

[54] **AGENT FOR THE POSTTREATMENT OF LAUNDRY**

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[51] **Int. Cl.**.....D06m 13/40, C11c 3/00

[58] **Field of Search**.....252/8.8, 8.9; 117/139.5 F, 117/143; 260/404, 211

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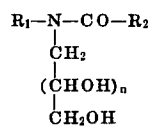
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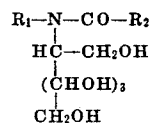
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[57] **ABSTRACT**

A softening agent for the posttreatment of washed laundry is disclosed, the same being adapted for addition to the rinse water either as a solid or in liquid form. The softening agent corresponds to the following formula:



and



wherein R₁ is alkyl containing 10 to 22 carbon atoms, and may be interrupted by either oxygen atoms in the vicinity of the N atom group, R₂ is alkyl containing seven to 21 carbon atoms and n is either 3 or 4. Preferably the total number of the carbon atoms contained in R₁ and R₂ amounts to at least 23 and preferably amounts to from 27 to 35. The aforesaid softening agents can be employed per se or in combination with other conventional laundry agents such as optical brighteners, acidifiers, disincrusting agents, colorants, perfumes, antimicrobial agents, etc.

3 Claims, 3 Drawing Figures

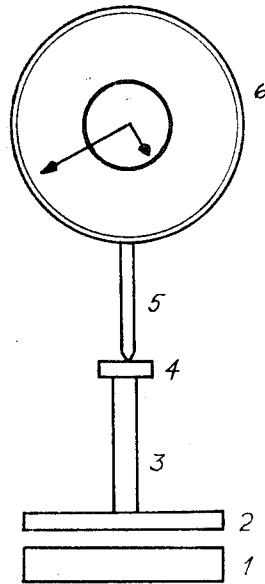


FIG. 1

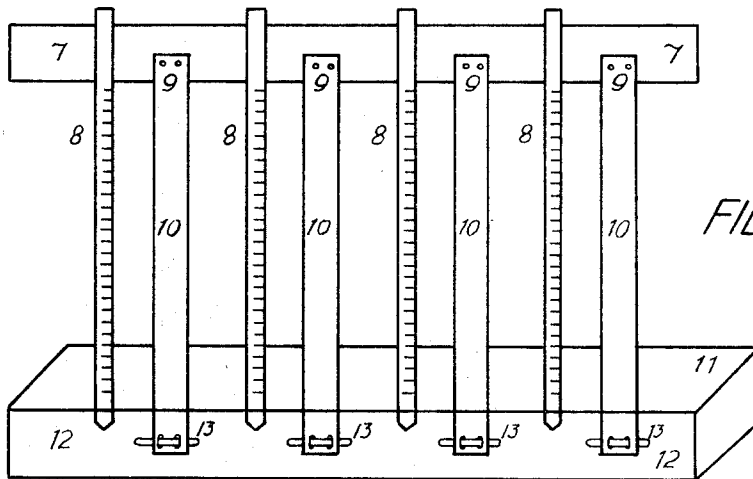


FIG. 2

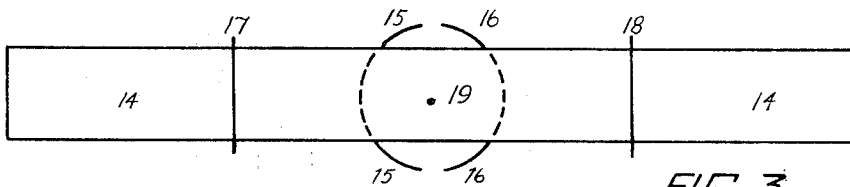


FIG. 3

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AGENT FOR THE POSTTREATMENT OF LAUNDRY

This invention relates to softening agents for use in the post-treatment of laundry, to compositions containing such softening agents as active ingredient and to the methods of preparing and using such agents and compositions.

Following the drying of wet textiles, particularly those prepared on a basis of cotton or similar cellulosic fibers, a perceptible hardening of the feel of the fabrics can be detected. This is particularly unpleasant in the case of underclothing, table linens, and handkerchiefs, i.e., articles which are subjected to frequent and repeated washing, especially when they are washed in drum-type washing machines, whether in the home or in an industrial laundry.

This undesirable loss of good feel in the laundering process can be avoided if cationic substances are added to the final rinse, which substances contain at least two fatty radicals of high molecular weight in their molecules. In practice, dialkyl dimethyl ammonium salts that are dispersible in water have become popular for this purpose, each of the two alkyl radicals in this latter compound contains 16 to 18 carbon atoms. These cationic softening agents are usually marketed in the form of their aqueous dispersions.

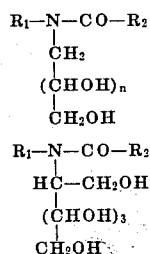
Although when these cationic softening agents are used in the final rinse the undesirable hardness is not found in the laundry after drying, the use of these products entails certain disadvantages. If the detergent dissolved in the final wash has not been entirely removed from the clothing, which may be the case, particularly in tub-type washing machines, precipitates are formed between the cationic softening agents and the anionic wash-active substances, thereby diminishing or obstructing the softening effect. Furthermore, these precipitates can themselves be deposited on the fibers and cause yellowing of the laundered items. Furthermore, the absorbency of the washed goods is adversely affected by the softening agents as known in the prior art. Even in the case of drum-type washing machines, in which the rinsing action is generally better than it is in tub-type machines, a reduction of the absorbency of the posttreated clothing can be observed, apparently because small amounts of the anionic wash-active substances are fixed on the fibers and then react with the cationic softening agents.

It is an object of this invention to eliminate disadvantages of the prior art methods of posttreating laundry by the provision of a new softening agent.

It is an additional object of the invention to provide laundry posttreatment agents which contain the active softening agent of the invention in a form designed for easy and practical use.

These and other objects and advantages of the invention will become apparent from a further consideration of the following disclosure.

According to the invention it has now been found that laundry treated, after washing, with an aqueous solution or dispersion of a softening agent, having the following formula:



wherein R_1 represents an alkyl radical having 10 to 22, preferably 12 to 20, and most preferably 16 to 18 carbon atoms, R_2 represents an alkyl radical having 7 to 21, preferably 11 to 19, and most preferably 15 to 17 carbon atoms, and n is either of the whole numbers 3 or 4, further wherein the alkyl radical R_1 can be interrupted by an ether oxygen atom in the vicinity of the NH_2 group, and the sum of the

carbon atoms contained in R_1 and R_2 amounts to at least 23 and preferably to from 27 to 35, is characterized by improved feel, i.e., softness and absorbency. Preferably $n=4$, i.e., N -alkyl- N -acylglycamines are employed as softening agents. The latter will hereinafter be called "glycamides" for the sake of simplicity. The data given on the glycamide softening agents also apply accordingly to compounds wherein $n=3$. These softening agents can be employed in the treatment of washed textiles, alone or together with other conventional laundry post-treatment agents, such as acidifiers or disincrusting agents, optical brighteners and/or heavy metal-free, oxidizing, water-soluble antimicrobial agents.

The textiles or other laundered items which have been rinsed with the products of the invention, possess, after drying, not only a high degree of fullness and softness, but they also possess excellent absorbency. Furthermore, the softening agents of the invention are quite compatible with anionic optical brightening agents. If any of the above-mentioned antimicrobial agents are used, not only are any microbes that might be present in the washed textiles or in the rinse water killed, but often antimicrobial properties are conferred upon the posttreated textiles.

The glycamides to be used in the method of the invention or contained in the preparations according to the invention can be prepared by amidation in the known manner from the appropriate glycamines which can be produced by the hydrogenation of hexoses or their oligomers using therefor Raney nickel catalyst in the presence of higher alkyl amines.

The principal hexoses involved are monosaccharides such as glucose, mannose, galactose, fructose, sorbose or their mixtures such as invert sugar; however, their oligomers can also be used, such as maltose, for example. Instead of hexoses, pentoses, mixtures of pentoses or mixtures of pentoses and hexoses can be used.

The amines suitable for use in the reaction are primary alkylmonoamines. The alkyl radicals of these amines, like the alkyl radicals of the fatty esters to be used in the amidation, can be saturated, straight-chained or branched, and of synthetic or natural origin. The radicals R_1 present in the glycamides can be derived from decyl, lauryl, myristyl, cetyl, stearyl, oleyl, arachyl, or behenyl amine. There are also suitable those amines whose carbon chain is interrupted by an ether oxygen atom in the vicinity of the N atom, preferably between carbon atoms 2 and 3 or 3 and 4 (counting from the N atom. Such amines, in which the N atom and the ether oxygen atom are separated from one another by three carbon atoms, are obtained for example by addition of acrylonitrile to a suitable fatty alcohol followed by reduction of the nitrile group.

The fatty acid radicals R_2-CO- can be derived from capric, lauric, myristic, palmitic, stearic, oleic, arachic or behenic acid. Glycamides can also be used in which the amine and/or the fatty acid radicals are present as isomer mixtures, such as those which can be obtained from fats of natural origin. Accordingly, these amines and fatty esters can be made from the fats derived from plants, land or marine animals, as, for example, from coconut oil, palm oil, linseed oil, cottonseed oil, peanut oil, rapeseed oil, etc., from hog lard, tallow or fish or whale oil or from fractions of the fatty acids contained in these fats. The amines or fatty esters to be used are not to be excessively unsaturated, i.e., they are not to have an iodine number higher than 50, and preferably not higher than 30. In fact, even substantially completely hardened products having iodine numbers below 10 and preferably below 5 can be used.

The methyl esters which are employed are preferably fatty acid esters. There can, however, be used to equal advantage the esters of monoalcohols having two to four carbon atoms, as, for example, the esters of ethyl, propyl, isopropyl, butyl and isobutyl alcohol etc. Suitable for use herein are also the fatty acid glycerides.

The amidation of the alkylglycamines is carried out by heating the fatty esters with the amines at temperatures of 150° to $170^\circ C.$, preferably with agitation. The course of the reaction

can easily be observed by intercepting the alcohol released by the reaction. When the amount of liberated alcohol has reached approximately the theoretical, the reaction is stopped. In no case is the reaction mixture or the reaction product that has been found to be heated so intensely or so long as to produce dehydration.

The glycamide thus obtained can be used directly for the purposes of the invention. If desired, however, it can first be purified, as for example by recrystallization. The glycamides are moderately soluble in water, but are readily soluble in water-soluble organic solvents, such as monohydric or polyhydric alcohols having one to four carbon atoms, or in ether alcohols, such as the monoethers formed by reaction of the above-mentioned univalent alcohols with ethylene glycol, diethylene glycol, propylene glycol, butylene glycol, or by the monoethers or diethers of the said monohydric alcohols formed by reaction of the monohydric alcohols with glycerine. The glycamides are also soluble in the lower water-soluble ketones. The aforesaid solvents, particularly those having boiling points below 100° C., are also suitable for the recrystallization of the glycamides.

In accordance with the invention, the washed textiles or other laundered items are contacted with a rinse water containing the glycamides in concentrations of approximately 0.05 to about 2 grams per liter and preferably from 0.1 to about 0.5 g./l.

The glycamides can be used directly in the first rinse, even if the remains of the washing medium have not been completely washed out of the goods. Since, of course, it has become an established practice to add the softening agent to the final rinse, the same can, obviously be done when using the glycamides of the invention or the softening agents containing them.

After the rinse, as effected in accordance with the invention, the laundry is dried in the conventional manner. The laundry is soft and full and has a pleasant feel, which becomes pleasantly observable especially in the case of articles which come in contact with the skin, such as underwear, bed linen, and handkerchiefs. In particular, wash-and-drying cloths exhibit a remarkably good absorbency, which is greatly appreciated in the case of dish towels and bath towels. Further the moist articles which have been soft-rinsed according to the invention yield their moisture more quickly than others on being subjected to drying. Finally, the textiles thus treated are observed to iron more easily than the conventionally laundered articles.

Since the glycamides act on the textiles or other laundered items for only a relatively short period especially in the case of automatic washing machines, it is advantageous to use the moderately water-soluble glycamides in finely divided form. This is accomplished in accordance with the invention by using the posttreatment agents in solid or liquid form and in a form in which the glycamide is mixed together with the conventional solid or liquid diluents and/or fillers.

Water-soluble inorganic, preferably weakly alkaline, neutral or weakly acid substances are suitable for use as solid fillers. For the manufacture of solid preparations, inorganic or organic salts are of primary importance, as for example the alkali salts of sulfuric acid, orthophosphoric acid or pyrophosphoric acid. However, salts of organic acids can also be used, such as the nonsurface-active salts of carboxylic acids or hydroxycarboxylic acids having one to 10, preferably one to six carbon atoms, as for example, salts of acetic acid, propionic acid, lactic acid, citric acid, tartaric acid, benzoic acid, phthalic acid, etc. Water-soluble inorganic or organic acid amides or their salts may also be present as solid fillers, as for example, amidosulfuric acid salts, amidophosphoric acid salts and water-soluble amides or carboxylic acids, such as urea, acetamide, etc. Solid polyethylene glycols are also usable as fillers.

The solid posttreating agents of the invention can be prepared for example by spraying a solution or dispersion of the glycamides in a suitable liquid onto the finely divided solid

filler substances. It is also possible to spray aqueous solutions or suspensions of glycamides having a percentage of fillers dissolved or dispersed therein, in the known manner, and thus to obtain readily soluble powders or granules. Lastly, solutions or dispersions of the glycamides in fused urea or in fused polyethylene glycols can be sprayed or otherwise converted into easily soluble powders or granules.

The time which is required for the glycamides added as concentrates to the rinse water to reach the desired concentration for use can be still further reduced by using liquid products in which the glycamides are dissolved in a solvent or dispersed in water. In this manner no time is lost, i.e., taken up in the dissolution of a solid filler, and instead the desired concentration for use establishes itself immediately after the addition of the liquid concentrate to the rinse water.

The liquid aqueous concentrates are prepared by dispersing the moderately water-soluble glycamides in an aqueous phase. It has proved advantageous for the glycamides first to be dissolved in one of the above-named water-soluble organic solvents and thereafter for the resulting solutions to combine with water. The solid glycamides thereupon separate in finely divided form, the organic solvents serving as solubilizers. These aqueous solutions can of course contain any of above-named solid fillers if the stability of dispersion is not adversely affected thereby. In many cases the stability may be improved by such addition. For this purpose, the alkali salts of lower carboxylic acids or hydroxycarboxylic acids containing up to six carbon atoms, and urea or acetamide have proved particularly suitable and can be used instead of, or together with the above-mentioned water-soluble organic solvents also as solubilizers.

The known dispersing and emulsifying agents or emulsion stabilizers can be used for effecting the stabilization of the aqueous glycamide dispersions.

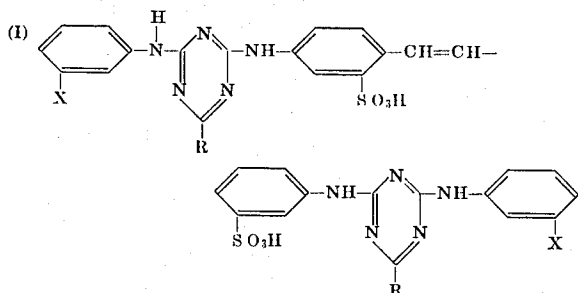
Suitable dispersing or emulsifying agents include the known anionic or nonionic wash-active substances whose action is based on the simultaneous presence of one hydrophobic and one hydrophilic radical in their molecules. The hydrophobic radical consists usually of an alkyl radical having eight to 20, preferably 12 to 18 carbon atoms. Anionic or nonionic groups are suitable as hydrophilic radicals. The anionic groups include carboxyl, sulfonic acid or sulfuric acid semiester groups; the nonionic groups include aggregations of hydroxyl groups or polyethylene glycol ether chains. Accordingly, alkylbenzenesulfonates, fatty alcohol sulfates, fatty alcohol glycol ether sulfates having one to five ethylene glycol ether radicals in the molecule, fatty acid monoglycerides, polyethylene glycol ethers of fatty alcohols or alkylphenols having five to 20 ethylene glycol ether radicals in the molecule are suitable for use as dispersing and emulsifying agents. In no case, however, must the amount of these dispersing and emulsifying agents be greater than the amount of the glycamide present and preferably the dispersing or emulsifying agent should be present in substantially lesser quantities amounting to at the most to 50 percent, and preferably amounting to from 5 to 25 percent of the glycamides.

Suitable emulsion stabilizers include the water-soluble colloids, such as the salts of ether carboxylic acids or ether sulfonic acids of cellulose, cellulose sulfate, polyacrylic acid or polymethacrylic acid salts, water-soluble polyacrylamides and polyethylene glycols, preferably those having a molecular weight greater than 600, and particularly the solid polyethylene glycols. Many of these emulsion stabilizers produce a great increase in the viscosity of the aqueous solutions, and therefore they are used in quantities preferably of less than 1 percent so that the posttreatment agents will still be readily fluid in nature. Others, particularly the polyethylene glycols, can be used in substantially greater quantities, without raising the viscosity to undesirable levels.

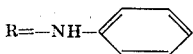
The glycamides according to the invention and the compositions containing such glycamides can advantageously be combined with other substances which have heretofore been used as posttreatment agents. These include, for example, op-

tical brighteners, acidifying and disincrusting agents, colorants, perfumes, etc. These substances can be incorporated into the solid or liquid posttreatment agents according to the invention.

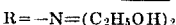
The combined use of the above-described softening agents with optical brighteners is to be considered as an important and novel feature of the invention. The brighteners are intended mainly for cellulose fibers, but brighteners for cellulose and/or other fibers, especially synthetic fibers, can be used as well. For example, the following types of brighteners can be used:



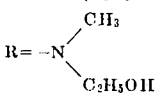
a: X=H;



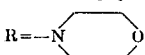
b: X=H;



c: X=H;

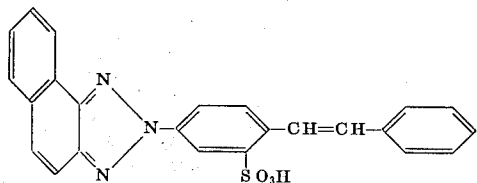


d: X=H;



e: X=SO₃H; R=N-(C₂H₅OH)₂'.

(II)



Suitable additives for acidification and/or for the removal of ash or other fiber incrustations include the nonoxidizing acids which have been found to be not harmful to the fibers to be treated and particularly those additives which form soluble alkaline earth salts, and complex compound formers. The acid reacting substances include for example amidosulfuric acid, urea compounds of orthophosphoric acid or solid organic acids like citric acid. The complex compound formers include for example tripolyphosphates or the higher but still water-soluble polyphosphates of alkalis, salts of nitrilotriacetic acid, ethylenediamine tetracetic acid, N-ox-yethylethylenediaminetriacetic acid and other conventional organic complex compound forming agents, such as the salts of certain di-, tri-, or tetraphosphonic acids.

The term antimicrobial agents as used herein is understood to mean both bactericidal and bacteriostatic, and fungicidal and/or fungistatic products. The antimicrobial agents are required to be water-soluble either per se or in the form of their salts. Examples of suitable antimicrobial agents for use herein are formaldehyde, halogenated phenols, nitrated mono- or polyalcohols, and antimicrobial surfactants.

As instances of halogenated phenols which may be used as antimicrobials, there may be mentioned, for example, chlorination and bromination products of phenol, such as pentachlorophenol, and also halogenated cresols, xylenols, such as 4-bromo-3,5-xyleneol or halogenated cyclohexylphenols, methylcyclohexylphenols or benzylphenols.

There may also be used the water-soluble antimicrobial substances of the cationic or hybrid ionic surfactant type, which serve in the final posttreatment agents also as dispersing agents.

Instances of cationic substances which are suitable as antimicrobials include quaternary ammonium compounds which contain on the nitrogen atom thereof an aliphatic hydrocarbon radical containing eight to 18, and preferably 10 to 14 carbon atoms, or alternatively contain on the nitrogen atom at least one aromatic radical or a radical having double bonds, the latter radical being linked to the nitrogen atom by an aliphatic carbon atom. The following are instances of the cationic compounds suitable for use in the invention include: diethylbenzyl-dodecyl ammonium chloride, diethylbenzyl-octyl ammonium chloride, and dibutylallyl, methylethylbenzyl, ethylcyclohexylallyl and ethylcrotyldiethylaminoethyl dodecyl ammonium chloride.

There are also tertiary amines suitable for use which can be prepared by condensing a primary or secondary amine containing up to four basic nitrogen atoms and one aliphatic or alkylaromatic radical having eight to 18 and preferably eight to 14 aliphatic carbon atoms with a phenol or lower aliphatic aldehyde, preferably formaldehyde or acetaldehyde. The phenol employed in the condensation reaction may have in its molecule a plurality of sites capable of condensation, and these may be substituted with lower alkyl, alkoxy or benzyl radicals containing up to four carbon atoms. The phenol, alkyl, alkoxy, and benzyl radicals may additionally be substituted with chlorine or bromine atoms and/or with nitro groups. Examples of such condensation products include, for instance, the following compounds: oxybenzyl-octylamine, oxybenzyl-dodecyl-diethylenetriamine, (2-oxy-5-chloro-6-methylbenzyl)-dodecyl-diethylenetriamine, (2-oxy-5-methylbenzyl)-octyl-diethylenetriamine, (2-oxy-5-chloro-4,6-dimethylbenzyl)-octyl-diethylenetriamine, (tris-oxybenzyl)-dodecyl-triethylenetetramine, o,o-bis-(tetradecylaminomethyl)-p-cresol, o,o-bis-(dodecyl-diethylenetriaminomethyl)-p-chloro-m-cresol, m,m-bis-(dodecyl-diethylenetriaminomethyl)-p-ox-ybenzoic acid, 2,2-bis-p-ox-y-m,m-di-(dodecyl-diethylenetriaminomethyl)-phenolpropane, o,o-bis-(dodecyl-diethylenetriaminomethyl)-p-nitrophenol, o,o-bis-(dodecyl-propylenediaminomethyl)-p-chloro-m,m-dimethylphenol etc. The aforesaid tertiary amine compounds have a weakly acid reacting hydroxyl group and thus represent an intermediate type between the purely cationic and the hybrid-ionic surfactants.

The hybrid-ionic surfactants which have achieved particular importance as antimicrobials include compounds of the aminocarboxylic acid, polyaminocarboxylic acid and betaine type.

Illustrative of the aminocarboxylic and polyaminocarboxylic acid antimicrobial agents are compounds having the following structural formula:

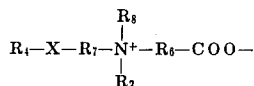


wherein R₄ represents an alkyl or alkylaryl radical of high molecular weight, preferably such a hydrocarbon radical having six to 18 and most preferably eight to 14 aliphatic carbon atoms, R₅ represents an ethylene or propylene radical, x represents a whole number of from 1 to 6 and R₃ represents an aliphatic-aromatic or aromatic bridge having one to six carbon atoms. The following compounds are illustrative of aminocarboxylic and polyaminocarboxylic compounds having the above-mentioned formula: dodecylaminopropylglycine, tetradecylaminoethyl-β-alanine, dodecyl-di-(aminoethyl)-glycine, dodecylaminoisopropylaminomethylsalicylic acid, hexadecyl-tri-(aminoethyl)-β-aminobutyric acid, dodecylbenzylaminopropyl-β-alanine, octylphenoxyethyl-di-(aminoethyl)-glycine, dodecylaminoethylphenylalanine, dodecylaminoethylaminobenzoic acid, etc.

The radical R₄ can also be interrupted by an ether oxygen atom, as is the case, for example, in the following compounds: dodecylxypropylaminopropionic acid, lauryl-1,3-oxypropyl-β-aminobutyric acid, C₁₂₋₁₄-alkyloxyethylaminoacetic acid,

dodecyl-1,2-oxypropylaminomethylsalicylic acid, octylphenoxyethyl-aminobenzoic acid.

The compounds that can be used according to the invention furthermore include carboxybetaines of the formula:



which can be used both in the betaine form, i.e., in the form of inner salts, and in the form of acidic or basic salts. R_4 has the same meaning as in the preceding formula R_7 represents an ethylene or propylene group, and R_6 , R_8 and R_9 represent aliphatic bridges of low molecular weight, X an ether oxygen atom or the $-CONH-$ group. Examples of such carboxybetaine compounds are lauroyl-1,3-midopropyldimethylaminoacetic acid, lauryloxyethyl-di-(hydroxyethyl)-aminopropionic acid, octylphenoxyethyl-di-(hydroxyethyl)-aminoacetic acid, etc.

The antimicrobial agents can be combined in any desired manner with solid or liquid diluents or fillers, with hydrotropic substances, with dispersing and emulsifying agents, with emulsion stabilizers, with brighteners and with acidification and disincrusting agents.

The amounts of the antimicrobial agents contained in the solid or liquid laundry posttreatment agents can vary within wide limits according to the subsequent intended use of the laundered articles, amounting to 0.5-30 percent and preferably 1-20 percent by weight. If the antimicrobial agents are hybrid-ionic surface active substances, their quantity is always smaller than the quantity of the softening agent; preferably the quantity of the hybrid-ionic surface-active antimicrobial agents amounts to no more than 50 percent of the quantity of the softening agent.

The laundry posttreatment agents according to the invention as noted above constitute solid products and as such have the following composition:

30-95 and preferably 70-90 weight percent of solid vehicle substance

70-5 and preferably 30-10 weight percent glycamide

0-20 and preferably 1-15 weight percent of an optical brightener, the same being present in a quantity amounting to 5 to 25 percent of the amount of glycamide present.

0-30 and preferably 1-15 weight percent of a surface-active dispersing or emulsifying agent, the quantity thereof always being less than the amount of the glycamide and preferably no more than 50 percent and most preferably from 5 to 25 percent of the amount of glycamide.

0-20 and preferably 0.1-10 weight percent of an emulsion stabilizer or thickening agent, the quantity thereof being regulated so that a 10 percent aqueous solution or dispersion of the entire solid preparation will have a good fluidity.

0-30 and preferably 1-20 weight percent of a least one of the above-mentioned antimicrobial agents, with the proviso that if they are antimicrobial hybrid-ion surfactants, the quantity thereof is always to be smaller than the quantity of the glycamide, amounting preferably to no more than 50 percent and most preferably to from 5 to 25 percent of the quantity of the glycamide.

The liquid, laundry posttreatment preparations in accordance with the invention have the following composition:

2-30 and preferably 5-15 weight percent glycamide.

0-50 and preferably 5-30 weight percent solid water-soluble hydrotropic substance and/or water-soluble organic solvent.

0-10 and preferably 1-8 weight percent of an optical brightener but preferably such a quantity of optical brightener as to amount to 5 to 25 percent of the amount of the glycamide.

0-10 and preferably 0-5 weight percent of a surface-active emulsifying or dispersing agent, the amount thereof al-

ways being less than the amount of glycamide and amounting preferably to no more than 50, and most preferably 5 to 25 weight percent of the amount of glycamide.

0-20 and preferably 0.1-10 weight percent of a water-soluble emulsion stabilizer or thickening agent, the quantity thereof, however, being regulated so that the final preparation will have good fluidity.

0-30 and preferably 1-20 weight percent of at least one of the above-named antimicrobial agents; if they are antimicrobial hybrid-ion surfactants, the quantity thereof always being less than the quantity of the glycamide, amounting preferably to no more than 50 percent and most preferably from 5 to 25 percent of the quantity of the glycamide.

Balance water.

In the liquid preparations the water can be entirely replaced by water-soluble organic solvents, or at least to such an extent that all of the components and especially the glycamide, are dissolved.

The examples given hereinafter serve to illustrate the invention without being in any way or form considered as limitative of the scope of the same.

The glycamides incorporated into the softening agents of the invention and tested in the manner to be described below were prepared on the basis of the following procedure:

In a three-necked flask equipped with a gas feed tube, agitator, condenser, and a receiver for the distillate, a mixture of 0.22 mole of N-alkylglycamine and 0.20 mole of fatty acid methyl ester was heated slowly under a current of nitrogen, with intense agitation, to 150° to 170° C. The methanol released in the reaction was distilled off and held in a graduated receiver. The reaction mixture was maintained at 150° to 170° C., until about 75 to 80 percent of the theoretical amount of methanol had passed over. This required from 2 to 5 hours depending on the starting material. Thereafter the rest of the methanol was removed by 2 hours of heating in vacuo; the methanol was frozen out in a cooling trap. As soon as 94 to 98 percent of the theoretical amount of methanol had been recovered, the reaction was ended.

The N-alkyl-N-acyl glycamines thus prepared were in the form of pastes or solids, depending on the chain length and on the chain length distribution of the alkyl or acyl radicals. If desired, they could be purified by recrystallization from the water-soluble organic solvents mentioned previously, or from benzene or acetic acid ethyl ester.

The advantageous effects that are achieved with the aforesaid glycamides, i.e., high degree of softness, fullness, and absorbency were demonstrated in tests carried out on ordinary cotton fabrics and on cotton terrycloth fabrics.

These fabrics were washed 15 times successively in a drum-type washing machine, using as washing agent a conventional commercially available washing machine detergent. The active substance in the detergent selected for use consisted substantially of a combination of surface-active sulfonates and soap. The highest temperature reached in the hot water washing cycle was between 95° and 100° C. A number of these fabric specimens were rinsed and dried without the addition of a softening agent. For purposes of comparison, in other experiments, the final rinse water had enough of a solution of the softening agent in isopropanol added to it to bring the concentration in the rinse water to 0.2 grams of active substance per liter. The rinsing cycle was 5 minutes in each case. Thereafter the fabric was dried, acclimated, and then subjected to the tests described hereinafter.

1. Manual Test of the Feel of the Fabric

A number of testers were involved in judging the softness of the treated terrycloth manually, each person scoring it according to the following scale:

1. The feel is very soft and full
2. The feel is soft
3. The feel is medium-stiff
4. The feel is stiff

In many cases the evaluation scores given by all of the testers were identical; where there were differences, an average was taken of the individual scores.

2. Compressibility Tests

In order to achieve an objective standard for evaluating the effect achieved, the compressibility of the washed fabrics was determined. This test is based on the fact that the individual fibers in soft textiles are loosened up, take more space and can be pressed together more easily than in textiles which have become stiff due to washing. Therefore, at the same degree of loading, soft textiles can be compressed much more than stiff textiles.

Terrycloth was used to determine compressibility. The principle of the design of the apparatus used for the measurement can be seen in FIG. 1. The material to be tested rests on the baseplate 1 and is compressed by the feeler descending vertically. The feeler consists of the feeler plate 2, the vertical shaft 3 and the bearing 4, and is guided by the vertical shaft 3, although during the measurement it rests fully on the specimen without any friction on the shaft. In the prolonged portion of the feeler there is a micrometer 6 having a dial on which the smallest unit that can be read is 0.01 mm. To set the micrometer at zero, the feeler plate 2 is lowered onto the baseplate and the feeler 5 is lowered until it contacts the bearing 4 of the feeler. In order to minimize the effect of the contact pressure of the micrometer, the moment at which contact occurs between feeler 5 and bearing 4 is indicated in some instances electrically, e.g., by a neon lamp, and in other instances of the apparatus it is indicated interferometrically in that the bearing 4, for example, is a black glass plate on which even the slightest deformations of the surface by the pressure of the micrometer can be detected interferometrically.

When the zero point of the dial micrometer has been determined, the fabric specimen is placed on the baseplate 1. The specimen must completely cover the baseplate and extend beyond the margins thereof. The feeler (surface area 25 cm.², weight 50 g.) is then lowered onto the specimen and its thickness is measured after a waiting period of 1 minute. The procedure is twice repeated on the same piece of fabric, the pressure being increased, by placing weights on the feeler plate 2, tenfold (500 grams) and twentyfivefold (1,250 grams).

If we assign the symbols d_2 , d_{20} , and d_{50} to the fabric thickness measured with the initial load (2p/cm.²), the tenfold load and the twentyfivefold load (20 and 50 p/cm.², respectively), the relative compressibility of the soft rinsed fabric, z_{rc} , can be calculated according to the following formulas:

$$z_{w^{20}} = \frac{d_2 - d_{20}}{d_2} \text{ and } z_{w^{50}} = \frac{d_2 - d_{50}}{d_2}$$

In order to establish a relationship with the corresponding values for the nonsoft-rinsed fabrics, the relative compressibility of the nonsoft-rinsed fabric, z_h , was determined in the same manner, and the percentage compressibilities for the 20 and 50 p/cm.² loads were calculated from the figures obtained. These values were averaged to form a figure which is taken as the percentage compressibility $Z_{\%}$ for the purpose of evaluating the effect achieved with various softening agents. The percentage compressibility of the nonsoft-rinsed fabrics is taken as 100 percent. The value $Z_{\%}$ is computed according to the following formula:

$$Z_{\%} = \frac{100Z_{w^{20}}}{2Z_h^{20}} + \frac{100Z_h^{50}}{2Z_h^{50}}$$

The magnitude

$$\Delta Z_{\%} = Z_{\%} - 100$$

is also calculated and reflects the percentage improvement of compressibility achieved by the soft-rinsing process.

3. Vertical Absorbency Test

The vertical absorbency was determined on strips of ordinary cotton fabric 20 cm. long and 2.5 cm. wide; the apparatus used for this purpose is represented diagrammatically in FIG. 2. It consists essentially of gauges 8 calibrated in millimeters,

and a rail 7 which is vertically displaceable in relation to these gauges and bears the holding means 9 for holding the textile strips 10. Below it is a trough 11 containing distilled water 12.

The gauges 8 are adjusted so that their zero mark is even with the surface of the water. The fabric strips 10 are kept tense by a weight attached to the bottom thereof, for example, by a glass rod 13 passed through two holes in each strip. To make the measurement, the rail 7 bearing the strips is lowered until the strips 10 dip into the water, and the height to which water rises is read on gauges after 1, 2, 5, 10, 20, 30, and 60 minutes.

In this manner, seven values are obtained for the water rise St . Quantitatively, these values are velocities (cm./min.). Accordingly, an increase in these values indicates an improvement in absorbency.

If the water rise in the nonsoft-rinsed material is designated as St_h , the water rise in the material that was soft-rinsed with the preparations of the invention as St_w , and the water rise of the material that was soft-rinsed with other preparations as St_r , we can obtain the percentage water rise St_x with reference to $St_h=100$ percent:

$$St_x = 100St_w / St_h (\%)$$

and the percentage improvement of water rise, ΔSt_w , with reference to the water rise St_r obtained with the prior art preparation, taken as 100 percent:

$$St_p = 100St_w / St_r - 100 (\%)$$

An average was computed from the values obtained for each textile strip, and these averages are reported in the following examples.

4. Determination of the Absorbency Factor by the Oxé and Schuster Method

The spread of water through a horizontally positioned fabric strip was determined by the method of Oxé and Schuster. The principle of the method can be seen in FIG. 3. The textile strip 14 of ordinary cotton fabric, measuring 12.5x2.5 cm., is stretched horizontally under a specified tension so that it touches two semicircular contact blades 15 and 16 and two straight contact blades 17 and 18, which are under electrical voltage. The diameter of the circle formed by blades 15 and 16 amounts to 3 cm., and the distance between blades 17 and 18 is 5 cm. If a specified amount of water is now injected into the center 19 of the circle formed by contact blades 15 and 16, it will spread out in the fabric. As the moisture front reaches the electrical contacts, first the one circuit and thereafter the second circuit is closed, thereby stopping, by means of an amplifier, two second counters which were started up when the water was injected.

For each textile strip there are two absorption times: one for the inner contact and one for the outer contacts. If the water front takes longer than 200 seconds to reach the contact blades, then evaporation of the water results in unreproducible measuring errors, and the values then obtained do not permit correct determination of the absorbency. In the case of the fabrics which were soft-rinsed with the prior art product dioctadecyl dimethyl ammonium chloride, the absorption times q were more than 200 seconds both at the inner and at the outer contacts. For this reason only the values measured at the inner contact blades were used in the comparison.

An absorbency meter as manufactured by 'Ernst Benz, Textilmaschinen' of Zurich, Switzerland, was used for the measurements.

The values determined represent reciprocal velocities (sec./cm.); an increase in the absorption time q accordingly corresponds to a loss of absorbency. In order to obtain an expression equivalent to the vertical absorbency, the reciprocal values

$$S = 1/q$$

were determined from the measured absorption time q , and these are proportional again in dimension (cm./sec.) to the absorbency. If the absorption values of the nonsoft-rinsed fabric are designated as S_h , those of the fabric soft-rinsed with the preparations of the invention as S_w and those of the fabric soft-rinsed with dioctadecyl dimethyl ammonium chloride as S_r , the percentage absorption values S_x , with reference to $S_h=100$ percent, will be:

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20
25
30
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40
45
50
55
60
65
70
75

$$S_x = 100S_{10}S_n (\%)$$

and the percentage improvement of absorbency value ΔS_y , taking the absorbency value S_x achieved with dioctadecyl dimethyl ammonium chloride as 100 percent, will be:

$$\Delta S_y = 100S_{10}S_y - 100 (\%)$$

Since, of course, the absorption times q with this prior art preparation were always more than 200 seconds, the absorption values ΔS_y themselves could not be determined, but only the absorption values corresponding to an absorption time of 200 sec.; the actual absorption values are in all these cases greater than the absorption values ΔS_y stated below.

All of the tests described in paragraph 1 to 4 above were carried out on specimens which had been acclimated at 20° C., and 65 percent relative humidity.

The following literature is referred to in connection with the testing methods used.

Determination of compressibility:

DIN Standard 53,855 of June 1959

H. Sommer and F. R. Winkler: 'Faserforschung und Textiltechnik' (Akademie-Verlag, Berlin), Vol. 5 (1954), pp. 77 and 78.

Determination of absorbency:

Lindner: Tenside, Textilhilfsmittel, Waschrohstoffe (Verlag Wissenschaftliche Verlagsgesellschaft mbH, Stuttgart), Vol. II (1964), pp. 1,808 and 1,809.

Jahrbuch für das Textil-Reinigungsgewerbe (Busschesche Verlagshandl. Herford), sixth Year (1964), pp., 189-196.

The specific glycamides which were manufactured in the above-described manner and used as afterrinsing agents are set out in the following listing. This listing also reflects the characteristics of the textiles which were soft-rinsed with the use of these products. For comparison, the characteristics of textiles are given which were washed and rinsed in the same manner, using no softening agents, using dioctadecyl dimethyl ammonium chloride, and using a glycamide that is not being claimed.

EXAMPLE 1

Glucamide $R_1=C_{12}H_{25}$ Feel: 2.0	$R_2=C_{17}H_{35}$	Total 29 C atoms
Compressibility $Z_q=127$ $\Delta Z_q=27$	Vertical absorbency $St_x=68\%$ $\Delta St_x=200\%$	Absorption value $S_x=73\%$ $\Delta S_y > 1700\%$

EXAMPLE 2

Glucamide $R_1=C_{11}H_{23}$ Feel: 2.0	$R_2=C_{13}H_{27}$	Total 29 C atoms
Compressibility $Z_q=122$ $\Delta Z_q=22$	Vertical Absorbency $St_x=52\%$ $\Delta St_x=140\%$	Absorption Value $S_x=36\%$ $\Delta S_y > 800\%$

EXAMPLE 3

Glucamide $R_1=C_3H_7OC_{10}H_{23}$ Feel: 1.5	$R_2=C_{13}H_{27}$	Total 34 C atoms
Compressibility $Z_q=132$ $\Delta Z_q=32$	Vertical absorbency $St_x=47\%$ $\Delta St_x=120\%$	Absorption value $S_x=23\%$ $\Delta S_y > 470\%$

EXAMPLE 4

Glucamide $R_1=C_{10}H_{21}$ Feel: 1.0	$R_2=C_{11}H_{23}$	Total 27 C atoms
Compressibility $Z_q=143$ $\Delta Z_q=43$	Vertical absorbency $St_x=30\%$ $\Delta St_x=35\%$	Absorption value Not determined because of lack of substance

EXAMPLE 5

Glucamide $R_1=C_{12}H_{25}$ Feel: 1.5	$R_2=C_7H_{15}$	Total 25 C atoms
Compressibility $Z_q=134$ $\Delta Z_q=34$	Vertical absorbency $St_x=36\%$ $\Delta St_x=61\%$	Absorption value Not determined because of lack of substance

EXAMPLE 6

Glucamide $R_1=C_{12} \cdot C_{12}$ mixture of coconut fatty acids, avg. = about C_{13} $R_2=C_{13} \cdot C_{17}$ mixture of tallow fatty acids, avg. = about C_{16} Total about 29 carbon atoms Feel: 1.0	Vertical absorbency $St_x=59\%$ $\Delta St_x=180\%$	Absorption value $S_x=67\%$ $\Delta S_y > 1500\%$
Compressibility $Z_q=143$ $\Delta Z_q=43$		

EXAMPLE 7

Glucamide $R_1=C_{12} \cdot C_{16}$ mixture of tallow fatty acids, avg. = about C_{17} $R_2=C_{11} \cdot C_{17}$ mixture of coconut fatty acids, avg. = about C_{13} Total about 30 C atoms. Feel: 1.0	Vertical absorbency $St_x=34\%$ $\Delta St_x=60\%$	Absorption value $S_x=20\%$ $S_y > 400\%$
Compressibility $Z_q=144$ $\Delta Z_q=44$		

EXAMPLE 8

Fructamide $R_1=C_{12} \cdot C_{16}$ mixture of coconut fatty acids avg. approx. C_{13} $R_2=C_{13} \cdot C_{17}$ mixture of tallow fatty acids avg. approx. C_{16} Feel: 1.0	Vertical absorbency $St_x=40\%$ $\Delta St_x=79\%$	Absorption value $S_x=23\%$ $S_y > 470\%$
Compressibility $Z_q=147$ $\Delta Z_q=47$		

EXAMPLE 9

Glycamide mixture from invert sugar $R_1=C_{12} \cdot C_{16}$ mixture of coconut fatty acids, avg. approx. C_{13} $R_2=C_{13} \cdot C_{17}$ mixture of tallow fatty acids, avg. approx. C_{16} Total approx. 29 C atoms Feel: 1.0	Vertical absorbency $St_x=46\%$ $\Delta St_x=100\%$	Absorption value $S_x=23\%$ $S_y > 490\%$
Compressibility $Z_q=155$ $\Delta Z_q=55$		

EXAMPLE 10

Glycamide mixture from invert sugar $R_1=C_{16} \cdot C_{16}$ mixture of tallow fatty acids avg. approx. C_{17} $R_2=C_{11} \cdot C_{17}$ mixture of coconut fatty acids avg. approx. C_{12} Total approx. 29 C atoms Feel: 1.0	Vertical absorbency $St_x=34\%$ $\Delta St_x=62\%$	Absorption value Not determined because of in- sufficient substance

Control Experiment A
No softening agent in the rinse water.

75 Feel: 4.0

Compressibility
 $Z_{90}=100$
 $\Delta Z_{90}=0$

Vertical absorbency
 $S_{12}=100\%$
 $\Delta S_{12}=380\%$

Absorption value
 $S_{22}=100\%$
 $\Delta S_{22}>2400\%$

Control Experiment B

Diocetadecyl dimethyl ammonium chloride

Feel: 1.0

Compressibility
 $Z_{90}=143-45$
 $\Delta Z_{90}=43-45$

Vertical absorbency
 $S_{12}=21\%$
 $\Delta S_{12}=0\%$

Absorption value
 $S_{22}<4\%$
 $\Delta S_{22}=0\%$

Control Experiment C

Glucamide $R_1=C_8H_{17}$

$R_2=C_{13}H_{27}$

Total 23 carbon atoms

Feel: 3.0

Compressibility: $Z_{90}=112$; $\Delta Z_{90}=12$

As can be seen by comparing the evaluation of the feel and the compressibility figures, all of the glycamides that come within the claimed scope exhibit a softening action which in some cases is satisfactory and in others excellent, and which is not inferior to that of the dioctyl dimethyl ammonium chloride as now used in actual practice. Further, although the vertical absorbency of the fabrics treated with this prior art product amounts to only about one-fifth of the vertical absorbencies of nonsoft-rinsed fabrics, the loss of absorbency produced by the use of the products of the invention is much lower, amounting to between one-third and two-thirds of the initial values. The same is true for the Oxé and Schuster absorbencies. The unclaimed glycamide of control experiment C is of no interest for practical use due to lack of sufficient softening action; for that reason the absorbencies were not determined.

Liquid rinsing agents of the type which have been used heretofore in conventional laundering procedures can easily be prepared by the incorporation of the claimed glycamides, i.e., by dissolving the glycamide and the optical brightener, if any, in an organic solvent and then dispersing this solution in water or in an aqueous solution of the other components. The products described in the following examples were obtained in this manner. The ethoxylated dimethylpolysiloxane as used therein, was the product as marketed by Wacker-Chemie GmbH, Munich, under the trade name DC 202.

All percentages given are percentages by weight.

EXAMPLE 11

10% glucamide according to example 6
 30% isopropanol
 2% nonyl phenyl + 9.5 ethoxy compound
 58% water

EXAMPLE 12

10% glucamide according to example 6
 30% ethyleneglycol monoethyl ether
 5% nonyl phenol + 9.5 ethoxy compound
 55% water

EXAMPLE 13

10% glucamide according to example 6
 30% isopropanol
 2% ethoxylated dimethylpolysiloxane
 10% citric acid
 48% water

EXAMPLE 14

10% glucamide according to example 6
 30% isopropanol
 2% ethoxylated dimethylpolysiloxane
 10% tartaric acid
 48% water

Example 15

10% glucamide according to example 6
 30% ethylene glycol monoethyl ether
 2% nonylphenol + 9.5 ethoxy compound

1% brightener as in formula Ia, above
 57% water

EXAMPLE 16

10% glucamide according to example 6
 30% ethylene glycol monoethyl ether
 2% nonyl phenol + 9.5 ethoxy compound
 0.5% brightener as in formula Id, above
 57.5% water

EXAMPLE 17

10% glucamide according to example 6
 20% ethylene glycol monoethyl ether
 2% nonyl phenol + 9.5 ethoxy compound
 1% brightener as in formula Ia, above
 67% water

EXAMPLE 18

10% glucamide according to example 6
 20% ethylene glycol monoethyl ether
 2% nonyl phenol + 9.5 ethoxy compound
 1% brightener as in formula Id, above
 10% polyethylene glycol 600
 57% water

EXAMPLE 19

A posttreatment agent according to the invention for use with textiles having the following composition was prepared:

10 wt.% glucamide according to example 6
 30 wt.% isopropanol
 2 wt.% ethoxylated dimethylpolysiloxane
 1 wt.% 2-bromo-2-nitro-propanediol-1,3
 10 wt.% tartaric acid
 47 wt.% water

EXAMPLE 20

A posttreatment agent for use with conventional laundry having the following composition was prepared:

10 wt.% glucamide according to example 1
 60 wt.% isopropanol
 2 wt.% ethoxylated dimethylpolysiloxane
 2 wt.% N-dodecyl-N-benzyl-N,N-dimethyl ammonium chloride
 10 wt.% tartaric acid
 16 wt.% water

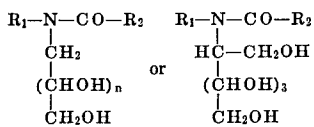
If the glycamide incorporated into the preparations of examples 11 to 19 is replaced by a compound according to examples 1-5 or 7-10, afterrinsing agents are obtained which have a similar action. Likewise, the glycamide contained in the preparation as disclosed in example 20 can be replaced with one of the compounds according to examples 2-10. The preparations of examples 11-18 can have antimicrobial agents added to them. Also, brightening agents can be incorporated, if needed, into the afterrinsing agents of examples 11-14 and 19-20. The brightening agents can be, for example, one of the compounds listed previously. In all these formulation changes, it may be advantageous, depending on the solubility of the glycamides or of the antimicrobial substance, to vary the nature and quantity of the organic solvent and/or of the emulsifying agent.

When the glycamides of the examples 1-20 were replaced by two other types, one of them containing $R_1=10$ and $R_2=19$ C atoms, the other containing $R_1=20$ and $R_2=9$ carbon atoms, similar results were obtained.

We claim:

1. A liquid composition adapted for softening of laundered textile fabrics consisting essentially of a solvent selected from the group consisting of water, a water-soluble organic solvent and a mixture of water and a water-soluble organic solvent and 2-30 wt. percent of an effective textile-fabric softening glycamide of the formula:

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wherein:

- R₁ is (1) alkyl or alkenyl of 10 to 22 carbon atoms or
- (2) alkyl or alkenyl of 10 to 22 carbon atoms with an ether oxygen linkage between either the 2 and 3 or 3 and 4 carbon atoms from the nitrogen atom,
- R₂ is alkyl or alkenyl of seven to 21 carbon atoms,
- R₁+R₂ have 23 to 39 carbon atoms, and
- n is 3 or 4,

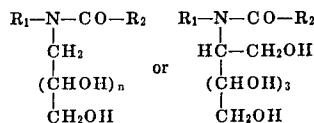
said water-soluble organic solvent being selected from the group consisting of unsubstituted aliphatic monohydric alcohols of one to four carbon atoms, ether alcohols thereof formed by the reaction of said aliphatic alcohols with a glycol selected from the group consisting of ethylene glycol, diethylene glycol, propylene glycol, and butylene glycol, lower ketones, and monoethers or diethers formed by reacting said aliphatic alcohols with glycerine.

2. The liquid composition of claim 1 consisting essentially of 5-15 wt. percent of said glycamide, wherein R₁ has 12 to 20

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carbon atoms, wherein R₂ has 11 to 19 carbon atoms, and wherein R₁+R₂ have a total of 27 to 35 carbon atoms.

3. A method for treating a textile-fabric article comprising laundering said article, and thereafter rinsing said article with water containing dissolved therein in the range of 0.05 to 2 grams per liter of a glycamide of the formula:



wherein:

- R₁ is (1) alkyl of 10 to 22 carbon atoms or
- (2) alkyl of 10 to 22 carbon atoms with an ether oxygen linkage between either the 2 and 3 or 3 and 4 carbon atoms from the nitrogen atom,
- R₂ is alkyl of seven to 21 carbon atoms,
- R₁+R₂ have 23 to 39 carbon atoms, and
- n is 3 or 4.

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