(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 062 372**
A1

(12) EUROPEAN PATENT APPLICATION

(21) Application number: **82200358.8**

(22) Date of filing: 24.03.82

(51) Int. Cl.³: **C 11 D 3/30**

(30) Priority: **02.04.81 US 250339**

(43) Date of publication of application: **13.10.82 Bulletin 82/41**

(84) Designated Contracting States: **AT BE DE FR GB IT NL**

(71) Applicant: **THE PROCTER & GAMBLE COMPANY, 301 East Sixth Street, Cincinnati Ohio 45202 (US)**

(72) Inventor: **Dzialo nee Brown, Kathleen Jeannette, 3445 Cster Drive, Cincinnati Ohio 45208 (US)**

(74) Representative: **Gibson, Tony Nicholas et al, Procter & Gamble (NTC) Limited Whitley Road, Longbenton Newcastle upon Tyne NE12 9TS (GB)**

(54) Fabric softening compositions.

(57) Laundry detergent compositions are provided which contain an effective textile softening agent for through the wash softening. The softening agent comprises a mixture of two specified classes of tertiary amines.

EP 0 062 372 A1

FABRIC SOFTENING COMPOSITIONS

Kathleen J. Brown

TECHNICAL FIELD

This invention relates to solid detergent compositions which clean well in cool water and at the same time provide softening to the laundered fabrics in a home laundering operation.

It is common practice for homemakers to provide a softened quality to laundered fabrics by treating laundered fabrics with a softening composition during the rinse cycle to deposit the softening agent on the fabric. By providing a detergent composition which can at the same time provide a softening quality to the fabrics while being washed makes the home laundering operation more convenient.

BACKGROUND

Numerous attempts have been made to formulate laundry detergent compositions which provide the good cleaning performance expected of them and which also have textile softening properties. Thus, attempts have been made to incorporate cationic textile softeners in anionic surfactant-based built detergent compositions employing various means of overcoming the natural antagonism between the anionic and cationic surfactant species. For instance, in British patent specification 1,518,529, detergent compositions are described comprising organic surfactant, builders, and in particulate form, a quaternary ammonium softener combined with a poorly water-soluble dispersion inhibitor which inhibits

premature dispersion of the cationic in the wash liquor. Even in these compositions, some compromise between cleaning and softening effectiveness has to be accepted. Another approach to providing anionic detergent compositions with textile softening ability has been the use of smectite-type clays, as described in British patent specificaiton 1,400,898. These compositions, although they clean well, require rather large contents of clay for effective softening, perhaps because the clay is not very efficiently deposited on the fabrics in the presence of anionic surfactants. Yet another approach to providing built detergent compositions with softening ability has been to employ nonionic surfactants instead of anionic with cationic softeners, and compositions of this type have been described in, for example, British patent specification 1,079,388, German Auslegeschrift 1,220,956 and US patent 3,607,763. However, it is found that if enough nonionic surfactant is employed to provide good cleaning, it impairs the softening effect of the cationic softener, so that, once again, a compromise between cleaning and softening effectiveness must be accepted.

The use of clay together with a water insoluble cationic compound and an electrically conductive metal salt as a softening composition adapted for use with anionic, nonionic, zwitterionic and amphoteric surfactants has been described in British patent specification 1,483,627. U.S. Patent No. 4,292,035 which issued on September 29, 1981 to

Charles F. Battrell

describes granular textile softening compositions comprising a complex of a

cationic softener and a smectite-type subsequently treated with an anionic surfactant. These compositions are intended primarily as rinse additives, where their cleaning performance is not of primary interest.

Recently it has been disclosed in British Patent specification 1,514,276 that certain tertiary amines with two long chain alkyl or alkenyl groups and one short chain alkyl group are effective fabric softeners in detergent compositions when chosen to have an isoelectric point in the pH range such that they are in anionic form in a normal alkaline wash liquor and are more in cationic form at the lower pH of a rinse liquor, and so become substantive to fabrics. Use of amines of this class, amongst others, in detergent compositions for foam control has also been previously disclosed in British patent specification 1,286,054. Use of amines of the class specified in B.P. 1,286,054 together with a smectite-type clay for laundry detergent compositions providing through-the-wash softening is claimed in European Application No. 79200656.1 Publication No. 0011340.

It has now been found that the combination of a di-long chain (C_{16}-C_{22}) tertiary amine with a di-shorter chain (C_8-C_{14}) tertiary amine in specific ratios provides improved softening compared to either tertiary amine alone.

SUMMARY OF THE INVENTION

According to the present invention there is provided a fabric softening detergent composition consisting

essentially of, by weight:

(a) from 3% to 40% of an organic surfactant;

(b) from 1% to 25% of a tertiary amine fabric softening agent containing two C_8 to C_{22} alkyl or alkenyl groups and a C_1 to C_4 alkyl group, attached to the nitrogen atom; and

(c) from 10% to 80% of one or more water soluble salts,

wherein the tertiary amine fabric softening agent is a mixture comprising 10-50% by weight of a tertiary amine containing two C_8-C_{14} alkyl or alkenyl groups and 90-50% by weight of a tertiary amine containing two C_{16}-C_{22} alkyl or alkenyl groups, wherein the pH of a 0.5% by weight aqueous solution of the composition is in the range of from 8.5 to 11.

DETAILED DESCRIPTION OF THE INVENTION

Organic Surfactant

The compositions of this invention contain from 3% to 40%, preferably from 5% to 25%, most preferably from 10% to 20%, of an organic surfactant selected from anionic, nonionic, cationic, amphoteric, and zwitterionic surfactants, and mixtures thereof as disclosed in U. S. Patent No. 3,929,678.

Anionic surfactants are much preferred for optimum combined

cleaning and textile softening performance but the other classes of organic surfactants and mixtures thereof may be used. When anionic surfactants are employed it is preferred that nonionic and other classes of surfactant be absent but, if mixtures containing anionics are used, it is preferred that the anionic surfactant forms the major part of the mixture.

Suitable anionic non-soap surfactants are water soluble salts of alkyl benzene sulfonates, alkyl toluene sulfonates, alkyl sulfates, alkyl polyethoxy ether sulfates, paraffin sulfonates, alpha-olefin sulfonates, alpha-sulfocarboxylates and their esters, alkyl glyceryl ether sulfonates, fatty acid monoglyceride sulfates and sulfonates, alkyl phenol polyethoxy ether sulfates, 2-acyloxy-alkane-1-sulfonates, and beta-alkyloxy alkane sulfonates. Soaps are also suitable anionic surfactants.

Especially preferred alkyl benzene sulfonates have 9 to 15 carbon atoms in a linear or branched alkyl chain, more especially 11 to 13 carbon atoms (especially valuable are linear straight chain alkyl benzene sulfonates in which the average of the alkyl groups is 11.8 carbon atoms and commonly abbreviated as $C_{11.8}$LAS). Suitable alkyl sulfates have 10 to 22 carbon atoms in the alkyl chain, more especially from 12 to 18 carbon atoms. Suitable alkyl polyethoxy ether sulfates have 9 to 18 carbon atoms in the alkyl chain and have an average of 1 to 12 $-CH_2CH_2O-$ groups abbr. as EO) per molecule, especially 9 to 16 carbon atoms in the alkyl chain and an average of 1 to 8 $-CH_2CH_2O-$ groups per molecule.

Suitable paraffin sulfonates are essentially linear and contain from 8 to 24 carbon atoms, more especially from 14 to 18 carbon atoms. Suitable alpha-olefin sulfonates have 10 to 24 carbon atoms, more especially 14 to 16 carbon atoms; alpha-olefin sulfonates can be made by reaction with sulfur trioxide followed by neutralization under conditions such that any sultones present are hydrolyzed to the corresponding hydroxy alkane sulfonates. Suitable alpha-sulfocarboxylates contain from 6 to 20 carbon atoms; included herein are not only the salts of alpha-sulfonated fatty acids but also their esters made from alcohols containing 1 to 14 carbon atoms.

Suitable alkyl glyceryl ether sulfates are ethers of alcohols having 10 to 18 carbon atoms, more especially those derived from coconut oil and tallow. Suitable alkyl phenol polyethoxy ether sulfates have 8 to 12 carbon atoms in the alkyl chain and an average of about 1 to about 6-CH_2CH_2O- groups per molecule. Suitable 2-acyloxy-alkane-1- sulfonates contain from 2 to 9 carbon atoms in the acyl group and 9 to 23 carbon atoms in the alkane moiety. Suitable beta-alkyloxy alkane sulfonates contain 1 to 3 carbon atoms in the alkyl group and 8 to 20 carbon atoms in the alkane moiety.

The alkyl chains of the foregoing non-soap anionic surfactants can be derived from natural sources such as coconut oil or tallow, or can be made synthetically as for example using the Ziegler or Oxo processes. Water solubility can be achieved

by using alkali metal, ammonium, or alkanol-ammonium cations; sodium is preferred. Mixtures of anionic surfactants are contemplated by this invention; a satisfactory mixture contains alkyl benzene sulfonate having 11 to 13 carbon atoms in the alkyl group and alkyl sulfate having 12 to 18 carbon atoms in the alkyl group.

Suitable soaps contain 8 to 24 carbon atoms, more especially 12 to 18 carbon atoms. Soaps can be made by direct saponification of natural fats and oils such as coconut oil, tallow and fish oil, or by the neutralization of free fatty acids obtained from either natural or synthetic sources. The soap cation can be alkali metal, ammonium or alkanolammonium; sodium is preferred.

The Organic Fabric Softening Agent

The second essential ingredient of this invention is a mixture of water insoluble tertiary amines which comprises from 1% to 25% of the composition. Preferred compositions contain from 2% to 15% of the mixture and most preferred compositions contain from 4% to 12% of the mixture.

The mixture of water insoluble tertiary amines comprises from 90% to 50%, preferably from 80% to 60%, and most preferably from 75% to 65%, of a tertiary amine having the formula $R_1R_2R_3N$ wherein R_1 and R_2 are independently selected from C_{16} to C_{22} alkyl or alkenyl groups and R_3 represents a C_1 to C_4 alkyl group, and from 10% to 50% of a tertiary amine having the formula $R_4R_5R_6N$ wherein R_4 and R_5 are independently selected from C_8 to C_{14} alkyl or alkylene groups, and R_6 represents a C_1 to C_4 alkyl group.

Suitable tertiary amines wherein R_1 and R_2 are selected from C_{16} to C_{22} groups include dicetyl methylamine, distearyl methylamine, diarachidyl methylamine, dibehenyl methylamine, di(mixed arachidyl/behenyl) methylamine, di(tallowyl) methylamine, and the corresponding ethylamines, propylamines and butylamines. Especially preferred is di-hydrogenated tallowyl methylamine. This is commercially available as Kemamine® T9701 from Humko Sheffield Chemical, a division of Kraft Inc.

Suitable tertiary amines wherein R_4 and R_5 are selected from C_8 to C_{14} groups include dioctyl methylamine, didecyl methylamine, dilauryl methylamine, dimyristyl methylamine, di(mixed lauryl/myristyl) methylamine, dicoconut methylamine, and the corresponding ethylamines, propylamines, and butylamines. Especially preferred is dicoconut methylamine. This is commercially available as Kemamine® T6501.

It was unexpected that the shorter chain length tertiary amines disclosed herein, which are liquid and have no particular value as fabric softening agents, improve cool water fabric softening through-the-wash when included with the longer chain length tertiary amines disclosed herein in a detergent composition.

Water-soluble Salts

The compositions of the invention contain from 10% to 80% of water soluble salts, preferably from 20% to 70%, and most usually from 30% to 60%, and these may be any which are such that the detergent composition in a 0.5% by weight aqueous solution has pH in the specified range, that is from 8.5 to 11, preferably from 9.5 to 10.5. At this pH the tertiary amines of the invention are in anionic form and

are therefore compatible with anionic surfactants.

Preferably the water soluble salts are detergency builders and these can be of the polyvalent inorganic and polyvalent organic types, or mixtures thereof. Non-limiting examples of suitable water-soluble, inorganic alkaline detergent builder salts include the alkali metal carbonates, borates, phosphates, polyphosphates, tripolyphosphates, bicarbonates, and silicates. Specific examples of such salts include the sodium and potassium tetraborates, bicarbonates, carbonates, tripolyphosphates, pyrophosphates, orthophosphates, pentapolyphosphates and hexametaphosphates. Sulfates are usually also present.

Examples of suitable organic alkaline detergency builders salts are:

(1) water-soluble amino polyacetates, e.g., sodium and potassium ethylenediaminetetraacetates, nitrilotriacetates, N-(2-Hydroxylethyl) nitrilodiacetates and diethylenetriamine pentaacetates;

(2) water-soluble salts of phytic acid, e.g. sodium and potassium phytates;

(3) water-soluble polyphosphonates, including sodium, potassium and lithium salts of methylenediphosphonic acid and the like and aminopolymethylene phosphonates such as ethyldiaminetetramethylenephosphonate and diethylenetriaminepentamethylene phosphonate, and polyphosphonates as described in the commonly assigned German Application DOS 2816770.

(4) water-soluble polycarboxylates such as the salts of lactic acid, succinic acid, malonic acid, maleic acid, citric acid,

carboxymethylsuccinic acid, 2-oxa-1,1, 3-propane tricarboxylic acid, 1,1,2-2-ethane tetracarboxylic acid, cyclopentane-cis, cis, cis - tetracarboxylic acid, mellitic acid and pyromellitic acid.

Mixtures of organic and/or inorganic builders can be used herein. One such mixture of builders is disclosed in Canadian Patent No. 755,038, e.g. a ternary mixture of sodium tripolyphosphate, trisodium nitrilotriacetate, and trisodium ethane-1-hydroxy-1, 1-diphosphonate.

Another type of detergency builder material useful in the present compositions and processes comprises a water-soluble material capable of forming a water-insoluble reaction production with water hardness cations prefereably in combination with a crystallization seed which is capable of providing growth sites for said reaction product. Such "seeded builder" compositions are fully disclosed in British Patent Specification No. 1,424,406.

Preferred water soluble builders are sodium tripolyphosphate and sodium silicate, and usually both are present. In particular it is preferred that a substantial proportion, for instance from 3 to 15% by weight of the composition of sodium silicate (solids) of ratio (weight ratio $SiO_2:Na_2O$) from 1:1 to 3.5:1 be employed.

A further class of detergency builder materials useful in the present invention are insoluble sodium aluminosilicates, particularly those described in Belgian Patent 814,874, issued November 12, 1974. This patent discloses and claims detergent compositions containing sodium aluminosilicates of the formula

$$Na_z(AlO_2)_z(SiO_2)_y xH_2O$$

wherein z and y are integers equal to at least 6, the molar ratio of z to y is in the range of from 1.0:1 to about 0.5:1 and x is an integer from about 15 to about 264. A preferred material is $Na_{12}(SiO_2AlO_2)_{12}$ $27H_2O$.

Preferably, the compositions contain from 20% to 70% of builders, more usually 30% to 60% by weight. If present, incorporation of about 5% to about 25% by weight of aluminosilicate is suitable, partially replacing water soluble builder salts, provided that sufficient water soluble alkaline salts remain to provide the specified pH of the composition in aqueous solution.

Optional Components

A valuable optional component of the present compositions consists of particular smectite clay materials, namely the alkali metal and certain alkaline earth metal varieties of montmorillonites, saponites, and hectorites. These materials are also useful in providing softening and antistatic benefits to fabrics. These materials are described in U. S. Patent 3,936,537.

The suitable smectite clays can be included at levels of from 1% to 25%, preferably from 2% to 20%, and most preferably from 3% to 10%, of the composition.

The suitable clay materials can be described as impalpable, expendable, three-layer clays, in which a sheet of aluminum/oxygen atoms or magnesium oxygen atoms lies between two layers of silicon/oxygen atoms, i.e., alumino-silicates and magnesium silicates, having an ion exchange capacity of at least 50 meq./100g., preferably at least 60 meq./100g., of clay. The term "impalpable" as used

herein means that the individual clay particles are of such a size that they cannot be perceived tactilely. Such particle sizes are within the range below about 100 microns in effective diameter. In general, the clays herein have an ultimate particle size within the range from 1 micron to 50 microns. The term "expandable" as used to describe clays relates to the ability of the layered clay structures to be swollen, or expanded on contact with water. A further property of the suitable clay materials used herein is that they exhibit a true 14Å X-ray diffraction pattern.

Specific non-limiting examples of such fabric softening smectite clay minerals are: sodium montmorillonite sold under the trade names Brock, Volclay BC, Gelwhite GP, Thixo-Jel No. 1, Ben-A-Gel; sodium hectorite sold under the trade names Veegum F and Laponite SP; sodium saponite sold under the tradename Barasym NAS100; calcium montmorillonite sold under the tradenames Soft Clark and Gelwhite L; and lithium hectorite sold under the tradename Barasym LIH200.

Most of the smectite clays useful in the compositions herein are commercially available under various tradenames, for example, Brock, Gelwhite GP and Thixo-Jel No. 1 from Georgia Kaolin Co., Elizabeth, New Jersey; Volclay BC and Volclay No. 325, from American Colloid Co., Skokie, Illinois; and Veegum F from R. T. Vanderbilt. It is to be recognized that such smectite minerals obtained under the foregoing tradenames can comprise mixtures of the various discrete mineral entities. Such mixtures are suitable for use herein.

Additional suitable optional fabric softening/anti-static agents in this invention are the conventionally water-insoluble quaternary ammonium compounds of the formula $R_1R_2R_3R_4N^+Y^-$ wherein R_1 and R_2 represent hydrocarbyl groups of from 10 to 22 carbon atoms, R_3 and R_4 represent hydrocarbyl groups containing from 1 to 4 carbon atoms, and Y represents an anion, (e.g., fluoride, chloride, bromide, or methylsulfate). Examples of these compounds include dioctadecyldimethylammonium chloride, ditallowdimethylammonium chloride, ditallowdiethylammonium bromide, cetyldecylmethylethylammonium chloride, bis-docosyldimethylammonium chloride, and the like. Also suitable are the single long chained quaternary ammonium compounds of the above formula wherein R_1 is C_{10} to C_{22} alkyl or alkenyl, preferably C_{16} to C_{20} alkyl, and R_2, R_3, and R_4 are C_1 to C_4 alkyl groups, or acyl groups such as benzyl, and Y is defined as above.

Yet other quaternary ammonium compounds are those having a single long chain wherein R_1 is a C_{10} to C_{22} alkyl group, R_2 is a C_1 to C_4 alkyl group or hydrogen, R_3 is $-(C_2H_4O)_xH$, and R_4 is $-(C_2H_4O)_yH$ wherein x and y are at least 1 and (x+y) is from 2 to 25. Examples are $(C_{18}H_{37})(CH_3)[(C_2H_5O)_8H][(C_2H_5O)_7H]N^+Cl^-$, $(C_{20}H_{41})(H)[(C_2H_5O)_5H][(C_2H_5O)_5)]N^+Cl^-$, and the like. Substances of this sort are sold under the trade name "Ethoquads."

Compositions of this invention can contain up to 10%, preferably from 0.5% to 5% of the above quaternary ammonium compounds.

The optional components usual in built laundry detergents may of course be present. These include bleaching agents such as sodium perborate, sodium percarbonate and other perhydrates, at levels from 5% to 35% by weight of the composition, and activators therefor, such as tetra acetyl ethylene diamine, tetra acetyl glycouril and others known in the art, and stabilisers therefor, such as magnesium silicate, and ethylene diamine tetra acetate.

A usual optional component is a processing aid, especially for the anionic surfactant. Such components include from 0.1% to 9%, preferably 0.5% to 3%, of sodium toluene sulfonate and/or sodium xylene sulfonate.

Suds controlling agents are often present. These include suds boosting or suds stabilising agents such as mono- or di-ethanolamides of fatty acids. More often in modern detergent compositions, suds suppressing agents are required. Soaps especially those having 16-22 carbon atoms, or the corresponding fatty acid, can act as effective suds suppressors if included in the anionic surfactant component of the present compositions. Usually 1% to 4% of such soap is effective as suds suppressor. Very suitable soaps when suds suppression is a primary reason for their use, are those derived from Hyfac (Trade name for hardened marine oil fatty acids predominantly C_{18} to C_{20}).

However, non-soap suds suppressors are preferred in synthetic detergent based compositions of the invention since soap or fatty acid is less effective in cool water washing and tends to give rise to a characteristic odour in these compositions.

Preferred suds suppressors comprise silicones. In particular there may be employed a particulate suds

suppressor comprising silicone and silianated silica releasably enclosed in water soluble or dispersible substantially non-surface active detergent impermeable carrier. Suds suppressing agents of this sort are disclosed in British patent specification 1,407,997. A very suitable granular (prilled) suds suppressing product comprises 7% silica/silicone (85% by weight silanated silica, 15% silicone, obtained from Messrs. Dow Corning), 65% sodium tripolyphosphate, 25% Tallow alcohol condensed with 25 molar proportions of ethylene oxide, and 3% moisture. The amount of silica/silicone suds suppressor employed depends upon the degree of suds suppression desired but is often in the range from 0.01% to 0.5% by weight of the detergent composition. Other suds suppressors which may be used are water insoluble, preferably microcrystalline, waxes having melting point in the range from 35 to 120°C and saponification value less than 100, as described in British patent specification 1,492,938.

Yet other suitable suds suppressing systems are mixtures of hydrocarbon oil, a hydrocarbon wax and hydrophobic silica as described in European laid open patent application No. 0000216 published January 10, 1979 and, especially, particulate suds suppressing compositions comprising such mixtures, combined with a nonionic ethoxylate having hydrophilic lipophilic balance in the range from 14-19 and a compatibilising agent capable of forming inclusion compounds, such as urea. These particulate suds suppressing compositions are described in European patent application 79200472.3 Publication No. 0008830.

Soil suspending agents are usually present at 0.1 to 10%, such as water soluble salts of carboxymethylcellulose, carboxyhydroxymethyl

cellulose, polyethylene glycols of molecular weight from 300 to 10000, polyacrylic acid, hydroxybutyl methyl cellulose and derivatives thereof, and copolymers of methylvinylether and maleic anhydride or acid, available from the General Aniline and Film Corporation under the Trade Name Gantrez.

Optical brighteners, which can be anionic, cationic, or nonionic types, are usually present at 0.01 to 1%. Especially suitable brighteners include the derivatives of sulfonated triazinyl diamino stilbene such as 4,4^1-bis[(6-anilino-4-morpholino-1.3.5-triazin-2-yl)amino] stilbene -2,2^1-disulfonate and the diphenyl type such as disodium 4,4^1-bis(2 sulfostyryl) diphenyl. These brighteners tend to cause yellowing of granular products, especially on aging, and it has been found useful to incorporate into the composition from 0.1% to 7% of polyethylene glycol of molecular weight of 300 to 1500 to prevent such yellowing.

Proteolytic, amylolytic or lipolytic enzymes, especially proteolytic may be present. A further useful additive is a photo activated bleach comprising a mixture of the tri and tetra sulphonated derivatives of zinc phthalocyanine as described in B.P. Specification Nos. 1372035 and 1408144.

Through the description herein, where sodium salts have been referred to, potassium, lithium or ammonium or amine salts may be used instead if their extra cost etc. are justified for special reasons.

Preparation of the Compositions

The detergent compositions may be prepared in any way, as appropriate to their physical form, as by mixing the components, co-agglomerating them or dispersing them in a liquid carrier. Preferably the compositions are granular and are prepared by spray drying an aqueous slurry of the non-heat-sensitive components to form spray dried granules into which may be admixed the heat sensitive components such as persalts, enzymes, perfumes, etc. Although the tertiary and quaternary amines are preferably included in the slurry for spray drying, they can be incorporated by being sprayed in liquid form on the spray dried granules before or after other heat sensitive solids have been dry mixed with them. The granules so made are surprisingly crisp and free flowing even though the amines are a combination of a waxy solid of low melting point and a liquid. Alternatively the amines in liquid form may be sprayed onto any particulate component or components of the composition which are able to act as carrier granules. The clay component may be added to the slurry for spray drying or may be dry mixed, as preferred for reasons unrelated to its softening effect, such as for optimum colour of the product.

EXAMPLE I

The fabric softening performance of a composition containing tertiary amines of this invention was determined by washing test terry towels (84% cotton/16% polyester) in a top-loading semiautomatic machine with the agitation provided by an impeller with reversing agitation and an extractor adjacent to the wash tub. The washing was done in 21°C water whose hardness was 0.79 grains (as $CaCO_3$)/liter

(3 gr./U.S. gal.) with the Ca/Mg ratio being 3/1. The product concentration was 0.15%, the water capacity of the wash was 30 liters and the water: cloth ratio was 30:1 and the cloth load was 1kg. and consisted of 8 test terry towels (each 12 x 18 inches and weighing about 50 g.) with the remainder of the load being soiled T-shirts. The fabric load was washed for 10 minutes, extracted for 2 minutes and then rinsed, extracted again and line dried. The test towels were then graded for softness in a round-robin panel test using 3 experienced judges who were not advised of the test details. Included in the test were a set of test towels wherein no organic fabric softener was present in the wash. The scale used by the judges was a 4 point scale wherein the scale had the following meaning: 0 - no difference; 1 - guess that there is a difference; 2 - small difference; 3 - modest difference; 4 - large difference. A statistically significant difference between treatments at the 95% confidence level is 0.5 panel score units.

The composition of the product was as follows:

Sodium $C_{11.8}$LAS	15.0%
Silicate solids ($SiO2:Na_2O=2.4$)	10.0
Sodium carbonate (dry-added)	10.0
Clay-calcium montmorillonite	6.0
Sodium pyrophosphate	15.1
Organic fabric softener*	5.75
Optical brightener	0.10
PEG 600 (added in slurry)	0.40
PEG 6000 (dry-added)	0.85
C_{12} amine oxide (spray-on)	0.25
H_2O	6.0
Sodium sulfate	to 100

*The organic fabric softener consisted of ditallow-methylamine, dicoconutmethylamine and mixtures thereof with the level in the product being constant.

The results of the test are summarized in Table I.

TABLE I

Composition No.	1	2	3	4	5	6	7
% Ditallow-methylamine	100	90	80	67	50	33	0
% Dicoconut-methylamine	0	10	20	33	50	67	100
Softness panel score	+0.6	+0.75	+0.9	+1.3	+0.7	+0.6	+0.2

As shown by the data the mixtures of tertiary amines surprisingly provided more softening than either amine alone with the best results observed when the ratio of ditallowmethylamine to dicoconutmethylamine being about 2:1.

EXAMPLE II

The following compositions are prepared:

	8	9	10	11
Sodium C_{12}LAS	15.0%	20.0%	18.0%	20.0%
Sodium orthophosphate	5	-	-	-
Sodium pyrophosphate	10	15	10	20
Sodium tripolyphosphate	5	5	10	-
Sodium toluenesulfonate	5	5	5	5
Sodium Silicate	12	12	12	12
Sodium Carbonate	10	10	15	10
Ditallow methylamine	8	14	6	2
Dicoconut methylamine	4	7	3	1
DTDMAC*	-	-	1	5
Clay-Ca montmorillonite	6	6	6	6
PEG-600	0.4	0.5	0.5	0.5
PEG-6000	0.9	1.0	1.0	1.0
Optical brightener	0.1	0.2	0.2	0.2
Moisture	6	6	6	6
Sodium Sulfate	to 100% ⟶			

*Ditallowdimethylammonium chloride

EXAMPLE III

The following compositions are prepared.

	12	13	14	15	16
Sodium C_{12}LAS	10%	18%	15%	25%	20%
Sodium toluene sulfonate	2	2	3	4	2
Sodium tallow alkyl sulfate	5	-	-	-	5
Sodium C_{12-13} alkyl $(EO)_3$ sulfate	3	-	5	-	5
Sodium pyrophosphate	12	18	8	10	8
Sodium tripoly-phosphate	5	-	8	-	8
Sodium silicate	10	12	10	10	12
Sodium carbonate	10	15	10	10	15
Sodium aluminosilicate	-	5	10	15	25
Dilauryl methylamine	7	-	3	-	2
Dimyristyl methylamine	-	7	-	3	2
Distearyl methylamine	-	14	6	-	4
Dibehenic methylamine	14	-	-	6	4
Clay-Ca montmorillo-nite	-	6	10	10	6
PEG 600	0.5	0.5	0.5	0.5	0.5
PEG 6000	1.0	1.0	1.0	1.0	1.0
Optical brightener	0.2	0.2	0.2	0.2	0.2
Moisture	6	6	6	6	6
Sodium sulfate	to 100 →				

CLAIMS

1. A fabric softening detergent composition consisting essentially of, by weight:

(a) from 3% to 40% of an organic surfactant;

(b) from 1% to 25% of a tertiary amine fabric softening agent containing two C_8 to C_{22} alkyl or alkenyl groups and a C_1 to C_4 alkyl group, attached to the nitrogen atom; and

(c) from 10% to 80% of one or more water soluble salts,

characterised in that the tertiary amine fabric softening agent is a mixture comprising 10-50% by weight of a tertiary amine containing two C_8-C_{14} alkyl or alkenyl groups and 90-50% by weight of a tertiary amine containing two C_{16}-C_{22} alkyl or alkenyl groups, wherein the pH of a 0.5% by weight aqueous solution of the composition is in the range of from 8.5 to 11.

2. A composition according to Claim 1 which contains from 2% to 15% of a mixture of tertiary amines wherein from 80% to 60% of the mixture is a tertiary amine selected from dicetyl methylamine, distearyl methylamine, dibehenyl methylamine, diarchidyl methylamine, ditallow methylamine, and mixtures thereof, and from 20% to 40% of a tertiary amine selected from doctyl methylamine, didecyl methylamine, dilauryl methylamine, dimyristyl methylamine, dicoconut methylamine, and mixtures thereof.

3. A composition according to Claim 2 which contains from 4% to 12% of a mixture of tertiary amines wherein from 75% to 65% of the mixture is ditallow methylamine, and from 35% to 25% of the mixture is dicoconut methylamine.

4. A composition according to any one of Claims 1-3 which additionally contains up to 10% of a water insoluble quaternary ammonium softener having the formula $R_1R_2R_3R_4N^+Y^-$ wherein R_1 and preferably R_2 represent an organic radical containing a group selected from a C_{16}-C_{22} aliphatic radical or an alkyl phenyl or alkyl benzyl radical having from 10 to 16 carbon atoms in the alkyl chain, R_3 and R_4 represent hydrocarbyl groups containing from 1 to 4 carbon atoms and Y^- represents an anion.

European Patent Office

EUROPEAN SEARCH REPORT

0062372
Application number

EP 82 20 0358.8

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim
D,A	EP - A1 - 0 011 340 (PROCTER & GAMBLE)	
A	DE - A1 - 2 646 995 (UNILEVER N.V.) * claims 1 to 5 *	
A	EP - A1 - 0 013 450 (PROCTER & GAMBLE) * page 32, example 5 *	
A	EP - A1 - 0 023 367 (PROCTER & GAMBLE) * claims 1 to 4 *	

CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)

C 11 D 3/30

TECHNICAL FIELDS SEARCHED (Int.Cl. 3)

C 11 D 3/00

CATEGORY OF CITED DOCUMENTS

X: particularly relevant if taken alone
Y: particularly relevant if combined with another document of the same category
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention
E: earlier patent document, but published on, or after the filing date
D: document cited in the application
L: document cited for other reasons

&: member of the same patent family, corresponding document

X The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Berlin	12-05-1982	SCHULTZE

EPO Form 1503.1 06.78