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FABRIC SOFTENER COMPOSITIONS

Walter F. Weiss, Wheaton, Ill., assignor to Arthur D. Little, Inc., Cambridge, Mass., a corporation of Massachusetts

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This application is a continuation-in-part of my copending application, Serial No. 216,763, filed Aug. 14, 1962, now abandoned.

This invention relates to compositions useful for imparting softness to fabrics and to an improved process for producing a fabric softener composition in a form most convenient for distribution and use.

At the present time, compositions useful for treating fabrics to improve the softness and feel characteristics thereof are known in the art. Normally, these softener compositions designated fabric softeners which are employed to treat fabrics during laundering are liquids which contain as the principal active component a quarternary ammonium compound, that is a substituted ammonium salt in which all four of the hydrogens of the ammonium radical are substituted by organic radicals, or other cationic nitrogen-containing compounds such as amines. The liquid nature of these fabric softeners detracts considerably from their commercial acceptance. While the physical form of a fabric softener composition does not affect its fabric softening properties, convenience in packaging, distribution and use makes a solid form extremely desirable. Accordingly, a major object of this invention is to provide a fabric softener composition of excellent softening characteristics in a solid form.

When used in domestic laundering, the fabric softeners are added to the rinse water during the rinse cycle which is generally from about 3 to 5 minutes' duration. Consequently, the fabric softener must be capable of rapid dispersion in the rinse water so as to produce an effective concentration of the product therein for fabric softening in the relatively short period of the rinse cycle. Liquid fabric softeners present no serious problems in this connection, however, the same cannot be said for fabric softeners in solid form. The physical form of the fabric softener has been found to be of considerable importance in achieving rapid dispersion in the rinse water during the rinse cycle and in particular it has been found that the solid fabric softener should be in the form of relatively thin flakes having a thickness not substantially in excess of about 0.025 inch and preferably about 0.005 to 0.015 inch. It is therefore a further object of this invention to provide a fabric softener composition which can readily be produced in the form of relatively thin flakes capable of rapid dispersion in water. The fabric softener composition can also be produced in granular form.

The significant, readily apparent improvement in the softness of fabrics resulting from the use of fabric softeners has resulted in the creation of a large and expanding consumer market for these products. This economic fact dictates that the fabric softener compositions be efficiently produced in large amounts in minimum processing time. It is therefore another object of this invention to provide a process for producing a fabric softener composition in solid form which process is capable of producing large quantities of the softener composition quickly and with the use of readily available production equipment.

The principal active component of the present fabric

softener composition is a cation-active organic compound having fabric softening properties. These cation-active organic compounds, which can be used as active fabric softeners, are known to the art and generally comprise cationic nitrogen-containing compounds such as quaternary ammonium compounds, and amines containing one or two straight-chain organic radicals of at least 8 carbon atoms and preferably from 12 to 22 carbon atoms. Representative of preferred cationic compounds are quaternary ammonium compounds having one or two straight-chain organic radicals containing from 12 to 22 carbons atoms; primary, secondary and tertiary amine compounds having at least one straight-chain organic radical containing from 12 to 22 carbon atoms; 1,3-propylene diamine compounds having a C_{12} to C_{22} straight-chain organic radical and like compounds. Specific preferred fabric softening compositions include such compounds as di-(hydrogenated tallow) dimethylammonium chloride, di-(hydrogenated tallow) dimethylammonium methyl sulfate, primary tallow amine, primary hydrogenated tallow amine, tallow 1,3-propylene diamine, oleyl 1,3-propylene diamine and coco 1,3-propylene diamine, di-(soya) dimethylammonium chloride, and d-(coco) dimethylammonium chloride.

The cationic nitrogen-containing component constitutes from about 4 to about 40 percent, preferably from about 18 to 25 percent by weight of the softener composition of the invention. Urea which is reacted with the cationic nitrogen-containing compound to form an adduct constitutes the major component of the softener composition and is employed in amounts ranging from about 50 to 90 percent by weight. The urea should be employed in an amount at least equal to that stoichiometrically required for reaction with the cationic nitrogen-containing compound and preferably the urea is employed in theoretical excess in a proportion ranging from about 2 to 180 moles of urea per mole of cationic nitrogen-containing compound.

In addition to the above major components of the softener composition, relatively small amounts of organic nonionic wetting agents or surface active agents can be employed, if desired, to counteract the hydrophobic characteristics of the cationic nitrogen-containing compounds. A variety of organic nonionic surface active agents are known to the art which are suitable for use in the present fabric softener composition such as, for example, Triton X–100 (a condensate of isooctyl phenol with about 8 moles of ethylene oxide), Ethomeens (oxyethylated secondary amines), Hyonics (e.g. fatty alkylolamides) and the like. The above representative wetting agents can be employed in the fabric softener compositions of the invention in amounts ranging from about 1 to 20%. Moreover, if desired, any suitable perfume or dye can be employed in the softener composition to improve the esthetic attractiveness of the softener product to the ultimate consumer. Such dyes and perfumes are employed in relatively small amounts, generally less than about 0.25 percent by weight of the composition.

A typical finished fabric softener composition prepared in accordance with the invention is as follows, the parts being by weight:

Urea	75.00
Cationic nitrogen-containing compound	22.00
Surface active agent	2.75
Perfume	0.250
Dye	0.005

The preparation of the fabric softener compositions is carried out in accordance with the invention by blending the urea, cationic nitrogen-containing compound and other components of the composition in the presence of a specified amount of water. I have found surprisingly that in order to achieve a solid granular product which can be readily granulated or flaked to the desired size, it is necessary to add water to the reaction mixture in a relatively narrow and critical range. More particularly, it is necessary to employ water in the reaction mixture in an amount from about 2 to about 15 percent and preferably from 3 to 5 percent of the softener composition. Employing water in the specified proportions and effecting reaction of the urea and cationic nitrogen-containing compound in the presence of the water constitute important features of the present invention. If less or substantially more than the stated amounts of water are employed in the preparation of the fabric softener composition, the products do not solidify satisfactorily and do not granulate or flake readily.

In one embodiment of the invention, urea is charged to a suitable mixing vessel and the cationic nitrogen-containing compound mixed therewith, preferably in liquefied form. The reaction is effected at substantially ambient temperatures. The cationic nitrogen-containing compound can be supplied to the reaction site in the form in which it is available commercially such as, for example, dissolved in low molecular weight alcohols. The requisite amount of water specified above, namely from about 2 to about 15 percent and preferably from 3 to 5 percent by weight is added to the mixture and the mixture agitated for a short period, for example, 5–8 minutes, until a homogeneous granular product is produced. The requisite water can be added to the reaction mixture before or after addition of the cationic-nitrogen-containing compound. Any water inherently present in the ingredients of the composition must be taken into account. The wetting agents in liquid form and dyes and perfumes, if any, are incorporated into the reaction mixture prior to solidification of the granular product. The granular product obtained from the reaction is then milled on rolling mills or the like to produce relatively small granules or thin flakes. The resulting particles are then screened to a desired size and the finished fabric softener composition packaged for distribution. The finished fabric softener product generally contains less than about 1% water.

The following specific examples will further illustrate the invention, the parts indicated being by weight unless otherwise indicated.

EXAMPLE I

Three hundred grams of urea are charged into a mixing bowl such as a 5-quart Hobart mixer. Ninety grams of a quaternary ammonium compound, di-(hydrogenated tallow) dimethylammonium chloride and 7.5 grams of a second cationic surface active agent, liquefied by warming, are added to the urea charge and blended for 30 to 45 seconds at medium mixing speed. A solution of 3.7 grams of an organic nonionic surface active agent in 8 grams of water, containing a perfume and soluble coloring dye is added to the blend of urea, quaternary ammonium compound and surfactant while mixing is continued.

The total amount of water employed corresponds to about 3% by weight of the mixture. Within 30 seconds after the addition of water a stiff dough forms. Within two minutes after the addition of water the doughy mass begins to granulate. Within 5 to 6 minutes after the addition of water granulation is completed.

A slightly moist non-greasy or non-waxy granulation is produced in less than eight minutes which can be passed through a flaking mill to produce a thin brittle flake.

These flakes are capable of rapid solution and dispersion in an automatic washing machine. Cationic fabric softener is liberated and dispersed by destruction of the flake and made available for deposition on the fabric being treated. The fabric softener prepared as indicated was evaluated as follows:

Subjective examination of treated fabric

Fabrics treated:

New gauze diapers scoured by 2 normal washings before treating with solid fabric softener.

New turkish towels scoured by 1 normal washing before treating with solid fabric softener.

[Gauze diapers examined by a 5 member panel]

Treatment:

Four grams of solid softener, rinse cycle treatment of a six pound load.

Three out of five panel members selected the treated diapers as softer than a control handled in an identical manner except for addition of solid softener in the rinse cycle.

Treatment:

Eight grams of solid softener, rinse cycle treatment of a six pound load.

Four out of five panel members selected the treated diapers as softer than a control handled in an identical manner except for the addition of solid softener in the rinse cycle.

Treatment:

Fifteen grams of solid softener, rinse cycle treatment of a six pound load.

Five out of five panel members selected the treated diapers as softer than a control handled in an identical manner except for the addition of solid softener in the rinse cycle.

[Turkish towels examined by a 5 member panel]

Treatment:

Ten grams of solid softener, rinse cycle treatment of a six pound load.

Five out of five panel members selected the treated towels as obviously softer than a control handled in an identical manner except for the addition of solid softener in the rinse cycle.

EXAMPLE II

Three hundred grams of urea are charged into a mixing vessel. Ninety grams of a quaternary ammonium compound purchased from Emery Industries and designated Emery 3484–D and 7.5 grams of a second cationic surface active agent, liquefied by warming, are added to the urea charge and blended for ½ minute. A water solution containing eight grams of water, 3.7 grams of an organic nonionic surface active agent, perfume and coloring are added to the blend with continuing mixing. The water employed corresponds to about 3% of the total mixture. A greasy mobile dough is formed which begins to granulate in about 6 minutes and is completed in about 11 minutes.

The granules formed are soft and somewhat self adherent but flake readily in one pass through cold flaking rolls. The flakes are soft and waxy initially but become somewhat brittle after several hours of aging.

EXAMPLE III

Three hundred grams of urea are charged into mixing vessel. Ninety grams of a quaternary ammonium compound, di-(hydrogenated tallow) dimethyl ammonium chloride and 7.5 grams of a second cationic surface active agent, liquefied by warming, are added to the urea charge and blended for ½ minute. A mixture of 2 grams total of water, 3.7 grams of an organic nonionic surface active agent, perfume and dye are added to the blend. The total amount of water employed corresponds to about 1.5% by weight of the mixture. A mobile greasy slurry is produced which begins to break up after 3 to 4 minutes into a soft irregular granulation as the quaternary ammonium compound cools below its liquefaction tem-

perature. The product is unsatisfactory in that the granulation is greasy and easily compacted and difficult to handle.

The soft greasy granulation must be flaked with cold rolls. The flake produced is soft, pliable and distinctly greasy. The flake became brittle and non-greasy after about 24 hours of aging.

EXAMPLE IV

Three hundred grams of urea are charged into a mixing bowl such as a 5-quart Hobart mixer. Ninety grams of a quaternary ammonium compound, di-(hydrogenated tallow) dimethylammonium chloride and 7.5 grams of a second cationic surfactant liquefied by warming, are added to the urea charge and blended for about ½ minute at medium speed. A solution of 3.7 grams of an organic nonionic surface active agent in 60 grams of water containing a perfume and a soluble coloring dye is added to the blend of urea, quaternary ammonium compound and wetting agent while mixing is continued. The amount of water employed, together with the water inherently present in the ingredients corresponds to about 16% by weight of the mixture. An extremely heavy dough results almost immediately. Granulation of a mixture containing this much water is effected only with difficulty and if granulation does occur it generally reverts to a stiff dough with continued mixing. The product can be milled into a flake product only with difficulty and requires many passes between mill rolls accompanied by drying of the product before flaking occurs.

EXAMPLE V

The data in the following table shows the significance of the amount of water added during the mixing and reaction of the cationic nitrogen-containing compound and urea. The products were extracted with chloroform with the chloroform-soluble fraction indicating the proportion of cationic nitrogen-containing compounds that have not complexed with urea.

TABLE I

Active Ingredient	Percent Active Ingredient Based on Final Product	Amount of Water Added Based on Weight of Reaction Mixture	Percent Chloroform Solubles
Di-(hydrogenated tallow) dimethyl ammonium chloride	22	0	14.4
Do	22	1	11.6
Do	22	2	7.8
Do	22	4	1.9
Do	22	15	1.6
Hydrogenated tallow amine	18	0	7.4
Do	18	2	9.0
Do	18	4	3.3
Do	18	6	2.5
Do	18	8	1.8
Do	18	10	1.8

The di-(hydrogenated tallow) dimethylammonium chloride as sold commercially contains 75% active ingredient with the other 25% being about 6% water and 19% isopropyl alcohol. Therefore, in addition to the added water shown in the above table, the reaction mixture also contained 1.3% water and 4.2% alcohol introduced with the quaternary.

EXAMPLE VI

Chloroform-soluble values were determined on a variety of other products and are summarized in Table II. The amounts of water and alcohol shown are the total amounts present including whatever may be inherently introduced with the active ingredients. All of the products were formulated to contain approximately 22% by weight of the cationic nitrogen-containing compound.

TABLE II

Active Ingredient	In Reaction Mixture		Percent Chloroform Solubles
	Percent Water	Percent Isopropyl Alcohol	
Primary Amines:			
Hydrogenated-tallow amine	6	0	2.2
Coco amine	6	4	3.5
Arachidyl-behenyl amine	2	8	5.0
Secondary Amines:			
Di-soy amine	2		10.4
Di-(hydrogenated tallow) amine	2	6	17.2
Di-(arachidyl-behenyl) amine	2		16.7
Tertiary Amines: Di-methyl hydrogenated-tallow amine	4	5	4.8
Quaternary Ammonium Compounds:			
Tallow trimethyl ammonium chloride	3	4	0.7
Di-(hydrogenated-tallow) dimethyl ammonium sulfate	3	4	5.0
Dicoco dimethyl ammonium chloride	5	4	2.0
Di-soy dimethyl ammonium chloride	3	4	3.8
Diamines:			
Coco 1,3-propylene diamine	2		5.4
Tallow 1,3-propylene diamine	5		6.3
Oleyl 1,3-propylene diamine	10	5	7.3
Arachidyl-behenyl 1,3-propylene diamine	12		0.7

The importance of employing water in the specified proportions during the preparation of the fabric softener composition is apparent from the above as is the speed with which the solid fabric softener of the invention can be prepared.

The fabric softener compositions of the invention lend themselves to packaging and packages may be made of sealed containers containing any desired amounts of such compositions which, being in dry form, may be readily distributed or shipped without difficulty. During such shipment they do not pack or compress or cake and are directly available without further preparation for use in treating fabrics to improve the softness and feel characteristics thereof. Moreover, the fabric softener compositions can be prepared at ambient temperatures which is an additional advantage.

Those modifications and equivalents which fall within the spirit of the invention and the scope of the appended claims are to be considered part of the invention.

I claim:

1. A process of preparing a stable solid water-dispersible composition useful for treating fabrics to improve the softness characteristics thereof which comprises reacting at substantially ambient temperatures in the presence of water a major amount of urea ranging from about 50 to 90 percent by weight of the composition and a cationic nitrogen-containing compound containing one to two straight-chain organic radicals containing from 8 to 22 carbon atoms and having fabric softening properties, the amount of water present in the reaction mixture corresponding to from about 2 to about 15 percent by weight of the composition.

2. A process of preparing a stable solid water-dispersible composition useful for treating fabrics to improve the softness characteristics thereof which comprises reacting at substantially ambient temperatures in the presence of water a major amount of urea ranging from 50 to 90 percent by weight of the composition with a cationic nitrogen-containing compound containing one to two straight-chain organic radicals containing from 12 to 22 carbon atoms and having fabric softening properties, the amount of water present in the reaction mixture corresponding to from about 3 to about 5 percent by weight of the composition.

3. A process of preparing a stable solid water-dispersible composition useful for treating fabrics to improve the softness characteristics thereof which comprises re-

acting at substantially ambient temperatures in the presence of water a major amount of urea ranging from about 50 to 90 percent based on the weight of the final composition and about 4 to 40 percent based on the weight of the final composition of a cationic nitrogen-containing compound containing one to two straight-chain organic radicals containing from 8 to 22 carbon atoms and having fabric softening properties, the amount of water present in the reaction mixture corresponding to from about 2 to about 15 percent by weight of the reaction mixture, agitating said reaction mixture for a period of time sufficient to produce a solid reaction product, removing said solid reaction product and flaking the solid reaction product to produce flakes.

4. A process of preparing a stable solid water-dispersible composition useful for treating fabrics to improve the softness characteristics thereof which comprises reacting at substantially ambient temperatures in the presence of water a major amount of urea ranging from about 50 to 90 percent based on the weight of the final composition, about 4 to 40 percent of di-(hydrogenated tallow) dimethylammonium chloride, the amount of water present in the reaction mixture corresponding to from about 2 to about 15 percent by weight of the reaction mixture,

agitating the said reaction mixture for a period of time sufficient to produce a solid reaction product, and flaking the solid reaction product to produce a finished flaked product ready for use.

5. A process of preparing a stable solid water-dispersible composition useful for treating fabrics to improve the softness characteristics thereof which comprises reacting at substantially ambient temperatures in the presence of water a major amount of urea ranging from about 50 to 90 percent based on the weight of the final composition with hydrogenated tallow amine, the amount of water present in the reaction mixture corresponding to from about 2 to about 15 percent by weight of the composition.

6. The product resulting from the process of claim **1.**

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JULIUS GREENWALD, *Primary Examiner.*

J. T. FEDIGAN, *Assistant Examiner.*