until homogeneity. A standard aqueous crutcher mix comprising the following ingredients is prepared (percentages listed relate to percent ingredients in the complete formulation after spray-drying).

	<u>Ingredients</u>	<u>Percent</u>
5	C ₁₁ -C ₁₂ alkyl benzene sulfonate	6.2
	Tallow alcohol ethoxylate (EO11)	1.0
	Sodium tripolyphosphate	24.0
10	Sodium sulfate	15.0
	Sodium silicate	8.0
	Smectite clay *	6.5
	Carboxymethyl cellulose	0.4
15	Polyacrylate (soil suspender)	1.7
	Enzymes	0.5
	Optical brightener	0.23
20	Sulfonated zinc phthalocyanine **	25 ppm
	EDTA	0.2
	Perfume/copper salts/minors	0.5
25	C ₁₂ -C ₁₄ alkyl trimethylammonium chloride	1.9
	Moisture	to 77%

The crutcher mix is then handled in entirely standard fashion, and spray-dried. After drying, sodium perborate (20%) and bleach activator (3% 3-5-5-trimethyl hexanoic acid, sulfaphenyl ester, sodium salt^{***}) (3%) are dry-mixed with the granules.

The tetracoconut terephthalic diamide/lauric acid melt is then sprayed onto the finished product.

* Natural smectite: ion exchange capacity above 50 meq/100 g clay

** U.S. Patent 3.927.967

35 *** U.S. Patents 4.483.778 & 4.539.130.

The composition of Example 1 was compared for through-the-wash softness vs. an identical composition which did not contain the tetracoconut terephthalic diamide/lauric acid premix (reference). The design of the test was such as to compare softness of textile pieces laundered 1 time (monocycle) and 4 times (multi-cycle) with invention and reference composition.

40 The testing conditions were as follows:

- automatic drum washing machine MIELE 423
- heating up from 15°C to 90°C; 50' at 90°C
- 1% product concentration in wash liquor
- 18 grains/gallon (0.31g/l) water hardness (3:1 Ca/Mg ratio).

45 The washed and line dried swatches were compared by a panel of two expert judges, working independently, by a paired comparison technique using a 9-point Scheffe scale. Differences were recorded in panel score units (psu), positive being performancewise better and the least significant difference (LSD) at 95% confidence was also calculated.

The testing results were as follows:

50

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Softness (Ex. 1 vs. Reference)

	1 cycle	4 cycles
5 Terry fabric (12)	+ 1.30 (0.49)*	+ 0.80 (0.44)
10 Polyester fabric (12)	+ 1.0 (0.63)	+ 1.00 (0.00)

* value between parentheses is LSD value (see text)

These results show the significant through-the-wash softness benefits derivable from the composition of the invention.

EXAMPLE 2

A low-P spray-dried detergent formulation is as follows :

	<u>Ingredients</u>	<u>Percent</u>
25	Zeolite A (1-10 microns)	26.0
	Sodium nitrilotriacetate	5.0
	Smectite clay *	3.0
30	Tetracoconut adipamide/lauric acid (3:1 wt ratio)**	5.0
	C ₁₁ -C ₁₂ alkyl benzene sulfonate	6.5
	Tallow ethoxylate (EO 9-11)	0.5
35	Sodium perborate 4H ₂ O ***	20
	Tetraacetyl ethylenediamine (TAED)***	3
	Sodium silicate	8
	CMC	1
40	Sodium sulfate	18
	Enzymes (1:1 amylase/protease)***	1.5
	Optical brightener	0.5
45	Water, minors	to 100

50 * As Gelwhite GP (TM) : CaCO₃ ion exchange capacity
70Meq/100 g

** Prepared as in Example 1 and sprayed onto the
finished product

55 *** Dry-mixed with composition.

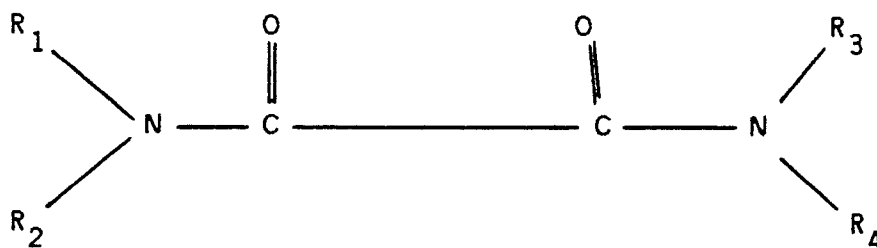
The composition of Example 2 is prepared in the manner described for Example 1. The composition of

Example 2 provides better softness than the composition wherein the amide/lauric acid complexe have been replaced by additional sodium sulfate.

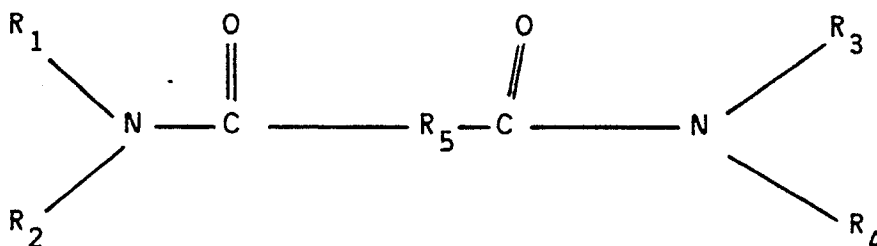
5 Claims

1. A detergent composition containing surfactant, a nitrogen-containing fabric softener and, if desired, deterative additives, characterized in that the nitrogen-containing softener is selected from the group of :

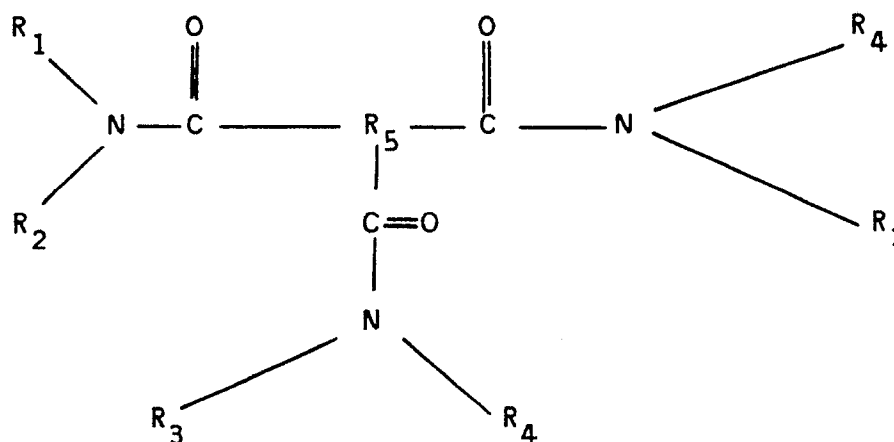
i) diamides of the formula :



ii) diamides of the formula :



iii) triamides of the formula :



wherein :

R_1, R_2, R_3, R_4 , are selected independently, C_{12-22} alk(en)yl, aryl, or alkyl-aryl groups possibly containing hydroxy groups, ester groups and/or ether linkages.

R_5 is selected from the group of : alk(en)yl groups containing from 1 to 10 carbon atoms ; aryl groups,

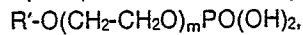
substituted aryl groups, heteroaromatic groups, substituted heteroaromatic groups, polyaromatic groups.

R₅ possibly containing hydroxy groups, ester groups, and/or ether linkages.

iv) sulfonamides corresponding to the amides described in i) to iv).

2. A composition in accordance with Claim 1 wherein the amide softening agent is predispersed with a dispersing agent selected from:

- nonionic surfactants resulting from the condensation of primary or secondary aliphatic alcohols;
- phosphate esters having the formula



with R' = C₁₂-C₁₄ and m = 1-5;

- fatty acids having from 10 to 20 carbon atoms in the alkyl chain.

3. A composition in accordance with Claim 2 wherein the dispersing agent is a fatty acid having from 10 to 20 carbon atoms in the alkyl chain and is present in a weight ratio of amide softening agent to fatty acid of from 1:1 to 10:1.

4. A composition in accordance with Claims 1-3 wherein R₁, R₂, R₃, R₄ are coconut alkyl or tallow alkyl groups.

5. A composition in accordance with Claims 1-4, wherein the nitrogen-containing softening agent has the formula ii), with R₅ being an alkyl group of from 1 to 5 carbon atoms.

6. A composition in accordance with claims 1-5 wherein the nitrogen-containing softening agent has the formula ii) with R₅ being a phenyl group or a substituted phenyl group.

7. A composition in accordance with Claim 6 wherein the nitrogen-containing softening agent is: tetratallow terephthalic diamide, or tetracoconut terephthalic diamide.

8. A composition in accordance with Claims 1 to 7 wherein the amount of nitrogen-containing softening agent is from 0.1% to 15% by weight.

9. A composition in accordance with Claims 1 to 8 which in addition contains a peroxyacid bleaching compound

10. A composition in accordance with Claim 9 wherein the peroxyacid bleaching compound is comprised of a peroxygen bleaching compound and an activator therefor.