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TEXTILE SOFTENER COMPOSITIONS

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13 Claims

Textile softener composition compatible with anionic detergents containing (1) 50–90 parts by wt. of an acid salt of a condensation product of a fatty derivative having 8–22 carbon atoms selected from fatty acids, fatty halides and lower alkyl esters of fatty acids and an aliphatic polyfunctional amine selected from lower alkanol amines and polyalkylene polyamines and (2) 10–50 parts of a particular type of polyoxyalkylene compound. The softener composition may be used in the wash cycle of a laundering operation along with conventional built detergents.

Textile softener compositions are utilized in the textile industry to give the fiber or fabric a better handle or feel and a better drape. They quite often also act as lubricants and antistatic agents. In the manufacture of textiles, after completion of the various process steps the fabric is finished by various treatments which often include the application of a softener. These finishes, particularly softeners, are removed by washing and recently a number of softeners for use in home and industrial washing machines have been placed on the market, which softeners restore to the washed goods the original feel and drape properties.

These "home laundry" softeners are recommended for use in the washing machine after the wash period and usually after at least one or more rinse periods. These softeners are ordinarily referred to as "after-rinse" softeners.

The after-rinse softeners are usually formulated into 4–8% active, liquid products and bottled for the "home laundry" use. To date, these products have been largely based on the dimethyl-di(hydrogenated tallow)-ammonium chloride type fabric softeners. Although these products are excellent for improving the handle of fibers and fabrics, they are incompatible with anionic detergents and have the tendency to build up on successive washes resulting in reduced absorbency of the washed and treated fabrics. In addition, these products are not always compatible with optical whiteners, and are also not compatible with germicidal compounds, even cationic germicidal compounds such as n-alkyl-dimethyl-aryl-ammonium chlorides. In the latter case a gelling effect takes place in this system and the product is no longer pourable from a bottle.

An object of the present invention is the obtention of a softener composition which is compatible with anionic washing detergents comprising (1) from 50 to 90 parts by weight of an acid salt of a water-miscible acid selected from the group consisting of inorganic acid and aliphatic carboxylic acid having from 2 to 6 carbon atoms, with a condensation product of an excess of an aliphatic fatty acid derivative having from 8 to 22 carbon atoms selected from the group consisting of acids, lower alkyl esters and acid halides, with an aliphatic polyfunctional amine having at least two functional groups selected from the group consisting of hydroxyl and amino and (2) from 10 to 50 parts by weight of a lower alkoxyated compound selected from the group consisting of alcohols and amines.

Another object of the present invention is the obtention of a detergent and softener composition comprising (A) from about 90 to about 99 parts by weight of a commercial built detergent containing sulfonates, alkalis, alkali metal phosphates and sodium sulfate, and (B) from about 1 to about 10 parts by weight of a softener com-

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position comprising (1) from 50 to 90 parts by weight of an acid salt of a water-miscible acid selected from the group consisting of inorganic acid and aliphatic carboxylic acid having from 2 to 6 carbon atoms, with a condensation product of an excess of an aliphatic fatty acid derivative having from 8 to 22 carbon atoms selected from the group consisting of acids, lower alkyl esters and acid halides, with an aliphatic polyfunctional amine having at least two functional groups selected from the group consisting of hydroxyl and amino and (2) from 10 to 50 parts by weight of a lower alkoxyated compound selected from the group consisting of alcohols and amines.

A yet further object of the present invention is the development of a process for washing and softening textiles in a machine washer which comprises filling said washer with water, adding textiles to said water, adding a commercial built detergent containing sulfonates, alkalis, alkali metal polyphosphates, and sodium sulfate, adding from about 500 to 1000 parts per million based on said textiles of a softener composition comprising (1) from 50 to 90 parts by weight of an acid salt of a water-miscible acid selected from the group consisting of inorganic acid and aliphatic carboxylic acid having from 2 to 6 carbon atoms, with a condensation product of an excess of an aliphatic fatty acid derivative having from 8 to 22 carbon atoms selected from the group consisting of acids, lower alkyl esters and acid halides, with an aliphatic polyfunctional amine having at least two functional groups selected from the group consisting of hydroxyl and amino and (2) from 10 to 50 parts by weight of a lower alkoxyated compound selected from the group consisting of alcohols and amines, washing said textiles, rinsing said textiles, and recovering said washed and softened textiles.

A still further object of the present invention is the development of a process for the preparation of a softener composition which is compatible with anionic washing detergents which comprises the steps of reacting a molar excess of an aliphatic fatty acid derivative having from 8 to 22 carbon atoms selected from the group consisting of acids, lower alkyl esters and acid halides with about one mol of an aliphatic polyfunctional amine having at least two functional groups selected from the group consisting of hydroxyl and amino at a temperature of from 100° C. to the decomposition temperature for a time sufficient to remove the reaction-formed water, adding from about 10 to 50 parts by weight of a lower alkoxyated compound selected from the group consisting of alcohols and amines to the molten condensation product, adding a sufficient amount of a water-miscible acid selected from the group consisting of inorganic acid and aliphatic carboxylic acid having from 2 to 6 carbon atoms to neutralize said molten condensation product, cooling the reaction mass and recovering said softener composition.

These and other objects of the invention will become more apparent as the description thereof proceeds.

We have now discovered softener compositions which have been found particularly suitable for fabric softeners. Our novel softener compositions can be utilized in the following manner:

- (1) As a softener during the wash cycle
- (2) As an after-rinse softener
- (3) In built-in detergents for heavy-duty cleaning that can be added:
 - (a) to the slurry before spray drying
 - (b) to heavy-duty liquids.

For the above uses a softener composition must perform as follows:

- (1) The softener must be substantive,
- (2) The softener must provide a good hand and fluffiness to the fabric,

- (3) The softener must have non-yellowing properties, and
 (4) The softener must be effective and stable in a pH range of 10 to 11.

Thus, the object of this invention is to provide compositions for treating various fibers that will not only improve the handle of the fiber or fabric, but are versatile enough that they can be applied as previously mentioned, as a softener during the wash cycle, as an after-rinse softener, and at the same time when reformulated, be compatible with additives such as optical whiteners, germicidal compounds, etc. In addition these compositions of the invention minimize the water-repellent effect encountered by the dimethyl dihydrogenated tallow ammonium chloride based products, and in most cases actually have no detrimental effect on the absorbency of the treated fabric.

In addition, these compositions, because of their uniqueness, can also be incorporated or added to built-in detergents for heavy-duty cleaning. The softener can either be added to the detergent slurry before spray drying or directly to heavy duty liquids. In this case it is necessary as previously mentioned, that the softener be compatible in an anionic system and stable and effective in some cases in a pH range of 10 to 11.

These built-in detergents with the softener component enable the housewife to wash and soften in one operation. In other words, in a single addition of built-in detergent with softener, the goods can now be scoured and, since the softener is substantive, softened in one cycle, thus eliminating the two-step operation in the housewife having to add detergent in the wash cycle and the softener in the after-rinse cycle.

In these anionic type built-in detergent systems the dimethyl dihydrogenated tallow ammonium chloride base softeners are not applicable. Being cationic in chemical nature and opposite in electronic charge they are not compatible with anionic materials.

The products of our invention are unique in the respect that although cationic in nature they are compatible with anionic built-in detergents containing, if desired, optical whiteners, germicidal compounds, etc. This is accomplished by using an excess of the fatty acid, fatty acid ester or fatty acid halide with a suitable aliphatic alkanolamine, polyamine or alkanol polyamine, incorporating in the resultant condensate an ethoxylated, propoxylated or mixed ethoxylated propoxylated compound and converting the resultant mix into its corresponding acid salt by the addition of an organic or inorganic acid.

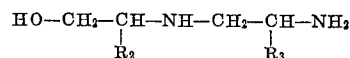
The softener compositions of the invention therefore are compatible with anionic washing detergents and comprise (1) from 50 to 90 parts by weight of an acid salt of a water-miscible acid selected from the group consisting of inorganic acid and aliphatic carboxylic acid having from 2 to 6 carbon atoms, with a condensation product of an excess of an aliphatic fatty acid derivative having from 8 to 22 carbon atoms selected from the group consisting of acids, lower alkyl esters and acid halides, with an aliphatic polyfunctional amine having at least two functional groups selected from the group consisting of hydroxyl and amino and (2) from 10 to 50 parts by weight of a lower alkoxylated compound selected from the group consisting of alcohols and amines. Preferentially the aliphatic polyfunctional amine contains both hydroxyl and amino groups such as an alkylolamine, although polyamines can be utilized.

The condensation of an excess of the fatty acid derivative with the polyfunctional amine results in a high molecular weight product having amide, imide and ester linkages. To obtain such linkages it is essential that more than one mol of fatty acid derivative be reacted per mol of polyfunctional amine, preferentially about one mol or slightly less of the fatty acid derivative is utilized for each hydroxyl and/or amino group of the polyfunctional amine. An amino group is to be understood to consist of a nitrogen atom attached to at least one hydrogen atom, the remaining valences are attached to at least one

carbon atom and either a carbon atom or a hydrogen atom. The resultant condensation product is then neutralized with a water-miscible aliphatic carboxylic acid having from 2 to 6 carbon atoms, or a water-miscible inorganic acid, preferentially an acid containing hydroxyl groups is utilized. Thereafter the acid salt is mixed with from 10 to 50 parts by weight, based on the total weight of the softener composition of an ethoxylated, propoxylated or mixed ethoxylated-propoxylated compound selected from the group consisting of alcohols and amines.

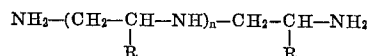
The aliphatic fatty acids which may be condensed with the alkanolamines or polyamines to obtain the acid-amine condensate are those which have from 8 to 22 carbon atoms such as lauric, caprylic, pelargonic, capric, undecylic, tridecylic, myristic, pentadecylic, palmitic, margaric, stearic, nonadecylic, arachidic and behenic acid. Esters and halides of these acids can also be used. Typical examples are the lower alkyl esters such as the ethyl, propyl and butyl esters, and such halides as chlorides and bromides.

Polyfunctional amines which may be suitably employed as reactants to form the initial acid-amine condensation product are preferably the alkanolamines such as monoethanolamine, diethanolamine, triethanolamine, N-methyl ethanolamine, N-ethyl ethanolamine, N-aminoethyl ethanolamine, N-methyl diethanolamine, monoisopropanolamine, diisopropanolamine, triisopropanolamine, mixed isopropanolamine, etc. Particularly alkanolamines having the formula



wherein R_2 and R_3 are members selected from the group consisting of hydrogen and lower alkyl, are preferred.

In addition, mixtures of these alkanolamines in all proportions may be utilized within the scope of the invention. In addition to the alkanolamines, polyamines can be employed, specifically those of the formula



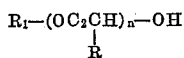
wherein n is an integer from 0 to 5 and R represents hydrogen or a lower alkyl, such as ethylene diamine, diethylene triamine, triethylene tetraamine, tetraethylene pentamine, propylene diamine, 1,3 - diaminopropane, imino-bispropylamine, dipropylenetriamine, etc. In addition other aliphatic polyamines may be employed such as alkanol alkylene polyamines, for example, dihydroxyethyl ethylene diamine (Nalco 706), monohydroxypropyl diethylene triamine (Nalco 1332), monohydroxy ethylpropylene triamine (Nalco L-1415), etc. In addition, mixtures of these polyamines or mixtures of alkanolamines and polyamines in all proportions may be utilized within the scope of the invention. Urea may be employed to react with excess amino if present in the acid amine condensate with the formation of carbamide groups to lower the cationic characteristics of the compound. Also, when an alkanolamine is employed in the preparation of the acid-amine condensate, urea may also be employed to react with excess hydroxyl groups if present in the acid-amine condensate with the formation of ester or urethane groups to lower the cationic characteristics of the compound.

The fatty acid and polyfunctional amine are reacted at temperatures above 100°C . while removing the reaction-formed water (if an acid is employed), hydrohalic acid (if an acid chloride is employed) or alcohol (if an acid ester is employed) under the usual reaction conditions. It is preferable to utilize temperatures in the range of 120°C . to 200°C . and higher temperatures up to the decomposition temperature may be employed. The upper limit of the reaction temperature also depends on the boiling point of the polyfunctional amine utilized.

The process of this invention utilizes the acid salt of the acid amine condensation product which may be ob-

tained by reacting the acid-amine condensation product with a water-miscible acid, such as an aliphatic carboxylic acid which may be hydroxy substituted. Among these water-miscible, aliphatic, mono-, di- and tricarboxylic acids are such acids as acetic, propionic, n-butyric, caproic and oxalic acid. Hydroxy derivatives of these acids such as glycolic, lactic, citric, gluconic and tartaric acids may also be employed and are in fact preferable from an odor standpoint. The process of this invention can also utilize the acid salt of the acid-amine condensation product which may be obtained by reacting the acid-amine condensation product with a water-miscible inorganic acid, such as hydrochloric, sulfuric, phosphoric and nitric acid. The acid salt of the acid-amine condensation product may be formed before, during or after the addition of the ethoxylated, propoxylated or mixed ethoxylated-propoxylated component. In any event the acid salt formation as well as the addition of the ethoxylated, propoxylated or mixed ethoxylated-propoxylated component is made to the molten condensation product. Thereafter the softener composition is cooled and flaked or dissolved in water or other solvent as desired.

The lower alkoxyalted compound selected from the group consisting of alcohols and amines are those compounds having the formula



wherein R is selected from the group consisting of hydrogen and lower alkyl and R_1 represents a member selected from the group consisting of alkoxyphenyl having from 13 to 20 carbon atoms, alkylphenyl having from 13 to 22 carbon atoms, alkylphenyl having from 13 to 22 carbon atoms, alkyl having from 8 to 22 carbon atoms and alkylaminoalkyl having from 12 to 22 carbon atoms, and n is an integer of from 2 to 50. As examples, these products include "Tergitol Nonionic NP-27" (nonyl phenol+7 mols of ethylene oxide), "Tergitol Nonionic 12-P-9" (dodecyl phenol+9 mols of ethylene oxide), "Tergitol Nonionic NP-35" (alkyl phenyl ether+15 mols of ethylene oxide), "Tergitol Nonionic NP-40" (nonyl phenol+20 mols of ethylene oxide), "Tergitol Nonionic NP-44" (nonyl phenol+40 mols of ethylene oxide), "Tergitol Nonionic NPX" (nonyl phenol+10.5 mols of ethylene oxide), "Tergitol Nonionic TP-9" (nonyl phenol+9 mols of ethylene oxide), "Tergitol Nonionic NP-33" (nonyl phenyl+13 mols of ethylene oxide), "Tergitol Nonionic 15-S-9" (secondary aliphatic alcohol having from 11 to 15 carbon atoms+9 mols of ethylene oxide), Alfonic 1218-6 (primary alkanol having from 12 to 18 carbon atoms and containing 60-65% of ethylene oxide units), "Lipal 6TD" (tridecyl alcohol+6 mols of ethylene oxide), etc. If biodegradability is a factor, the ethoxylated primary alcohols such as Alfonic 1218-6 would be preferred.

As examples of the ethoxylated amines, these products include "Ethomeen C/20" (coconut oil fatty acid amine+10 mols of ethylene oxide), "Ethomeen C/25" (coconut oil fatty acid amine+15 mols of ethylene oxide), "Ethomeen S/20" (Soybean oil fatty acid amine+10 mols of ethylene oxide), "Ethomeen S/25" (Soybean oil fatty acid amine+15 mols of ethylene oxide), "Ethomeen T/25" (tallow fatty acid amine+15 mols of ethylene oxide), "Ethomeen 0/15" (oleyl amine+5 mols of ethylene oxide), and "Ethomeen 18/60" (Stearyl amine+50 mols of ethylene oxide).

In addition, mixtures of ethoxylated alcohols, ethoxylated amines, or ethoxylated alcohols-ethoxylated amines may be utilized. It is also possible to utilize propoxylated derivatives of the various above-listed compounds, or

more preferably, mixed ethoxylated-propoxylated derivatives of the aforesaid compounds.

Fibers which can be rendered softened by the process of the present invention can be natural or synthetic, such as cotton and polyamide fibers, respectively. Polyamide fibers such as those derived from hexamethylene-diamine and adipic acid, and fibers of other synthetic materials such as rayon, Dacron (polyethylene terephthalate), Orlon (predominantly polyacrylonitrile), and blends of such fibers and fabrics containing these fibers or fiber blends are particularly adaptable to treatment by the softening compounds according to this invention.

The softener compositions of the invention are compatible with the customary "built" washing detergents. These detergents generally contain as active detergent from about 10 to 35% of sulfonates such as alkylbenzene sulfonates or sulfates such as fatty alcohol sulfates, 30 to 50% of alkali metal phosphates such as sodium tripolyphosphate, tetrasodium pyrophosphate, trisodium phosphate, etc., 2.5 to 5% of alkalis such as sodium silicate, 7 to 25% of sodium sulfate, minor amounts of optical whiteners such as fluorescent dyes and suspension agents such as water-soluble cellulose ethers and the remainder water. The products may be either in liquid form or in solid, powder or flake form depending on the water concentration. These detergents are ordinarily utilized in amounts of about 100 gm. per wash load of 8 pounds (1-1½ cups detergent for top loading automatic washing machines).

As previously indicated the softener compositions of the invention can be formulated with the above "built" washing detergents in an amount of from 1 to 10 parts by weight of the detergent composition. They can be added by a mechanical mixture with the solid products or dissolved in the liquid products. Preferably they are added to the detergent composition before or during the spray-drying operation to obtain a uniform solid detergent and softening composition which is stable and which readily dissolves in the washing machine to effect both a washing or soil removal and softening or deposition of the softener to the textile fabric being processed.

In order to demonstrate the products of this invention the following procedure was used:

The softener was tested in a normal automatic laundry equipment at temperatures of approximately 120° F. and

- added to the detergent wash
- added to the rinse, using a commercial "built" washing detergent of the approximate composition:

	Percent
Mixture of dodecylbenzenesulfonates and tri-decyl benzenesulfonates	31.7
Sodium pyrophosphate	7.4
Sodium tripolyphosphate	21.4
Sodium sulfate	22.5
Sodium chloride	1.1
Sodium silicate	4.2
Carboxymethyl cellulose	0.8
Magnesium silicate	0.1
Flourescent dye	0.1
Water	10.7
	100.0

as the standard detergent.

The use concentration of the softener varied from 500 to 1000 p.p.m. (100% active) based on the weight of the fabric, or amounts roughly to 1.8-3.6 grams active, based on a wash load of 8 pounds.

The following examples illustrate the process and products of the invention. It is to be understood however that other expedients may be employed and therefore these examples are not to be deemed limitative in any manner.

EXAMPLE I

(A) Acid-amine condensation

	Gm.
Hydrogenated tallow fatty acids (2.00 mols) -----	540.0
N-aminoethyl ethanolamine (1.05 mols) -----	114.4
Total -----	654.4

The reaction was run in a suitable vessel equipped with agitator, thermometer and distillation condenser. The reaction was gradually heated to a top temperature of 180° C. The water formed by the reaction was removed as formed and the reaction continued at 180° C. until a free acidity (as oleic acid) of approximately 7% was reached. The acid-amine condensation was then cooled to 120° C.

Yield:

Start -----	gm--	654.4
Water lost -----	gm--	34.0
Difference -----	gm--	620.4
Percent yield -----		94.8

(B) Preparation of softener

	Percent
Above acid-amine condensation -----	73.34
"Alfonic 1218-6" -----	25.00
Glycolic acid, 70% -----	1.66
	100.00

After cooling the acid-amine condensation to 120° C., the "Alfonic 1218-6" was added followed by the glycolic acid, with agitation. After thoroughly mixing, the softener was cooled to approximately 95° C. and the product flaked.

The product is a solid composition dispersible in hot water. A preservative such as formalin may be added to increase stability during storage. Numerous preservatives for cationic softeners other than formalin are known in the art and are applicable for use with the compositions used in this invention.

"Alfonic 1218-6" is an ethoxylate alcohol of an alkanol containing from 12 to 18 carbon atoms which is 60 to 65% ethoxylated.

(C) Analysis

pH (1% solution) -----	6.1-6.5
Total alkalinity (mg. KOH/g.) -----	mg./g-- 26.69
Acidity (as oleic acid) -----	percent-- 6.4

Compatibility test with Arctic White Acid Stable. Arctic White Acid Stable is an optical whitener and is a dark, amber colored aqueous solution having a pH of 7.5 to 8.5. According to the manufacturer, the product chemically is a substituted s-triazinyl aminodisulfostilbene.

	Percent
Softener of Example I -----	0.6
Arctic White Acid Stable -----	0.1

2.4 gm. of the softener of Example I was weighed into a 600 cc. beaker along with 1/3 of the total water to be used (132 gm.). The temperature was raised to 175° F. under agitation. After a few minutes the balance of the water (265.2 gm.) was added followed by mixing a few minutes longer. Finally the 0.4 gm. Arctic White Acid Stable was added with agitation and the resultant bath temperature was 140° F. The solution remained stable.

(D) Evaluation as a household laundry softner

The product was screened for potential possibilities as a household laundry softener as follows: the softener was tested in an automatic laundry machine at a temperature of approximately 120° F., adding the product

- to the detergent wash,
- to the rinse cycle.

In both cases the standard detergent was used. The softener use concentration varied from 500-1000 p.p.m. (100% active) based on the weight of the fabric, or amounts roughly 1.8-3.6 gm. active, based on a wash load of 8 pounds.

The softener, in addition to being compatible with the standard anionic detergent showed the following advantages:

- (1) Substantivity
- (2) Good hand
- (3) Nonyellowing
- (4) Compatible with anionic detergents

(E) Compatibility with germicidal compounds

The softener of Example I was checked for compatibility with the germicidal compound "BTC-2125." The following formulation was used:

	Percent
Softener (Example I) -----	6.0
"BTC-2125" -----	2.4
Water -----	91.6
	100.0

Compatibility of the softener with the germicidal compound was found to be excellent.

"BTC-2125" consists of:

- 25% n-alkyl (60% C₁₄, 30% C₁₆, 5% C₁₂, 5% C₁₈) dimethylbenzyl ammonium chlorides
- 25% n-alkyl (50% C₁₂, 30% C₁₄, 17% C₁₆, 3% C₁₈) dimethyl ethylbenzyl ammonium chlorides
- 50% inert ingredients

EXAMPLE II

(A) Acid-amine condensation

	Gm.
Palmitic acid (2.00 mols) -----	524.0
N-aminoethyl ethanolamine (1.05 mols) -----	114.0
Total -----	638.0

The reaction was run in a suitable vessel equipped with agitator, thermometer and distillation condenser. The reaction was gradually heated to a top temperature of 180-184° C., the water formed by the reaction was removed as formed, and the reaction continued at 180-184° C. until a free acidity (as oleic acid) of approximately 7% was obtained. The acid-amine condensate was then cooled to 120° C.

Yield:

Start -----	gm--	638.4
Water lost -----	gm--	34.0
Total yield -----	gm--	604.4
Percent yield -----		94.7

(B) Preparation of softener

	Percent
Acid-amine condensation -----	73.34
"Tergitol Nonionic NPX" -----	25.00
Glycolic acid, 70% -----	1.66
	100.00

After cooling the acid-amine condensation to 120° C., the "Tergitol Nonionic NPX" was added followed by the glycolic acid, with agitation. After thoroughly mixing, the product was flaked.

"Tergitol Nonionic NPX" is a nonylphenol ethoxylated with an average of 10.5 mols of ethylene oxide.

(C) Analysis

Moisture -----	percent--	1.0
Solids at 105° C. -----	do--	99.1
pH (1% solution) -----		6.4
Compatibility test with Arctic White Acid Stable -----		satisfactory.

Compatibility with BTC-2125 satisfactory.

EXAMPLE III

(A) Acid-amine condensation

	Gm.
Isostearic acid ("Emery 3101") (2.0 mols) -----	624.0
N-aminoethyl ethanolamine (1.4 mols) -----	145.8
Total -----	769.8

The reaction was run in a suitable vessel equipped with agitator, thermometer, and distillation condenser. The reaction was gradually heated to a top temperature of 180–184° C., the water formed by the reaction was removed as formed, and the reaction continued at 180–184° C. until a free acidity (as oleic acid) of approximately 6–7% was obtained. The acid-amine condensate was then cooled to 60–70° C.

Yield:

Start -----	gm--	769.8
Water lost -----	gm--	36.0
Total yield -----	gm--	733.8
Percent yield -----		95.3

(B) Preparation of softener

	Percent
Acid-amine condensation -----	67.5
"Alfonic 1218-6" -----	25.0
Glycolic acid, 70% -----	7.5
Total -----	100.0

After cooling the acid-amine condensation to 60–70° C., the "Alfonic 1218-6" was added followed by the glycolic acid, with agitation. After thoroughly mixing, the softener was cooled to room temperature resulting in a thick transparent liquid. Below room temperature, the softener is a soft hazy paste.

"Emery 3101" isostearic acid is a liquid isomer of stearic acid that combines the saturation of stearic acid with the liquid nature and solubility characteristics of oleic acid. It enables the manufacturer to produce derivatives resembling oleic derivatives that have stearic acid's stability against oxidation and darkening.

Chemically, isostearic acid is a C_{18} saturated fatty acid of the formula $C_{17}H_{35}COOH$. Evidence to date indicates that it is a complex mixture of isomers, primarily of the methyl-branched series that are mutually soluble and virtually inseparable. As a result, its titer or freezing point is much lower than is ordinarily encountered in a saturated fatty acid of this molecular weight.

EXAMPLE IV

(A) Acid-amine condensation

	Gm.
Hydrogenated tallow fatty acids (2.00 mols) -----	540.0
Diethylene triamine (1.1 mols) -----	113.5
Total -----	653.5

The reaction was run as in Example I, except that the reaction was gradually heated to a top temperature of 190° C.

Yield:

Start -----	gm--	653.5
Water lost -----	gm--	36.0
Difference -----	gm--	617.5
Percent yield -----		94.5

(B) Preparation of softener

	Percent
Above acid-amine condensation -----	73.34
"Alfonic 1218-6" -----	25.00
Glycolic acid, 70% -----	1.66
Total -----	100.00

The reaction was run as in Example I(B).

EXAMPLE V

The reaction was run as in Example IV, the charge was as follows:

(A) Acid-amine condensation

(A) Acid-amine condensation		Gm.
15	Hydrogenated tallow fatty acids (3.0 mols) -----	810.0
	Diethylene triamine -----	113.5
	Total -----	923.5
	Yield:	
20	Start -----	gm-- 923.5
	Water lost -----	gm-- 48.0
	Difference -----	gm-- 875.5
	Percent yield -----	94.8

(B) Preparation of softener

	Percent
Above acid-amine condensation -----	73.34
"Alfonic 1218-6" -----	25.00
Glycolic acid, 70% -----	1.66
Total -----	100.00

The reaction was run as in Example I(B).

EXAMPLE VI

(A) Acid-amine condensation

	Gm.
Butyl stearate (2.0 mols) -----	680.0
N-aminoethyl ethanolamine (1.1 mols) -----	114.4
Total -----	794.4

The reaction was run in a suitable vessel equipped with agitator, thermometer and distillation condenser. The reaction was gradually heated to a top temperature of 195° C. The butanol formed by the reaction was removed as formed and the reaction continued at 195° C. until about two mols of the butanol were collected. During the reaction, nitrogen may be bubbled through the reaction mix to assist in removing the butanol and also to improve the color of the acid-amine condensation product. The acid-amine condensation was then cooled to 120° C.

(B) Preparation of softener

	Percent
Above acid-amine condensation -----	71.50
"Alfonic 1218-6" -----	25.00
Glycolic Acid, 70% -----	3.50
Total -----	100.00

The reaction was run as in Example I(B).

EXAMPLE VII

In Example VII the butyl stearate can be replaced by the corresponding halides, for example stearyl chloride, as follows:

(A) Acid-amine condensation

	Gm.
Stearyl chloride (2.0 mols) -----	605.8
N-aminoethyl ethanolamine (1.1 mols) -----	114.4
Total -----	720.2

During the reaction the hydrogen chloride evolved is complexed by the acid-amine condensation product, which

in turn aids in the solubilization of the acid-amine condensate.

The acid-amine condensate product is then compounded with an ethoxylated alcohol or amine such as "Alfonic 1218-6" or "Ethomeen T/25" in the approximate ratio of 75:25 (75 parts acid-amine condensate to 25 parts ethoxylated compound). Depending upon the solubility desired, additional organic or inorganic acid may be added in a similar manner as shown in the previous examples.

EXAMPLE VIII

(A) Acid-amine condensation

	Gm.
Hydrogenated tallow fatty acids (2.0 mols) -----	540.0
Diethanolamine (1.1 mols) -----	115.7
Total -----	655.7

The reaction was run in a suitable vessel equipped with agitator, thermometer and distillation condenser. The reaction was gradually heated to a top temperature of 185° C. The water formed by the reaction was removed as formed and the reaction continued at 185° C. until a free acidity (as oleic acid) of approximately 5.5% was reached. The acid-amine condensation was then cooled to 120° C.

(B) Preparation of softener

	Percent
Above acid-amine condensation -----	72.20
"Alfonic 1218-6" -----	25.00
Glycolic acid, 70% -----	2.80
Total -----	100.00

The reaction was run as in Example I(B).

EXAMPLE IX

Preparation of softener

	Percent
Acid-amine condensate of Example I -----	73.34
"Ethomeen T/25" -----	25.00
Glycolic acid, 70% -----	1.66
Total -----	100.00

The reaction was run as in Example I(B).

EXAMPLE X

(A) Acid-amine condensate

	Gm.
Stearic acid (2.0 mols) -----	534.0
N-aminoethyl ethanolamine (1.5 mols) -----	156.0
Urea (1.0 mol) -----	60.0
Total -----	750.0

The reaction was run in a suitable vessel equipped with agitator, thermometer and distillation condenser. The stearic acid and N-aminoethyl ethanolamine were charged into the reactor, and the reaction gradually heated to a top temperature of 195° C. under a nitrogen blanket. Approximately 42 gms. of water were collected, and a free acidity (as oleic acid) of 6-7% was reached. The acid-amine condensation was then cooled to 140° C. and the urea then added in three portions allowing the frothing to cease between additions. After all the urea has been added, the reaction temperature is gradually raised to 170° C. and held at this top temperature of 170° C. for 15 minutes. The carbamide thus formed is cooled to 110-120° C.

Note: As soon as the urea is added, ammonia gas is formed. The ammonia is absorbed in a suitable acid or vented off to the atmosphere. Nitrogen gas was used at the end to expel all remaining ammonia gas.

Yield:

Start -----	gm-- 750.0
Water lost -----	gm-- 42.0
Ammonia gas lost -----	gm-- 17.0
Difference -----	gm-- 691.0
Percent yield -----	92.13

(B) Preparation of softener

	Percent
Above carbamide -----	82.2
"Alfonic 1218-6" -----	14.0
Acetic acid (glacial) -----	3.8
Total -----	100.0

The reaction was run as in Example I(B).

EXAMPLE XI

Softener compositions represented by Examples I through X may then be incorporated in built-in detergents for heavy-duty cleaning and can be added:

- (a) to the slurry before spray drying,
- (b) to the heavy-duty liquid.

A typical composition for a detergent-softener household product is as follows:

	Percent
Mixture of dodecylbenzenesulfonates and tridecyl benzenesulfonates -----	30.43
Sodium pyrophosphate -----	7.10
Sodium tripolyphosphate -----	20.54
Sodium sulfate -----	21.60
Sodium chloride -----	1.06
Sodium silicate -----	4.03
Carboxymethyl cellulose -----	0.77
Magnesium silicate -----	0.10
Florescent dye -----	0.10
Water -----	10.27
Softener composition of Example I -----	4.00
Total -----	100.00

The softener composition may be added to the detergent slurry before spray drying or intimately mixed in the finely granulated form with the powdered detergent.

In a laboratory evaluation, three eight-pound loads of soiled clothing consisting of cotton undergarments, hand towels, bath towels, etc., were washed in a standard washing machine in the following manner:

- (a) With the standard anionic detergent alone,
- (b) With the standard anionic detergent plus the softener composition of Example I in the washing cycle,
- (c) With the standard anionic detergent alone, followed by application of "Arquad 2HT-75" softener in the after-rinse cycle.

Based on washing instructions for the above detergent, 1½ cups of the detergent (approximately 100 gms.) was used for a top loading washing machine, and 1000 parts per million (100% active) of the softener based on the weight of the fabric was used. This amounted to 3.6 grams active softener for an eight-pound load. Note that due to incompatibility, the "Arquad 2HT-75" was used in the after-rinse cycle which is present day standard operating procedure.

After the fabric loads were scoured, softened, extracted and dried in the normal manner and the loads conditioned at 65% R.H. and 70° F. for 12 hours, a panel of four observers concluded the following:

- (1) The fabric load scoured and softened with the detergent softener mix in the scouring cycle was softer, fluffier and had an improved handle as compared to the fabric load which was only scoured with the standard anionic detergent.

(2) The fabric load scoured and softened with the detergent-softener mix in the scouring cycle was equal or slightly better in softness, fluffiness and handle when compared to the fabric load scoured with the standard anionic detergent in the washing cycle and separately softened with "Arquad 2HT-75" in the after-rinse cycle.

To illustrate the difference between the softener compositions of our invention as compared to products which are currently used and sold in household after-rinse softener formulations, examples of which are "Aquad 2HT-75" (dimethyl distearyl ammonium chloride) and "Armosoft AB" (1-methyl-1-alkylamidoethyl-2-alkyl-imidazolium methosulfate), the following compatibility tests were made with five commercial detergents presently sold on the American and European markets.

COMPATIBILITY TEST OF VARIOUS SOFTENERS WITH DETERGENTS

	"Fab"	"Tide"	"Dixan"	"Persil 65"	"Sator"
Softener of Ex. I	Comp.	Comp.	Comp.	Comp.	Comp.
Softener of Ex. III	Comp.	Comp.	Comp.	Comp.	Comp.
Softener of Ex. IX	Comp.	Comp.	Comp.	Comp.	Comp.
Softener of Ex. X	Comp.	Comp.	Comp.	Comp.	Comp.
"Arquad 2HT-75"	Incomp.	Incomp.	Incomp.	Incomp.	Incomp.
"Armosoft AB"	Incomp.	Incomp.	Incomp.	Incomp.	Incomp.

The above test was based on the use of 1½ cups (approximately 100 gm.) of detergent for a top loading machine, and a wash load of eight pounds. The use concentration of the softener was 1000 p.p.m. (100% active) based on the weight of the fabric, or 3.6 grams active based on a wash load of eight pounds. According to manufacturers' information pertaining to household washing machines, the volume of water during the washing cycle is approximately 8-12 gallons. For the purpose of the above compatibility test, the following bath composition was used:

	Percent
Detergent	0.26
Softener (100% active)	0.01
Water at 130-140° F.	99.73
	100.00

In the above softener-detergent compatibility test, the composition of the detergents used, based on active constituents (those other than water) basically fall into one of the following categories.

- (1) Alkylbenzene sulfonates, fatty alcohol sulfates, carboxymethyl cellulose, sodium silicate, sodium tripolyphosphate, sodium sulfate, sodium chloride and fluorescent dye.
- (2) Alkylbenzene sulfonates, carboxymethyl cellulose, sodium silicate, sodium pyrophosphate, sodium tripolyphosphate, sodium sulfate, sodium chloride, magnesium silicate and fluorescent dye.
- (3) Alkylbenzene sulfonates, soap, ethylene oxide adducts, carboxymethyl cellulose, sodium tripolyphosphate, sodium silicate, sodium perborate and fluorescent dye.

In some cases the composition of the alkylbenzene sulfonates and soap is the main difference in the products. Compatibility test of various softeners with "Arctic White Acid Stable" optical whitener:

Softener of Ex. I	Compatible
Softener of Ex. III	Compatible

Softener of Ex. IX	Compatible
Softener of Ex. X	Compatible
"Arguad 2HT-75"	Incompatible
"Armosoft AB"	Incompatible

Procedure.—2.4 gm. of the softener was weighed into a 600 cc. beaker along with ⅓ of the total water to be used (132 gm.). The temperature was raised to 175° F. under agitation. After a few minutes the balance of the water (265.2 gm.) was added followed by mixing a few minutes longer. Finally the 0.4 gm. "Arctic White Acid Stable" was added with agitation and the resultant bath temperature was 140° F. The test was conducted for a period of 12 hours.

The Arctic White Acid Stable chemically is a substituted

s-triazinyl aminodisulfostilbene derivative. Coumarin type optical brightening agents, for example "BMU" (4-methyl-7-hydroxycoumarin), was found to produce similar compatibility data.

Compatibility test of various softeners with germicidal compound "BTC-2125":

Softener composition of Ex. 1	Coamptible
Softener composition of Ex. IX	Compatible
Softener composition of Ex. X	Compatible
"Arquad 2HT-75" (gels)	Incompatible

The above compatibility tests were performed using the following formulation:

	Percent
Softener solution (6% active)	97.6
"BTC-2125"	2.4
	100.0

Stability of the formulations were checked for a period of two weeks.

In order to demonstrate that the dimethyl-di(hydrogenated tallow)-ammonium chloride type fabric softener has the tendency to build up on successive washes, resulting in reduced absorbency of the washed and treated fabric, the following comparison was made in the wick-up properties of toweling softened with "Arquad 2HT-75" (a dimethyl distearyl ammonium chloride softener) as compared to a towel softened with a softener composition of our invention as represented by Example III.

Wick-up test (American Dyestuff Reporter, Apr. 12, 1943, page 167): The softened toweling is cut 6" long by ¾" wide in the warp direction. A Draves "hook and weight" is attached to the lower end of the sample exactly ½" from the end. A ruled line parallel to the width is also made at this ½" mark for ease of measuring. The sample is then supported in deionized water, tinted red with "Neolan Red 3B" (or any other water-soluble red dyestuff) up to the ½" mark. The height to which the red liquid rises over a 5-minute period is noted and reported in inches.

Comparison of "Arquad 2HT-75" versus softener composition of Example III in the wick-up test:

	Wick-up (inches) (wrap direction) after—				
	1 min.	2 mins.	3 mins.	4 mins.	5 mins.
After 1 washing and softening:					
(a) "Arquad 2HT-75"-----	2	2½	3	3¼	3½
(b) Softener Ex. III-----	2	2½	2¾	3½	3¾
After 2 washings and softening:					
(a) "Arquad 2HT-75"-----	1¼	1½	2	2¼	2¾
(b) Softener Ex. III-----	2	2½	2¾	3½	3¾
After 3 washings and softening:					
(a) "Arquad 2HT-75"-----	1¼	1½	2	2¼	2¾
(b) Softener Ex. III-----	2	2½	2¾	3½	3¾
After 4 washings and softening:					
(a) "Arquad 2HT-75"-----	1¾	1½	2½	2¼	2½
(b) Softener Ex. III-----	2½	2½	2½	3¼	3½
After 5 washings and softening:					
(a) "Arquad 2HT-75"-----	1¼	1½	1¾	2	2¼
(b) Softener Ex. III-----	2	2½	2¾	3½	3¾

The original terry toweling as received from a towel mill, after the bleaching operation, demonstrated the following wick-up properties:

Minutes:	Inches
1 -----	2
2 -----	2½
3 -----	2¾
4 -----	3½
5 -----	3¾

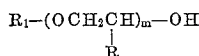
The above data illustrates the following:

- (1) Repeated washing and softening with "Arquad 2HT-75" type softeners appreciably reduces the absorbency of the toweling.
- (2) Softener compounds of our invention have little or no effect on the absorbency of toweling after repeated laundering and softening.

The preceding specific embodiments are illustrative of the invention. It is to be understood however that other expedients may be employed.

We claim:

1. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of a hydroxyl-containing, water-miscible aliphatic hydrocarbon carboxylic acid having from 2 to 6 carbon atoms with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of an excess of an aliphatic saturated, fatty acid having from 8 to 22 carbon atoms, with N-aminoethyl ethanolamine, said fatty acid being utilized in a ratio of about 2 mols for each mol of said alkanolamine, said acid-amine condensation product being utilized in a ratio of about 2 mols for each mol of said alkanolamine at a temperature of from 100° C. to the decomposition temperature for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of a lower alkoxyated compound of the formula



wherein R is selected from the group consisting of hydrogen and methyl and R₁ represents a member selected from the group consisting of alkoxyphenyl having from 13 to 20 carbon atoms, alkylphenyl having from 13 to 22 carbon atoms, alkylnaphthyl having from 13 to 22 carbon atoms, alkyl having from 8 to 22 carbon atoms and alkyl-aminoalkyl having from 12 to 22 carbon atoms, and m is an integer of from 2 to 50.

2. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of hydrogenated tallow fatty acid with about 1 mol

of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said hydrogen-

ated tallow fatty acid with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

3. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of palmitic acid with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said palmitic acid with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated nonyl phenol containing about 10.5 mols of ethylene oxide.

4. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of isostearic acid with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said isostearic acid with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

5. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of butyl stearate with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said butyl stearate with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed butanol, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

6. A softener composition compatible with anionic washing detergents, consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of hydrochloric acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of stearyl chloride with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said stearyl chloride with said N-aminoethyl ethanolamine at a temperature

of from 120° C. to 200° C. for a time sufficient to effect substantially complete condensation, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

7. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of hydrochloric acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of stearyl chloride with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said stearyl chloride with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to effect substantially complete condensation, and (2) from 10 to 50 parts by weight of an ethoxylated tallow fatty acid-amine containing about 15 mols of ethylene oxide.

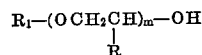
8. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of hydrogenated tallow fatty acid with about 1 mol of diethanolamine, said acid-amine condensation product being produced by reacting said hydrogenated tallow fatty acid with said diethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

9. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of hydrogenated tallow fatty acid with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said hydrogenated tallow fatty acid with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated tallow fatty acid amine containing about 15 mols of ethylene oxide.

10. A softener composition compatible with anionic washing detergents consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of acetic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of stearic acid with about 1.5 mols of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said stearic acid with said N-aminoethyl ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

11. A detergent and softener composition consisting essentially of (A) from about 90 to about 99 parts by weight of a commercial built detergent containing from about 10 to 35% of organic sulfonate detergents, from about 30 to 50% of alkali metal phosphates and from about 7 to 25% of sodium sulfate and (B) from about 1 to about 10 parts by weight of a softener composition comprising (1) from 50 to 90 parts by weight of an acid salt of a hydroxyl-containing, water-miscible aliphatic hydrocarbon carboxylic acid having from 2 to 6 carbon atoms with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of an excess of an aliphatic saturated, fatty acid having from 8 to 22 carbon atoms, with N-aminoethyl ethanolamine, said fatty acid being utilized in a ratio of about 2 mols for each mol of said alkanolamine, said

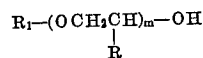
acid-amine condensation product being produced by reacting said aliphatic fatty acid with said alkanolamine at a temperature of from 100° C. to the decomposition temperature for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of a lower alkoxyated compound of the formula



wherein R is selected from the group consisting of hydrogen and methyl and R₁ represents a member selected from the group consisting of alkoxyphenyl having from 13 to 20 carbon atoms, alkylphenyl having from 13 to 22 carbon atoms, alkynaphthyl having from 13 to 22 carbon atoms, alkyl having from 8 to 22 carbon atoms and alkylaminoalkyl having from 12 to 22 carbon atoms, and *m* is an integer of from 2 to 50.

12. A detergent and softener composition consisting essentially of (A) from about 90 to about 99 parts by weight of a commercial built detergent containing from about 10 to 35% of organic sulfonate detergents, from about 30 to 50% of alkali metal phosphates and from about 7 to 25% of sodium sulfonate and (B) from about 1 to about 10 parts by weight of a softener composition comprising (1) from 50 to 90 parts by weight of an acid salt of glycolic acid with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of about 2 mols of hydrogenated tallow fatty acid with about 1 mol of N-aminoethyl ethanolamine, said acid-amine condensation product being produced by reacting said hydrogenated tallow fatty acid with said N-amino ethanolamine at a temperature of from 120° C. to 200° C. for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of an ethoxylated primary alkanol having from 12 to 18 carbon atoms and containing about 60% to 65% of ethylene oxide units.

13. A process for washing and softening textiles in a machine washer which comprises filling said washer with water, adding textiles to said water, adding a commercial built detergent containing from about 10 to 35% of organic sulfonate detergents, from about 30 to 50% of alkali metal polyphosphates, and from about 7 to 25% of sodium sulfate, adding from about 500 to 1000 parts per million based on said textiles of a softener composition consisting essentially of (1) from 50 to 90 parts by weight of an acid salt of a hydroxyl-containing, water-miscible aliphatic hydrocarbon carboxylic acid having from 2 to 6 carbon atoms with an acid-amine condensation product having a free acidity of not more than 7%, calculated as oleic acid, of an excess of an aliphatic saturated, fatty acid having from 8 to 22 carbon atoms, with N-aminoethyl ethanolamine, said fatty acid being utilized in a ratio of about 2 mols for each mol of said alkanolamine, said acid-amine condensation product being produced by reacting said aliphatic fatty acid with said alkanolamine at a temperature of from 100° C. to the decomposition temperature for a time sufficient to remove the reaction-formed water, and (2) from 10 to 50 parts by weight of a lower alkoxyated compound of the formula



wherein R is selected from the group consisting of hydrogen and methyl and R₁ represents a member selected from the group consisting of alkoxyphenyl having from 13 to 20 carbon atoms, alkylphenyl having from 13 to 22 carbon atoms, alkynaphthyl having from 13 to 22 carbon atoms, alkyl having from 8 to 22 carbon atoms and alkylaminoalkyl having from 12 to 22 carbon atoms, and *m* is an integer of from 2 to 50, washing said textiles,

rinsing said textiles and recovering said washed and softened textiles.

3,122,504	2/1964	Wedell	232—8.8 X
3,351,483	11/1967	Miner et al.	252—137
3,360,470	12/1967	Wixon	252—137

References Cited

UNITED STATES PATENTS

2,734,830	2/1956	Haage et al.	252—8.8 X
3,044,962	7/1962	Brunt et al.	252—8.8 X

5

HERBERT B. GUYNN, *Primary Examiner.*

U.S. Cl. X.R.

117—139.5; 252—8.75, 137, 152

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,454,494 Dated July 8, 1969

Inventor(s) John E. Clark et al

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Patent

<u>Col.</u>	<u>Line</u>	
<u>7</u>	<u>67</u>	Please correct the spelling of "softener"
10	16	Please insert -- (1.1 mols) --
14	3	Please correct the spelling of "Arquad"
14	41	Please correct the spelling of, "compatible"
15	53	The words "utilized in a ratio of about 2 mols for each mol of" should read -- produced by reacting said aliphatic fatty acid with --
18	24	The word "sulfonate" should read -- sulfate --.

SIGNED AND
SEALED
JUN 23 1970

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

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Commissioner of Patents