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[54] ENCAPSULATED FABRIC SOFTENER

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[57] ABSTRACT

A fabric softener for treatment of fibrous materials, preferably in laundry being dried, is in particulate form, with a protective coating over it which is abraded, ruptured, dissolved or melted during contacts with moving laundry, so that a gradual release of softener to the laundry is effected, avoiding overapplication of such compounds to particular locations on the materials and preventing objectionable staining of treated materials by applications of excessive amounts of softener.

8 Claims, No Drawings

ENCAPSULATED FABRIC SOFTENER

This invention relates to softening of fabrics and in particular, to the softening of laundry in automatic clothes dryers by means of a fabric softener which includes a fabric softening compound coated with a protective and release controlling material. The invention is of the novel encapsulated fabric softeners and methods in which they are employed. With respect to some fabric softeners, the softening action referred to herein is also accompanied by antistatic activity, which will not usually be explicitly referred to in this specification.

The addition of conditioning chemicals in wash and rinse waters and their sorption by laundry, fibrous materials or fabrics being treated or washed are known. A recognized disadvantage of using a fabric softener in an automatic laundry rinsing operation is that the consumer has to be present when the machine reaches the rinsing cycle. Also, only a minor proportion of the various available softening materials is sufficiently substantive to materials being rinsed so as to enable them to be satisfactorily employed and exert desired softening effects. Many other such materials will be washed out with the rinse water, doing little to soften the fabrics. Similarly, in today's average wash load, including cottons and a variety of synthetics and blends, the fabrics employed exert different attractive forces and possess different substantivities with respect to the softening chemicals and therefore, the treated fabrics may be softened differently. When applied in the wash water, some softeners interact objectionably with other detergent constituents and in many cases the detergent counteracts or prevents substantive action.

Because of the inconvenience and inefficiency of fabric softeners which have to be added to the wash water or rinse water, other techniques have recently been developed to allow for depositing a fabric softener onto laundry or other fabrics being subjected to a drying operation. By following such a method, the consumer does not have to be present at a particular time in the washing or rinsing cycle and merely adds the conditioning material to the laundry in the dryer at the time the dryer is charged. During the drying operation, the fabric softener contacts the materials being treated and is deposited in a thin film on the surfaces thereof. Similar effects are obtained when the softener is not spread on the fabric in a continuous film but is deposited in a series of locations, sufficiently well distributed over the fabric to effectively soften it and yet, not of such a thickness in any location as to be noticeable. Unfortunately, some softening agents, such as the quaternary ammonium halide fabric softeners, tend to be strongly substantive to the fabrics treated and once deposited thereon in objectionably large quantities, tend to remain at the site of deposit and form oily spots or other objectionable discolorations, which are especially apparent on light colored materials and which are not easily removed during tumbling of the fabric in the dryer. In some case, such spots are even difficult to remove by a subsequent washing operation.

To overcome some of the difficulties of employing highly substantive and staining softeners, various techniques have been suggested. Plasticizers have been mixed with the softeners to reduce their substantivities and make them more readily removable on subsequent washings. Different softeners have been employed, such as anionic, nonionic and amphoteric surface active materials, which do not form such tight bonds to

the fabrics being treated. Aerosol sprays have been used which distribute the softening ingredients over the fabrics as a fine spray, preventing excessive deposits of any portions of the fabrics. Mechanical devices have been designed to release solutions of treating agents slowly through valves or outlets during the tumbling of the fabrics in the automatic laundry dryer. Although these methods have been helpful in overcoming the problem associated with uneven application of fabric softeners to materials being treated in the dryer, each requires either formulation changes, physical form modifications or the utilization of mechanical devices. By means of the present invention, a highly convenient controlled application of softener may be made and the softening agent may be any of the wide variety of suitable materials. The products of this invention are easy to employ, comparatively simple to manufacture, inexpensive and give good results. They are in an attractive form, are stable on storage, are dry and can be so designed as to release softening agent at a convenient time in the drying cycle or continuously, when so desired, and permit the use of different normally incompatible conditioners.

In accordance with the present invention a fabric softener for the treatment of fibrous materials comprises at least one fabric softening compound coated with a suitable non-staining protective and release controlling material, normally an organic polymer. The fabric softener will normally be spherical in form and the protective polymer will usually be a water soluble colloid or synthetic plastic. The fabric softener particles will generally be small, usually spherical and from 0.020 to 5 mm. in diameter but, to keep them from being prematurely blown out through the exhaust in some high speed air flow dryers, they may be attached to suitable sized substrates. The invention also relates to a method of controlling the releasing of a fabric softening compound and contacting a fabric to be softened with it gradually by tumbling the fabric at an elevated temperature with encapsulated softening compound in which the coatings are of suitably different susceptibilities to releasing their contents under dryer conditions. The fabric softening compound utilized in the present fabric softeners may be any of such suitable materials or mixtures thereof known in the art to exert useful softening, and often, anti-static effects on textiles. Among such materials, the surface active softeners are those which are preferably employed, including cationic, anionic, nonionic and amphoteric compounds. Of these, the cationics appear to have best softening activities and are antistatic. In addition, they present the most serious problems with respect to stainings of the substrates when they are applied in excess.

Of the cationic compounds those which are quaternary ammonium salts are excellent softeners. Such compounds are mentioned in U.S. Pat. No. 3,442,692 and therefore, a lengthy compilation of them is not given here. In addition to the quaternary ammonium salts, corresponding quaternary phosphonium compounds may also be employed. The quaternary compounds will usually contain a plurality of lower alkyl groups on the quaternary atom, for example, on the nitrogen atom, and one or two higher alkyls, benzyls or equivalent groups thereon. Salt-forming ions will preferably be a halogen, such as chloride or bromide ion, but may also be any other useful solubilizing group. A preferred quaternary compound that has been used

very successfully is distearyl dimethyl ammonium chloride but other quaternaries of similar activity are also acceptable, including cetyl trimethyl ammonium bromide, dimethyl dilauryl ammonium chloride, diethyl distearyl ammonium chloride, dimethyl di- (hydrogenated tallow alkyl) ammonium chloride, stearyl dimethyl benzyl ammonium chloride and lauryl methyl dibenzyl ammonium bromide. Also useful are various other cationics, such as alkyl pyridine salts, alkyl imidazolines, higher alkyl amines of the primary, secondary or tertiary types and higher alkyl guanidine salts, e.g., 1-methyl-1-stearyl aminoethyl-2-stearyl imidazolinium methyl sulfate, stearyl pyridinium halides, cetyl isoquinolinium bromide and higher alkyl morpholinium chlorides. In the aforementioned cases lower alkyl is of 1 to 5 carbon atoms, preferably 1 or 2 carbon atoms, and higher alkyl is of about 8 to 20 carbon atoms, preferably 12 to 18 carbon atoms. Although mixtures of the cationic softeners and/or antistatic agents with nonionic or amphoteric softeners may be employed, before encapsulation such mixtures with anionics will usually be avoided, due to objectionable chemical interactions which often result.

The amphoteric conditioners which have been found to be most useful are the complex fatty amido compounds, such as the Soromines, e.g., Soromine AT and Soromine AL, sold by GAF Corp. Various other amphoteric compounds, usually possessing primarily cationic properties under final use conditions, include the higher alkyl beta-alanines, the N-higher alkyl taurines, e.g., reaction products of lauryl amine with sodium isethionate, the N-higher alkyl aspartic acids and the Miranols, described in U.S. Pat. No. 2,528,378. Related compounds which are useful include the zwitterionic surface active agents such as betaine, betaines, sulfobetaines and similar surfactants which form inner salts.

The anionic surface active conditioners sometimes possess more antistatic activity than softening effect but are validly considered to be softening agents, nevertheless. They include compounds such as the water soluble salt of the sulfuric reaction products which have an alkyl radical of from about 8 or 10 to about 18 or 20 carbon atoms and either a sulfonic acid or sulfuric acid ester radical. The corresponding compounds containing acyl groups of similar chain lengths are also satisfactory. Representative of these compounds are higher alkyl sulfates of from 10 to 20 carbon atoms, preferably of 16 to 20 carbon atoms; higher alkyl benzene sulfonates, preferably the linear alkyl benzene sulfonates wherein the alkyl group is of 10 to 18 carbon atoms, preferably of 12 to 15 carbon atoms; higher fatty acyl taurides and isethionates; higher fatty acyl glycidates and sarcosides; higher fatty acid monoglyceride sulfates and sulfonates; higher fatty glycerol ether sulfonates; sulfuric acid esters of reaction products of 1 mole of higher fatty alcohol with from one to six moles of lower alkylene oxide; and the alkyl phenyl lower alkylene oxide ether sulfates containing from 1 to 10 moles of lower alkylene oxide per molecule. Specific examples of such compounds include sodium tallow alcohol sulfate, sodium hydrogenated tallow alcohol sulfate, sodium lauryl sulfate, triethanolamine n-hexadecyl sulfate, trimethylamine cetyl sulfate, potassium n-octadecyl sulfonate, sodium coconut oil fatty acids monoglyceride sulfate, sodium N-lauroyl sarcoside, sodium n-dodecyl benzene sulfonate, sodium tet-

radecyl toluyl sulfonate, nonyl phenyl polyoxyethylene sulfate wherein the polyoxyethylene group is of 5 moles of ethylene oxide, sodium dodecyl glycerol ether sulfonate and potassium oleyl N-methyl tauride. In addition to those anionic conditioners, conventionally referred to as synthetic anionic organic detergents, water soluble higher fatty acid soaps, which technically are in the same class, may also be employed. These are usually the alkali metal, e.g., sodium salts of higher fatty acids of 8 to 20 carbon atoms preferably of 12 to 18 carbon atoms and are normally derived from natural sources, such as coconut oil, palm oil, corn oil, tallow, greases and mixtures thereof. However, trialkanolamine and trialkylamine salts, such as trialkanolamine soaps, may be used, as may be other known soluble soaps suitable for the present purposes. Exemplary of such materials are the sodium soap of an 85:15 mixture of tallow and coconut oil fatty acids, the potassium soap of stearic acid, the mixed sodium and potassium soaps of a 50:50 mixture of tallow and coconut oil fatty acids, sodium "cocate", potassium stearate, triethanolamine stearate and sodium laurate. In addition to water soluble salts, the water insoluble soaps of calcium, magnesium, lithium and "heavy metals" may also be used but it is normally preferred to employ the water soluble products.

Of the anionic materials mentioned it is most preferred to use either the higher alkyl sulfates, sarcosides or water soluble soaps of higher fatty acids. For example, hydrogenated tallow alcohol sulfates, usually as sodium salts, and sodium cocotallow soaps comprising 85% tallow and 15% coconut oil soaps, optionally with a portion of the sodium, e.g., from 10 to 40%, being replaced by potassium, are among the preferred compounds.

Of the nonionic surface active materials which are useful as fabric conditioners, some preferred embodiments include the polyoxy-lower alkylene higher alkyl ethers, e.g., polyoxyethylene lauryl ether having four ethoxy groups (Brij 30); higher alkyl phenoxy poly(lower alkoxy) lower alkanols, e.g., nonyl phenoxy polyethoxy ethanol (Igepal CO-880); and balanced hydrophilic-lipophilic compounds made by the condensation of lower alkylene oxides with an organic hydrophobic material, e.g., Ucons Pluronics. The nonionic softeners usually include lipophilic groups having higher alkyl components, generally of 8 to 20 carbon atoms, and hydrophilic components which are poly-lower alkylene oxides, often having 4 to 20 moles of lower alkylene oxide per mole. The lower alkylene oxides are of 2 to 3 carbon atoms, preferably being ethylene oxide. Other useful nonionic softeners are the amine oxides and the alkanolamides. The former are usually higher alkyl di-lower alkyl amine oxide wherein the higher alkyl is 8 to 20 carbon atoms and lower alkyl is of 1 to 4 carbon atoms, preferably being methyl. The alkanolamides may be mono- and di-lower alkanolamides of the higher fatty acids, e.g., myristic diethanolamide, lauric myristic diethanolamide and palmitic monoethanolamide.

Various other softening and anti-electrostatic charge conditioning agents are found listed in DETERGENTS AND EMULSIFIERS, 1969 Annual Edition, by John W. McCutcheon, who classifies the various surface active agents as anionic, cationic, nonionic and amphoteric and lists those having good fabric softening and antistatic properties. In the description herein and in the listings, the higher alkyls will usually be of 8 to 20

carbon atoms and lower alkyls will be of 1 to 4 carbon atoms, with the preferred compounds often having higher alkyls of 12 to 18 carbon atoms and lower alkyls of 1 or 2 carbon atoms, unless otherwise indicated.

Various mixtures of the softening agents may be utilized, but, as was mentioned, it is highly preferable to avoid mixing anionic and cationic softening compounds before encapsulation. However, after encapsulation, mixtures of such softeners may be employed successfully.

In addition to the surface active softening and antistatic compounds, other such materials which are of waxy, film-forming, coating, lubricating, or chemical properties so that they affect the textiles to which applied and soften them or render them antistatic may also be employed. Among such compounds are synthetic organic plastics, waxy lubricants, e.g., paraffinic materials, mono-, di- and triglycerides, preferably modified to make them less lipophilic, and synthetic waxes. Some of such materials may sometimes be considered to be surface active nonionics but generally, due to their low solubilities in the solvents employed, they will be considered to have little surface activity in the normal sense.

The softeners will preferably be in a solid state at room temperature and in a semi-plastic state or liquid at dryer temperatures. However, due to the use of encapsulation, it is possible to employ liquid conditioning agents, as well as those which fuse at the dryer conditions or which dissolve in the aqueous medium in the dryer.

To encapsulate or coat the softening compounds any of a wide variety of materials may be used. In some cases, other softening compounds than those of the central portions of the fabric softener capsules are employed, providing that they do not cause staining of the fabrics being treated. Thus, it may be desirable to utilize higher fatty acid alkanolamides, solid polyoxyalkylene waxes or nonionic agents, or higher fatty acid soaps to form a suitable non-staining protective and release controlling coating. Although such materials are useful, it is found that best results are obtainable when the coating is an organic polymeric material, such as a colloidal agent or a synthetic organic plastic. An almost limitless number of such substances may be used, including the natural and synthetic organic gums and inorganic gum-like materials, and the synthetic organic water soluble and water insoluble plastics. Of the gums, representative types include alginates, gelatins, starches, cellulosic compounds, carageenan, tragacanth, agar agar, carob bean, locust bean, and various other resinous and gum-like extracts from plant and animal matter and derivatives of these. The synthetic gums and thickeners include sodium carboxymethyl cellulose, hydroxypropyl methyl cellulose, hydroxyethyl cellulose, polyvinyl pyrrolidone, polyacrylamide, modified starches, starch glycolates, and various other known cellulosic, starch and amylopectin derivatives having thickening and film-forming properties. The water soluble polymers include polyvinyl alcohol, polyacrylamide and polyvinyl acetate, the latter being hydrolyzable to the alcohol to a significant extent. Corresponding allyl compounds and polyacrylic acids are also in this group, as are various mixed derivatives of such compounds. Finally, other film formers which are essentially water insoluble include polyvinyl chloride, chlorinated PVC, polyvinylidene chloride, polyethyl-

ene, polypropylene, polymethacrylates, nylons, polyurethanes, silicones, ABS, polyesters and polyethers. Such materials and equivalents are described in greater detail in any conventional handbook on synthetic organic plastics, for example, in *Modern Plastics*, Encyclopedia Volume, Vol. 47, No. 10A (for 1970-1971), at pages 768-787. In addition to the synthetic organic plastics, natural water insoluble film forming materials such as rubbers, shellacs and other resins may be used.

Softening compounds are coated with a selected protective release controlling agent or a combination or mixture of such agents by any of the well known encapsulation methods. These are described at length in an article by Herman Nack entitled *Microencapsulation Techniques, Applications and Problems*, which appeared in the *Journal of the Society of Cosmetic Chemists* at Vol. 21 pages 85-98 (Feb. 4, 1970). In addition to the various processes described therein, of which aqueous phase separation, non-aqueous phase separation, interfacial polymerization, multi-orifice rotating cylinder, fluidized bed spray coating, melt prilling in a fluidized bed, spray drying diffusional exchange and meltable dispersion techniques are representative various other coating methods known in the art may also be used, especially when the capsules are in the larger range of sizes described in the specification. The selection of the process to employ will depend on the types of softening compounds and coatings used and these in turn, will vary, depending on the particular application of the product intended and how the release of softening compound is to be regulated. For example, where a normally liquid softening compound is employed the encapsulation method will usually be different from that when such a compound is a solid, because liquids cannot be coated by the simpler encapsulation techniques known. Also, if thick coatings are to be made, different techniques will usually be applied than will be the case when thinner or partial coatings are desired. In some cases it may be desired to produce secondary films about the primary coatings on the capsules to further modify the susceptibilities of the capsules to rupture and release of conditioning agent. The means for accomplishing these ends are set forth in the article described and from it it is considered to be evident which methods of coating may be employed in a particular application. In addition to the mentioned article other encapsulation methods may be found in the patent literature, of which U.S. Reissue Pat. No. 24,899 and German Application No. 1,268,316 may be considered as representative.

The particles of fabric softening compound or composition are of such size that after coating with protective or release controlling material they will be of a suitable diameter or thickness for application to fabrics in an automatic laundry dryer or similar machine. Usually, the coated particles will be from 0.020 to 5 millimeters thick, preferably from 1 to 4 mm. in diameter, and will be spherical. In most cases, the particles will be entirely coated with organic polymeric material having a thickness of about 0.001 to 2 mm. and preferably 0.2 to 1 mm. However, the coating may be of uneven thicknesses in the mentioned ranges, if desired, and only a part or parts of the fabric softening compound may be coated. In cases where partial coating is practiced the uncoated portions will preferably be between coated sections so that contact of the particle with damp laundry or other fabric will be with the coating,

not directly with the fabric softener compound. Generally, only a small proportion of the fabric softener will be uncoated, usually to facilitate release of the fabric softening compound when that is desired. In almost all cases, at least 40% of the surface will be coated and usually more than 80% is coated, with it being highly preferable to coat the entire surface. Of course, the coatings will be of sufficient strengths and hardnesses so as to be maintained on the particles during storage and shipment and to prevent fusion or caking.

Because in some applications wherein air flows through a drying machine are high and the fabric softener capsules are of low density and small, they might tend to be swept out through the air exhaust opening, it is sometimes desirable to agglomerate the encapsulated materials into larger masses which will resist being discharged from the dryer before application of the fabric softening compounds to the materials to be treated. In such cases, the capsules or spheres may be adhered together with the use of an adhesive or by fusion of portions of the coatings, or they may be joined to a carrier, such as flexible or inflexible materials, e.g., paper, polyurethane foam strips, synthetic plastic spheres or other shaped articles, or metal screens. Such materials will preferably have a density greater than 1.5 grams/c. cm. and will have an external surface area of from 0.1 to 500 sq. cm., preferably from 1 to 50 sq. cm. Also preferably, the capsules will be adhered to the surfaces of such carriers, rather than to interior portions thereof.

Although the shaping of the carrier material and the very fact that the capsules are adhered to it will control to some extent the rate of release of fabric softening compound from the capsules, other techniques may also be employed. For example, the sizes of the capsules may be controlled, the proportion of softening compound in the capsule may be varied, the types of coating materials may be chosen to regulate release and varying thicknesses of coatings may be employed. For controlled release of softening agent so that a gradual dispensing of it is obtained during the entire drying period, different thicknesses of coating within the described ranges may be intentionally utilized. Of course, the thinner coatings will be ruptured first, releasing softening compounds, and afterward, the thicker coatings will be broken. Such actions will prevent agglomeration of softening compound into large masses which might otherwise leave visible stains on the materials being treated. In a similar fashion, the uses of different polymeric coatings on different portions of the fabric softener capsules will have substantially the same effect. Thus, more highly water soluble or fusible materials or more readily fractured materials will release fabric softening compound earlier than the more hydrophobic and stronger coatings. To obtain a longer sustained release period, thin, water soluble or weak coatings may be utilized, together with thicker, more resistant coatings. The distribution of less and more readily rupturable, soluble, abradable and fusible coatings on the fabric softening compounds will preferably be such as to yield a uniformly releasable (with respect to time) active material. However, if otherwise desired, release may be controlled so as to be at a particular time in the drying operation.

The ratio of softening compound to coating material may be varied over wide ranges but for most efficient use it is preferred that at least as much softening com-

pound be present as of the coating thereon. Generally, from 1 to 50 times as much softening compound will be employed as of coating material and preferably this will be from 3 to 10 times as much.

In addition to controlled release of softening compounds, the present encapsulated fabric softeners possess other special advantages. For example, due to encapsulation of the softeners, incompatible softening materials may be utilized in the same composition and may be released at different times in the softening operation. Particular sequences of applications of different softening agents may be obtained. The coating material may itself have softening properties or may yield other desirable effects when applied to the fabrics to be treated. Thus, when the colloidal gums are utilized, they may have a bodying effect on the fabrics to counteract some excessive limpness often resulting from the use of softening compounds. They may facilitate the release of subsequently deposited soil from the treated fabrics. They may have bactericidal or other treating agents intentionally included in the coating compositions to impart additional properties to the treated fabrics. Such agents are useful even if they would be incompatible with the softener, if mixed with it. The incompatible materials can be compatible when deposited on fabrics, due to the fabrics tying up at least one of them, although they would not be capable of existing in the same softener composition unless insulated from contact.

The present fabric softeners are used by adding them to a load of fibrous materials to be conditioned and tumbling such materials with the encapsulated softeners until the fabric softening compounds penetrate the coatings and are applied to the materials to be conditioned, which subsequently spread them over other such materials by repeated contacts with them. Because the capsules are at different positions when ruptured or otherwise penetrated, the softening compound is not applied in excessive quantities to any particular locations and because releases are not simultaneous, staining is prevented.

The preferred machine for employment as a tumbling and conditioning device is an automatic laundry dryer and preferably the fabrics treated are in laundry to be softened. Such dryers contain a tumbling drum which revolves at a speed of about 10 to 60 r.p.m., and has means for circulating air through the drum, which air may be heated. Generally, the tumbling and drying operation continues for from five minutes to an hour, and preferably from 15 to 45 minutes. In this time, there may be an initial period in which the drum is unheated or is heated without sufficient air circulation to carry out the small particles of fabric softener. Such period is followed by a more intense heating, during which moisture is removed from the laundry, which normally has been washed shortly before. It is during the drying period, usually the latter part thereof, that the coating material, now dry and usually non-adherent to the fabrics being treated, is carried out of the dryer with the exhaust air. After completion of the drying and fabric softening, the flow of hot air is halted for the dryer is cooled by having unheated air flow through it, as may be done for permanent press materials. The hot drying air employed is usually in the temperature range of 40° to 95°C., preferably from 50° to 75°C. Of course, while the fabrics contain moisture in equilibrium with gas being removed, they will be at the wet bulb tempera-

ture, generally lower than that of the drying air. In those cases wherein the fabric softener might otherwise be carried out with the gases, an initial heating period of from 1 to 20 minutes, preferably from 1 to 5 minutes may be used (before much drying air is permitted to pass through the laundry) so as to facilitate rupturing of the capsules and distribute the softening compound over the fabrics. Such preliminary heating period may then be followed by a normal 5 minutes to 1 hour drying period wherein the drying air is discharged from the dryer, containing moisture removed from the laundry.

The materials to be treated with the softeners include all those now in current use for clothing items such as shirts, trousers, dresses, blouses and skirts and for other household items such as towels, sheets, diapers and furniture and cushion covers. For the most part, these materials are cottons or synthetic fibers of the nylon, polyester, acetate, rayon, and acrylic types and include blends of the synthetics, usually with cotton. The main need for softening is with respect to cottons and blends of cotton and synthetics and the synthetic products themselves are most improved with respect to reductions of their tendencies to accumulate static electrical charges. Concentrations of softening compounds to be deposited on the materials being treated will usually determine the quantities of capsules to be employed. In terms of weights applied, the softening agents will normally be deposited to the extent of 0.00001 to 0.5 g./sq. cm., preferably from 0.0001 to 0.005 g./sq. cm. Typically, from 0.1 to 50 grams, more usually from 1 to 10 grams of softening compound will be used to treat about 10 lbs. of laundry.

The following examples illustrate but do not limit the present invention. All parts are by weight and all temperatures are in °C., unless otherwise indicated.

EXAMPLE 1

One hundred and fifty parts of distearyl dimethyl ammonium chloride (Arquad R-40, manufactured by Armour Industrial Chemical Company, 40% active), twenty parts of polyoxyethylene lauryl ether having four ethoxy groups per mole (Brij 30, mfd. by Atlas Chemical Corporation) and one part of perfume are blended together homogeneously and are encapsulated by a coacervation technique which results in the production of spherical microcapsules with diameters regularly distributed over the 0.1 to 3 millimeter range, with a thickness of sodium carboxymethyl cellulose coating thereon about one-fifth the diameter, ranging from 0.02 to 0.6 mm.

The microcapsules are joined to the exterior of an open celled polyurethane foam sponge strip measuring about 5 cm. × 10 cm. × 1 cm. and having crushed cells therein of about 0.2 cm. diameter, so that approximately 7 grams of the mixture of fabric softening compound is present on the sponge, with about 2 grams of coating material also present.

A load of mixed laundry, just washed, and comprising 8 pounds of anhydrous laundry and an additional 6 lbs. of water therein is charged to an automatic laundry dryer of the horizontal rotating drum type. The laundry is approximately half cotton and half synthetic fibers, including cotton-synthetic blends. Among the synthetics charged are permanent press treated polyester-cotton blends of 65-35 parts proportion, Dacron and nylon, with the permanent press items charged being the greater proportion of the synthetics. Some of the

materials are light blue in color, on which oily deposits of materials, such as excessive quantities of cationic softeners, are easily discerned, especially after subsequent heating and pressing. After loading the laundry to be dried, softened and made static-free, the polyurethane strip containing the microcapsules of softener compounds is placed on top of the laundry and the dryer is immediately set into motion.

Drying air at 70°C. is admitted to the dryer and the dryer is turned at about 30 r.p.m. As the tumbling of the sponge with the laundry continues, the microcapsules are ruptured by dissolving and expansion of the sodium carboxymethyl cellulose coating thereon and the softening compounds are released from the capsules continuously, over a period of about 5 minutes. The release continues over this period because of the different thicknesses of the coating agent on the softener core. The sodium carboxymethyl cellulose is also released and both it and the softening compounds are initially transferred to the damp laundry with which the sponge is contacted and are subsequently transferred to other pieces of laundry.

Drying is continued for a total of 45 minutes, after which the laundry is cooled by addition of unheated air and continued movement of the drum for another 5 minutes.

Upon completion of the drying-softening operation the laundry is found to be soft and does not possess objectionable static charges. There are no objectionably apparent stains on the laundry, even on the light blue items which are most susceptible to greasy staining from softening compounds, such as cationic softeners. The coating agent adds bodying and soil-release properties to the treated laundry, but, because it tends to dry out and flake more than the softeners, it is partially removed from the laundry during the drying operation, so as not to have present on the laundered items an excess of bodying agent to the point where the clothes become objectionably stiff in parts.

When the same experiment is repeated, using unencapsulated particles of softening materials, maintained cold enough to be solids and size reduced to particles as small as the cores of the encapsulated materials, with care being taken to prevent the conditioners from being immediately removed in the exhaust from the dryer (they are not held to a substrate) the light blue articles being laundered show some greasy stains, apparently from agglomeration of the powders in the dryer and depositing of excess amounts of softeners at particular locations, from which they are not sufficiently transferred to other portions of the fabrics.

When the experiment of Example 1 is run, utilizing the same softener composition formulation but coating cores thereof with different polymeric materials, half the particles being coated with polyvinyl alcohol and half with polyvinyl chloride, similar results are obtained, although the different coating agents are ruptured at different times, with the polyvinyl chloride coating being most stable. In such products the thickness of the polyvinyl chloride coating is about 0.1 to 0.5 mm. and that of the polyvinyl alcohol is from 0.02 to 0.3 mm.

Also, when the procedures are repeated with other softening compositions of the formulas of Examples 2-7, good softening is obtained, without staining, while some losses of softening material and stainings occur

when the powdered or liquid softening agents are used without prior encapsulation.

When, instead of following the technique described, the different conditioning agents are separately encapsulated, either by suitable coacervation methods or by other coating techniques, such as by spraying solutions of coating materials onto the surfaces of finely divided powders, e.g., CMC on distearyl dimethyl ammonium chloride, and CMC on Brij 30, using the same proportions of active materials, results like those described above are obtained but it appears that the presence of the mixture of the cationic and nonionic agents in the same capsule results in a better distribution of the cationic on the treated fabrics and therefore, is preferable to avoid staining.

EXAMPLE 2

	Parts
Triethanolamine stearate	7.0
Soromine AT (complex fatty acids amphoteric softener, made by GAF Corp., 20% active)	2.5
Perfume	0.05

EXAMPLE 3

Sodium tallow alcohol sulfate	30
Brij 30	20
Perfume	0.5

EXAMPLE 4

Stearyl dimethyl amine oxide	38
Pluronic F-68 (block copolymer of polyoxyethylene, polyoxypropylene and propylene glycol)	10
sodium N-lauroyl sarcoside	25
Perfume	1

EXAMPLE 5

	Parts
Sodium soap of 80 tallow-20 coconut oil mixture	88
Lauric myristic diethanolamide	50
Perfume	1

EXAMPLE 6

Igepal CO-880 (nonylphenoxy polyethoxy ethanol, made by GAF Corp.)	25
Tween 61 (polyoxyethylene sorbitan monostearate, made by Atlas Chemical Industries)	50
Perfume	1

EXAMPLE 7

Stearyl dimethyl amine oxide	76
Culversoft WS-30 (cationic fatty amidoalkyl ammonium chloride, made by Culver Chemical Co., 30% active)	100
Perfume	2

The above compositions of Examples 2-7 are encapsulated by spraying coating particles with hydroxypropyl methyl cellulose, gelatin (hardened) and polyvinyl acetate, in different operations, so that the eighteen encapsulated products obtained are of about 2 millimeters diameter and have coating thicknesses of about 0.5 mm. The particles are within the 0.1 to 3 mm. range and the coating thicknesses are about one-fourth to one-sixth the diameters. The coating materials are about one-third the total weights of the capsules. Instead of utilizing the spray coating method, the encapsulation methods described in the Nack article or the German patent application previously described are utilized. Also, in other products the proportions of coating and core materials are varied within the ranges described in the specification and the individual softeners of the formulas are used alone with the various coating materials, instead of in multiple component compositions.

Mixed laundry charges of the type described in Example 1 are treated according to the method mentioned therein but with the difference that the encapsulated materials are not held to a substrate in some of the experiments. Because of this distinction, when no holding substrate is utilized the initial part of the drying operation, the first 3 minutes thereof, is conducted without air passing through the dryer. By this method, the encapsulated particles are distributed throughout the clothing and are less apt to be carried out the exhaust after drying with air throughput commences.

The fabric softening treatments are effective and objectionable staining does not result when using the encapsulated softeners. Some of the PVA remains on the laundry and acts as a soil-repellant and bodying agent but almost all of the PVC is carried out with the exhaust air.

When a substrate is employed, it is a formed nylon sphere, having roughened edges and having the encapsulated softener fastened to it by a suitable adhesive, e.g., casein glue, epoxy resin cement, and excellent softening and antistatic actions on the fabrics are obtainable without staining. The extent of softening is controllable by increasing or decreasing the proportion of capsules charged to the dryer, for example, the weight used may be such that from 2 to 10 grams of softening compound are used rather than 7 grams thereof, and the result will be a correspondingly decreased or increased softening and antistatic effect.

The invention has been described with reference to specific examples and illustrations but it is appreciated that it is not limited to them since it will be clear to one of skill in the art that equivalents and alternatives may be utilized in place of ingredients of the compositions and steps in the process without departing from the spirit of the invention.

What is claimed is:

1. A fabric softener in particulate capsule form for the treatment of fibrous materials which consists essentially of at least one fabric softening compound selected from the group consisting of quaternary ammonium fabric softeners, alkyl pyridine salts, alkyl imidazoles, higher alkyl primary, secondary or tertiary amines, higher alkyl guanidine salts, higher alkyl morpholinium chlorides, higher alkyl beta-alanines, N-higher alkyl taurines, N-higher alkyl aspartic acids, betaines and sulfobetaines suitable for treatment of damp

laundry to soften it and improve the anti-static properties thereof when the laundry contains materials which are prone to the accumulation of static charges, said softener having a coating of a water-soluble or dispersible organic polymeric colloid material, said coating being from 0.001 to 2 millimeters thick over at least 40% of the surface of said softening compound, the capsule particles having a thickness of from 1 to 4 millimeters.

2. A fabric softener according to claim 1 wherein the quaternary ammonium fabric softener is selected from the group consisting of distearyl dimethyl ammonium chloride, dimethyl dilauryl ammonium chloride, diethyl distearyl ammonium chloride, dimethyl di-(hydrogenated tallow alkyl) ammonium chloride and stearyl dimethyl benzyl ammonium chloride and the alkyl pyridine salt is a stearyl pyridinium halide.

3. A fabric softener according to claim 1 wherein the capsules are held to a flexible carrier material having an external surface area of 0.1 to 500 sq. cm.

4. A fabric softener according to claim 1 wherein the fabric softener particles have coating thicknesses thereon distributed over a range of thicknesses so that a gradual release of the softening compound is obtained as the coatings are abraded, ruptured, dissolved and/or melted, when brought into repeated contacts with fabrics to be softened.

5. A fabric softener according to claim 1 wherein a

plurality of organic polymeric colloid materials is used to coat the fabric softening compound(s) and such organic polymeric coating materials are of different susceptibilities to abrasion, rupture and/or solution fusion when brought into repeated contacts with fabrics to be softened, so that the controlled or gradual release of softening compound is obtained as the coatings are abraded, ruptured, dissolved and/or fused.

6. A fabric softener composition according to claim 1 wherein said particulate capsules are in the form of fused agglomerates.

7. A fabric softener according to claim 1 wherein the fabric softening compound is a di-higher alkyl di-lower alkyl ammonium halide and the coating material is a suitable material selected from the group consisting of sodium carboxymethyl cellulose, hydroxypropyl methyl cellulose, hydroxyethyl cellulose, polyvinyl alcohol, polyvinyl pyrrolidone, polyvinyl acetate, polacrylamide and polyacrylates.

8. A fabric softener according to claim 7 wherein the fabric softener particles have coating thicknesses distributed over the range of 0.2 to 1 mm. so that a gradual release of softening compound is obtained as the coatings are abraded, ruptured, dissolved and/or melted when brought into repeated contact with fabrics to be softened.

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