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(54) Title: METHODS FOR ODOR CONTROL AND CONCENTRATED, NON-LIQUID COMPOSITIONS THEREFOR

(57) Abstract: A method of reducing malodor on fabrics in a wash or rinse solution of a laundry process. The method comprises the step of contacting said fabrics with an effective amount to reduce malodor on said fabrics of an odor control agent selected from the group consisting of: uncomplexed cyclodextrin; odor blocker; class I and/or class II aldehydes; flavanoid; metallic salt; and mixtures thereof. Concentrated, non-liquid additive compositions are used in the methods herein. The concentrated, non-liquid compositions include powders/granules, tablets, gels, pastes, substrates/sheets, and laundry bars.

# METHODS FOR ODOR CONTROL AND CONCENTRATED, NON-LIQUID COMPOSITIONS THEREFOR

#### TECHNICAL FIELD

The present invention relates to improvements in the laundry process, including the provision of methods to improve the odor of fabrics that retain odor, especially malodor, after a conventional laundry process. The invention also includes concentrated, non-liquid compositions for use in the laundry process, especially concentrated additive compositions that can be used selectively on such fabrics and articles comprising said compositions in association with instructions for practicing the method and/or obtaining the benefits that can be derived from the method. Preferably the compositions restore and/or maintain freshness by reducing malodor.

# **BACKGROUND OF THE INVENTION**

Typical laundry processes remove odors from normal fabrics containing relatively low levels of malodors. However, as the water temperature for laundry wash cycles has gotten lower, or when the laundry load has fabrics with high levels of odorants, or when there is some other factor like overloading involved, there is sometimes a lingering malodor. This lingering malodor is different from malodor that is present in some detergent compositions, or is generated after the wash, e.g., by microbial action, or which thereafter becomes attached to the fabrics and is sometimes accompanied by the presence of large amounts of hydrophobic soils. This problem has not been generally recognized, since the general expectation is that the wash cycle removes all odors. However, some consumers have noticed the problem and have taken extreme measures such as doing such fabrics only in separate loads. In general, consumers do not take steps to remove, or counteract the odor, such as, e.g., washing the article again, since the additional measures are not successful. Also, such a second washing is wasteful of time, water, and detergent, and causes increased wear on clothing. Using more detergent is usually undesirable, since that may cause the article to have detergent remaining after the rinse step.

Cyclodextrin has been used to control odors from detergent compositions, to protect perfumes in detergent compositions, improve the solubility of compounds like nonionic surfactants to improve their removal, and dyes to prevent their transfer to other fabrics by keeping them suspended.

The present invention relates to solving problems associated with having odor, especially a malodor, remaining after the wash process is completed, preferably by the addition of cyclodextrin to help remove/control the malodor, or, less optimally, provide malodor

counteractants, like odor blockers or materials that react with the malodors or mask the malodors. The preferred approach uses those materials that result in the removal, or tying up of the malodor. The preferred methods and compositions are used as additives, since the majority of fabric laundry loads do not have the problem and since many of the materials that can neutralize the malodor have their own problems. Cyclodextrin tends to react with perfumes, and surfactants when incorporated in detergent compositions and the level required for malodor control is very high. Odor blockers, when used at the high levels needed for malodor control, block the desirable odors of perfumes as well as the malodors. Similarly, the masking compounds block other desirable odors and reactants can destroy desirable odors.

There is anecdotal information that indicates some consumers may have noticed the problem and have found some ways of solving the problem using materials that are part of the invention herein. However, to avoid causing problems, it is important to provide the general consumer with the identity of the laundry processes, soils, loads, conditions, etc. that typically provide insufficient removal of malodors and the level of ingredients needed to see the benefit. This allows the use of the additive when it is needed. Prior to this invention, the efforts to counteract malodor were based on insufficient information to ensure good results without wasteful use of excess material.

As stated before, in general, provision of such counteractants in the detergent, or fabric softener, is not efficient, since for some loads the benefit is not needed. Also, the level of many ingredients needed to provide good malodor removal/elimination is usually quite high, even for those counteractants that are really effective. Selection of the best counteractant can provide superior results. It is important to avoid the inclusion in the additive compositions of high levels of materials that interfere with the portion of the laundry process where the additive is used. For example, large amounts of acid materials usually hurts detergency by lowering the pH of the wash liquor; anionic materials are usually not compatible with cationic fabric softeners; etc.

#### SUMMARY OF THE INVENTION

The present invention relates to the method of applying an effective amount of a odor control agent ("counteractant"), preferably via a concentrated, non-liquid composition, to at least one step of a laundry process to provide a consumer noticeable improvement in the laundry process by either eliminating odor, especially malodor, or improving the removal of hydrophobic soils, in an efficient way. Generally, because of the high level of ingredients required for this benefit, it is essential to supply the consumer with the requisite information required to make good decisions, e.g., as to when to use the method by defining the areas of greatest benefit, the

amount of malodor counteractant required to provide such a benefit, etc. and providing concentrated, non-liquid compositions and delivery methods that minimize the use of too much or too little counteractant. The compositions are preferably supplied in a package in association with this information. The best counteractants provide some residual malodor prevention effects as well as providing superior end results for the laundry process.

# DETAILED DESCRIPTION OF THE INVENTION

### I. METHODS OF USE FOR ODOR CONTROL

The concentrated, non-liquid compositions described hereinafter can be used by adding an effective amount of the compositions to fabrics in one, or more of the steps in a typical laundry cycle including a presoak, a wash step, a rinse step, or a water removal step, e.g., wringing or spinning, drying, etc. An effective amount as defined herein means an amount sufficient to absorb or counteract malodor to the point that it is less objectionable, preferably not discernible by the human sense of smell. As discussed herein, for certain odors, the level in the atmosphere around the fabrics, "head space", should be less than the minimum detectable concentration for that odor. The term "non-liquid" is meant to describe compositions that have a viscosity greater than about 5,000 centipoise ("cPs"), measured at 25°C via a Brookfield viscometer at 60 RPM. As such, the term "non-liquid" is meant to include product forms such as powders/granules, gels, pastes, tablets, substrates/sheets (water-soluble and water-insoluble), and the like.

The kinds of soils that are most likely to cause a severe malodor include: soils like those found on mechanics' clothes; food handlers, especially butchers' and kitchen workers' clothes; sewer workers' clothes; bar tenders' clothes; fire fighters' clothes; farm clothes; athletic clothing; factory workers' clothes; heavy machinery operators' clothes; etc. Such soils have an associated malodor that is almost impossible to counteract without the present invention. Such soils also have a relatively high level of hydrophobic soils such as lubricating oil, grease, food oils, body soils, smoke, and the like. The preferred cyclodextrin malodor counteractant improves the removal of such soils.

For control of malodors, beta-cyclodextrin and alpha-cyclodextrin are preferred. Gamma-cyclodextrin has too large a cavity to control most malodor molecules. Substituted cyclodextrins can be especially valuable where they are more soluble than the corresponding unsubstituted cyclodextrin. The preferred compositions are concentrated, non-liquid compositions, in forms such as powders/granules, gels, pastes, tablets, and substrates/sheets (including water-soluble and water-insoluble substrates/sheets), to minimize packaging while maximizing the speed of action. Cyclodextrins can complex with surfactants and perfumes in the wash or rinse waters, thus it is important to disperse the cyclodextrin as soon as possible. It is

surprising that the cyclodextrin is not inactivated by, e.g., the surfactant. Using an additive containing cyclodextrin rather than adding cyclodextrin to the detergent or softening composition minimizes the interaction of the cyclodextrin with the ingredients of the detergent and/or softening compositions.

The level of cyclodextrin required for odor removal is high, but it tends to be less than that required for solubilizing surfactant. Furthermore, it is important that in any detergent composition or softening composition, the cyclodextrin, if present, should be separated (protected) from the actives that could form complexes with the cyclodextrin if one wants to obtain malodor removal from the laundry fabrics. Cyclodextrin that is added to remove odors from the detergent ingredients or to solubilize surfactants is not available for malodor control. Thus the additive concentrated, non-liquid compositions used herein to practice the method are preferably substantially free (i.e., there is not enough of the material so that uncomplexed cyclodextrin is still available) of materials that will complex with the cyclodextrin, such as enzymes, nonionic surfactants that will complex with the cyclodextrin, cationic fabric softener actives containing straight alkyl chains, fatty acids and their soaps and derivatives thereof, perfumes that complex with the cyclodextrin, and the like (i.e. materials, especially surfactants, that are not cyclodextrin-compatible).

The level of cyclodextrin, preferably uncomplexed, is related to the soil and/or odor level. The minimum levels are in progressively preferred approximate amounts, especially as the level of soil/odor increase, about 20 ppm, 30 ppm, 40 ppm, and 60 ppm respectively and the maximum levels in increasing order of preference are about 500 ppm, 300 ppm, 200 ppm, and 110 ppm respectively. These levels relate to the amount of cyclodextrin in the treatment solution. for example, the wash or rinse solution in a typical laundry washing machine (e.g. a 20 gallon washing machine).

The following table illustrates typical methods of use of a concentrated, non-liquid product of the current invention, as disclosed hereinafter, during a wash or rinse cycle.

Treatment	Use during wash cycle or rinse cycle
Conditions	
Machine Type	Kenmore 20 gallon washing machine represents a typical type of top
	loading washer
Laundry Load	Normal Load : about 7 lb

Temperature setting	Normal, hot, or cold
Typical Directions	Use about 10 grams for normal uses, or for extra odor removal use about 15 grams. Add directly to the washing machine during wash cycle or final rinse cycle. This is based upon cyclodextrin levels of from about 20 ppm to about 200 ppm, and preferably from about 30 ppm to about 110 ppm, by weight of the wash or rinse liquor for normal usage and from about 40 ppm to about 500 ppm, preferably from about 60 ppm to about 300 ppm, by weight of the wash or rinse liquor for extra odor removal.

The following examples illustrate the surprising added malodor removal benefit of a typical concentrated composition of the current invention to Tide® powder laundry detergent on fabrics during a wash or rinse cycle.

Method of Use	Dry Grease Odor Grades After Treatment	Dry Synthetic Body Odor Grade After treatment
	Initial = 90 -100*	Initial = 90 -100*
½ Cup Tide® detergent only in wash	40	35
½ cup Tide <sup>®</sup> detergent and 10 grams of an odor removal, non-liquid concentrate¹ added during wash cycle	5	15

1/2 cup Tide® detergent in	0	15
wash and 10 grams of an		
odor removal, non-liquid		
concentrate added during		
final rinse cycle		

<sup>&</sup>lt;sup>1</sup> The non-liquid concentrate contains: 89% hydroxypropyl beta-cyclodextrin, 9% HCO-60, and 2% perfume.

\*The dry fabric odor grades are based upon the evaluation by an expert perfume panel, using a grading scale where 0 = no odor and 100 = extremely strong odor. The final grade is a measure of the overall effectiveness on odor removal with the lower number being better. A 15 units difference in final grade normally represents a consumer noticeable difference in product performance. Furthermore, a final odor grade of less than 20 is generally not detectable by consumer.

If the present concentrated, non-liquid composition is a gel or a paste, or similar product form, it can be used as a fabric pretreater to further enhance its odor control performance. As a fabric pretreater, the recommendation is to apply product directly to the soiled fabric evenly. For best results, the instructions are to apply the composition to the soiled fabric evenly until slightly damp and then add the garment to the wash.

In the preferred compositions herein, the presence of a cyclodextrin-compatible surfactant promotes spreading of the solution and the antimicrobial active provides improved odor control as well as antimicrobial action, by minimizing the formation of odors. Both the surfactant and the antimicrobial active provide improved performance.

For compositions containing odor blockers as a odor control agent, the level of odor blocker is sufficient to reduce the odor, and is preferably from about 0.004 ppm to about 10 ppm, more preferably from about 0.007 ppm to about 5 ppm by weight of the treatment solution, either wash water or rinse water, e.g., the wash or rinse solution in a 20 gallon machine, for normal odor levels; and preferably from 0.007 ppm to about 30 ppm, more preferably from about 0.01 ppm to about 7 ppm, by weight of the treatment solution, for higher odor levels. For materials that react with the odor, like aldehydes, sulfites, and the like as odor control agents, the level is preferably from about 0.05 ppm to about 10 ppm, more preferably from about 0.1 ppm to about 7 ppm. by weight of the treatment solution for normal odor levels; and is preferably from about 0.1 ppm to about 30 ppm, more preferably from about 0.5 ppm to about 15 ppm, by weight of the treatment solution, for higher odor levels. For materials like flavanoids, as odor control agents that mask

the malodor, the level is preferably from about 0.1 ppm to about 40 ppm, more preferably from about 0.5 ppm to about 10 ppm, by weight of the treatment solution, for normal odor levels; and is preferably from about 0.2 ppm to about 140 ppm, more preferably from about 1 ppm to about 20 ppm, by weight of the treatment solution, for higher odor levels.

While the preferred methods of the present invention relate to methods utilizing automatic laundry washing and drying machines, the present methods also encompass methods utilizing non-automatic (e.g. hand-washing) laundry cleaning steps. An even more pronounced reduction in malodor impression on fabrics can be observed when the methods of the present invention include a drying step that does not use an automatic laundry dryer (i.e. a gas or electric laundry dryer). For example, the drying step is preferably an air-drying or line-drying (e.g. drying of the clothes (after rinsing) on a line, cord, or wire, exposed to the sun, or air outside one's house) step. Line-drying will tend to evaporate less water and hence, give cyclodextrin and the other counteractants more time to complex with the malodorous molecules or compounds. Line-drying will also tend to result in less volatilization of some perfume components resulting in improved freshness impression on fabrics.

The important new information discovered by Applicants is that there is a relatively wide-spread significant problem associated with high soil loads for some soils as discussed hereinbefore. The problem includes inefficient removal and/or, especially, malodor associated with these soils. Therefore, it is important that any product containing these odor counteractants have sufficient odor counteractant(s) to provide sufficient reduction in soil and/or odor and that the product, preferably in a package, be in association with instructions to use the product at sufficient levels to provide the benefit(s) and that the soils be identified for the consumer. Moreover, this high soil load is more prevalent in hand-washing geographies where water is in low supply. Hence, the invention is particularly more useful for these geographies.

In addition to the reduction in soil and/or malodor achieved using the present methods that utilize the compositions described herein, the present methods also encompass methods of preventing malodor from developing on fabrics. Malodor prevention is different from malodor reduction or removal, in that malodor prevention is a proactive method to minimize the possibility for malodor to develop on fabrics, especially after being laundered. Malodor typically develops on clothing fabrics either during "in wear" conditions of the clothing fabrics or during storage of clothing fabrics, such as in closets or environments susceptible to mold or mildew. The development of malodor on clothing fabrics during "in wear" conditions can prove quite embarrassing to the individual wearing the clothing fabrics. The present methods can help prevent these malodors from develop on the clothing fabrics, especially during "in wear" conditions.

The present methods of preventing malodor from developing on fabrics comprises the step of adding an effective amount of the concentrated, non-liquid compositions described herein to a wash or rinse cycle of a typical laundry process in order to prevent malodor from developing on the fabrics. To obtain malodor prevention, an effective amount of the odor control agents described herein needs to be deposited on the fabrics such that a sufficient amount of the odor control agent remains on the fabrics after the washing process to prevent malodor from developing on the fabrics.

A preferred odor control agent for preventing malodor from developing on fabrics is cyclodextrin. The present methods of preventing malodor from developing on fabrics preferably further comprises depositing an effective amount of cyclodextrin on the fabrics to prevent malodor. Typically, the amount of cyclodextrin to remain on the fabrics to effectively prevent malodor from developing on the fabrics will be at least about 0.001%, preferably at least about 0.01%, and more preferably at least about 0.1%, by weight of the fabric. Furthermore, it is important to provide instructions to a consumer of the compositions of the present invention in order to communicate the malodor prevention benefits of the compositions and instruct the consumer to use the requisite amounts of the compositions to achieve the benefits.

A preferred composition for use in the malodor prevention methods of the present invention comprises cyclodextrin, uncomplexed perfume, and perfume carrier complex, preferably cyclodextrin/perfume inclusion complex. Another preferred embodiment further comprises a cyclodextrin-compatible surfactant, and a cyclodextrin-compatible antimicrobial active. In using this composition, the amount of antimicrobial active remaining on the fabric to provide malodor prevention is typically at least about 0.001%, preferably at least about 0.11%, by weight of the fabric.

#### II. CONCENTRATED, NON-LIQUID COMPOSITIONS FOR ODOR CONTROL

The present invention further encompasses concentrated, non-liquid compositions, preferably to be used in the methods for controlling odor, especially malodor, described hereinbefore in Section I, *supra*. The concentrated, non-liquid compositions herein generally comprise:

- (a) an effective amount to reduce malodor impression on fabrics in a laundry process of an odor control agent selected from the group consisting of:
  - (i) an effective amount to absorb malodors, typically at least about 1%, preferably at least about 5%, more preferably at least about 10%, even more preferably at least about 20%, and still more preferably at least about 40%, by weight of the composition, of uncomplexed cyclodextrin;

(ii) an effective amount of odor blocker typically from about 0.005% to about 10% by weight of the composition, preferably from about 0.01% to about 5%, more preferably from about 0.05% to about 2% by weight of the composition;

- (iii) an effective amount of class I and/or class II aldehydes typically from about 0.1% to about 10% by weight of composition, preferably from about 0.5% to about 5%;
- (iv) an effective amount of flavanoid, typically from about 0.1% to about 50%, and preferably from about 0.5% to about 10%, by weight of the composition;
- (v) an effective amount of metallic salt, preferably from about 1% to about 90%, more preferably from about 0.2% to about 80%, even more preferably from about 0.3% to about 50% by weight of the usage composition, especially water soluble copper and/or zinc salts, for improved odor benefit; and
- (vi) mixtures thereof;
- (b) optionally, an effective amount to provide a freshness impression on fabrics in a laundry process of uncomplexed perfume, typically from about 0.03% to about 2%, preferably from about 0.1% to about 1%, more preferably from about 0.2% to about 0.5%, by weight of the concentrated, non-liquid composition; said perfume being in addition to said odor blocker, class I aldehydes, class II aldehydes, and/or flavanoids; said perfume preferably comprising at least about 50%, more preferably at least about 60%, and even more preferably at least about 70%, and yet still more preferably at least about 80%, by weight of the perfume of perfume ingredients that have a ClogP of greater than about 3, preferably greater than about 3.5 and/or a molecular weight of greater than 210, preferably greater than about 220; said perfume, when present, being in addition to said odor control agents such as odor blocker, class I aldehydes, class II aldehydes, flavanoids, and/or metallic salts;
- (c) optionally, a perfume carrier complex, preferably selected from the group consisting of cyclodextrin/perfume inclusion complexes, matrix perfume microcapsules, and mixtures thereof;
- (d) optionally, cyclodextrin-compatible surfactant, preferably selected from the group consisting of: block copolymer surfactant, siloxane surfactant, anionic surfactant, castor oil surfactant, sorbitan ester surfactant, polyethoxylated fatty alcohol surfactant, glycerol mono-fatty acid ester surfactant, polyethylene glycol fatty acid

ester surfactant, fluorocarbon surfactant, and mixtures thereof; and more preferably selected from the group consisting of castor oil surfactant, sorbitan ester surfactant, polyethoxylated fatty alcohol surfactant, glycerol mono-fatty acid ester surfactant, polyethylene glycol fatty acid ester surfactant, fluorocarbon surfactant, and mixtures thereof;

- (e) optionally, an effective amount to kill or reduce microorganisms of an antimicrobial active, preferably selected from the group consisting of: biguanides, quaternary compounds, peroxygen bleach / bleach activator system, and mixtures thereof;
- (f) optionally, filler materials; preferably selected from the group consisting of alkaline earth metals such as sodium and/or magnesium, calcium and even aluminum salts of sulfates, carbonates, bicarbonates, chlorides, silicates (clay), and mixtures thereof.

Preferably, the concentrated, non-liquid compositions herein are capable of dissolving in a laundry wash or rinse solution within about 5 minutes, preferably within about 3 minutes, starting from the point at which the composition is added to the wash or rinse solution. As used herein, the term "dissolved" means that no visible residue from the composition is apparent in the wash or rinse solution.

Since the present concentrated, non-liquid compositions are preferably additive compositions to be used in conjunction with a conventional laundry detergent composition in the laundry process, the compositions herein are preferably essentially free of, or free of, conventional laundry detergent surfactants, including anionic surfactants such as alkyl benzene sulfonates, alkyl sulfates, alkyl ether sulfates, and the like.

Upon dilution in a wash or rinse solution in a laundry process, the present compositions provide a pH in the wash or rinse solution of from about 7 to about 12, preferably from about 7 to about 11, and more preferably from about 7 to about 10.

#### A. ODOR CONTROL AGENT

The present concentrated, non-liquid compositions for controlling odor comprise an effective amount of odor control agent to significantly reduce odor, especially malodor, that exists on fabric in a laundry process, particularly malodor that survives a typical laundry process. The amount required to significantly reduce odor in fabrics typically varies according to the particular odor control agent as described hereinafter. The odor control agent is preferably selected from the group consisting of: cyclodextrin, preferably uncomplexed cyclodextrin; odor blocker; class I aldehydes; class II aldehydes; flavanoids; and mixtures thereof.

#### 1. CYCLODEXTRIN

As used herein, the term "cyclodextrin" includes any of the known cyclodextrins such as unsubstituted cyclodextrins containing from six to twelve glucose units, especially, alphacyclodextrin, beta-cyclodextrin, gamma-cyclodextrin and/or their derivatives and/or mixtures thereof. The alpha-cyclodextrin consists of six glucose units, the beta-cyclodextrin consists of seven glucose units, and the gamma-cyclodextrin consists of eight glucose units arranged in donut-shaped rings. The specific coupling and conformation of the glucose units give the cyclodextrins a rigid, conical molecular structures with hollow interiors of specific volumes. The "lining" of each internal cavity is formed by hydrogen atoms and glycosidic bridging oxygen atoms; therefore, this surface is fairly hydrophobic. The unique shape and physical-chemical properties of the cavity enable the cyclodextrin molecules to absorb (form inclusion complexes with) organic molecules or parts of organic molecules which can fit into the cavity. Many odorous molecules can fit into the cavity including many malodorous molecules and perfume molecules. Therefore, cyclodextrins, and especially mixtures of cyclodextrins with different size cavities, can be used to control odors caused by a broad spectrum of organic odoriferous materials, which may, or may not, contain reactive functional groups. The complexation between cyclodextrin and malodorous molecules occurs rapidly in the presence of water. However, the extent of the complex formation also depends on the polarity of the absorbed molecules. In an aqueous solution, strongly hydrophilic molecules (those which are highly water-soluble) are only partially absorbed, if at all. Therefore, cyclodextrin does not complex effectively with some very low molecular weight organic amines and acids when they are present at low levels on fabrics. As the water is being removed however, e.g., water is being removed from fabrics during a spin cycle, electric drying cycle, or line-drying step, some low molecular weight organic amines and acids have more affinity and will complex with the cyclodextrins more readily.

The cavities within the cyclodextrin in the concentrated, non-liquid composition of the present invention should remain essentially unfilled (the cyclodextrin remains uncomplexed), in order to allow the cyclodextrin to absorb various odor molecules when the solution is applied to a surface. Non-derivatised (normal) beta-cyclodextrin can be present at a level up to its solubility limit of about 1.85% (about 1.85g in 100 grams of water) under the conditions of use at room temperature.

Preferably, the cyclodextrin used in the present invention is highly water-soluble such as, alpha-cyclodextrin and/or derivatives thereof, gamma-cyclodextrin and/or derivatives thereof, derivatised beta-cyclodextrins, and/or mixtures thereof. The derivatives of cyclodextrin consist mainly of molecules wherein some of the OH groups are converted to OR groups. Cyclodextrin

derivatives include, e.g., those with short chain alkyl groups such as methylated cyclodextrins, and ethylated cyclodextrins, wherein R is a methyl or an ethyl group; those with hydroxyalkyl substituted groups, such as hydroxypropyl cyclodextrins and/or hydroxyethyl cyclodextrins, wherein R is a -CH<sub>2</sub>-CH(OH)-CH<sub>3</sub> or a -CH<sub>2</sub>CH<sub>2</sub>-OH group; branched cyclodextrins such as maltose-bonded cyclodextrins; cationic cyclodextrins such as those containing 2-hydroxy-3-(dimethylamino)propyl ether, wherein R is CH2-CH(OH)-CH2-N(CH3)2 which is cationic at low pH; quaternary ammonium, e.g., 2-hydroxy-3-(trimethylammonio)propyl ether chloride groups, wherein R is CH2-CH(OH)-CH2-N+(CH3)3Cl-; anionic cyclodextrins such as carboxymethyl cyclodextrins, cyclodextrin sulfates, and cyclodextrin succinylates; amphoteric cyclodextrins such as carboxymethyl/quaternary ammonium cyclodextrins; cyclodextrins wherein at least one glucopyranose unit has a 3-6-anhydro-cyclomalto structure, e.g., the mono-3-6anhydrocyclodextrins, as disclosed in "Optimal Performances with Minimal Chemical Modification of Cyclodextrins", F. Diedaini-Pilard and B. Perly, The 7th International Cyclodextrin Symposium Abstracts, April 1994, p. 49, said references being incorporated herein by reference; and mixtures thereof. Other cyclodextrin derivatives are disclosed in U.S. Pat. Nos: 3.426.011, Parmerter et al., issued Feb. 4, 1969; 3,453,257; 3,453,258; 3,453,259; and 3,453,260, all in the names of Parmerter et al., and all issued July 1, 1969; 3,459,731, Gramera et al., issued Aug. 5, 1969; 3,553,191, Parmerter et al., issued Jan. 5, 1971; 3,565,887, Parmerter et al., issued Feb. 23, 1971; 4,535,152, Szejtli et al., issued Aug. 13, 1985; 4,616,008, Hirai et al., issued Oct. 7, 1986; 4,678,598, Ogino et al., issued Jul. 7, 1987; 4,638,058, Brandt et al., issued Jan. 20, 1987; and 4,746,734, Tsuchiyama et al., issued May 24, 1988; all of said patents being incorporated herein by reference. Further cyclodextrin derivatives suitable herein include those disclosed in V. T. D'Souza and K. B. Lipkowitz, CHEMICAL REVIEWS: CYLCODEXTRINS, Vol. 98, No. 5 (American Chemical Society, July/August 1998), which is incorporated herein by reference.

Highly water-soluble cyclodextrins are those having water solubility of at least about 10 g in 100 ml of water at room temperature, preferably at least about 20 g in 100 ml of water, more preferably at least about 25 g in 100 ml of water at room temperature. The availability of uncomplexed cyclodextrins is important for effective and efficient odor control performance. Water-soluble cyclodextrin can exhibit more efficient odor control performance than non-water-soluble cyclodextrin when deposited onto fabrics, especially during a laundry process.

Examples of preferred water-soluble cyclodextrin derivatives suitable for use herein are hydroxypropyl alpha-cyclodextrin, methylated alpha-cyclodextrin, methylated beta-cyclodextrin, hydroxyethyl beta-cyclodextrin, and hydroxypropyl beta-cyclodextrin. Hydroxyalkyl cyclodextrin derivatives preferably have a degree of substitution of from about 1 to about 14, more preferably from about 1.5 to about 7, wherein the total number of OR groups per cyclodextrin is defined as the degree of substitution. Methylated cyclodextrin derivatives typically have a degree of substitution of from about 1 to about 18, preferably from about 3 to about 16. A known methylated beta-cyclodextrin is heptakis-2,6-di-O-methyl-β-cyclodextrin, commonly known as DIMEB, in which each glucose unit has about 2 methyl groups with a degree of substitution of about 14. A preferred, more commercially available, methylated betacyclodextrin is a randomly methylated beta-cyclodextrin, commonly known as RAMEB, having different degrees of substitution, normally of about 12.6. RAMEB is more preferred than DIMEB, since DIMEB affects the surface activity of the preferred surfactants more than RAMEB. The preferred cyclodextrins are available, e.g., from Cerestar USA, Inc. and Wacker Chemicals (USA), Inc.

It is also preferable to use a mixture of cyclodextrins. Such mixtures absorb odors more broadly by complexing with a wider range of odoriferous molecules having a wider range of molecular sizes. Preferably at least a portion of the cyclodextrin is alpha-cyclodextrin and its derivatives thereof, gamma-cyclodextrin and its derivatives thereof, and/or derivatised beta-cyclodextrin, more preferably a mixture of alpha-cyclodextrin, or an alpha-cyclodextrin derivative, and derivatised beta-cyclodextrin, even more preferably a mixture of derivatised alpha-cyclodextrin and derivatised beta-cyclodextrin, most preferably a mixture of hydroxypropyl alpha-cyclodextrin and hydroxypropyl beta-cyclodextrin, and/or a mixture of methylated alpha-cyclodextrin and methylated beta-cyclodextrin.

While cyclodextrin is an effective odor absorbing active, some small molecules are not sufficiently absorbed by the cyclodextrin molecules because the cavity of the cyclodextrin molecule may be too large to adequately hold the smaller organic molecule. If a small sized organic odor molecule is not sufficiently absorbed into the cyclodextrin cavity, a substantial amount of malodor can remain. In order to alleviate this problem, low molecular weight polyols can be added to the composition as discussed hereinafter, to enhance the formation of cyclodextrin inclusion complexes. Furthermore, optional water soluble metal salts can be added as discussed hereinafter, to complex with some nitrogen-containing and sulfur-containing malodor molecules.

Since cyclodextrin can be a prime breeding ground for certain microorganisms, albeit to a lesser degree in non-liquid compositions, it can be preferable to include an antimicrobial preservative, which is effective for inhibiting and/or regulating microbial growth, when the composition does not contain an antimicrobial active as described hereinafter. Suitable antimicrobial preservatives are described in detail in U.S. Patent 5,942,217 issued August 24, 1999 to Woo et al., at col. 24, line 22 to col. 29, line 55, which is incorporated herein by reference. Such antimicrobial preservatives can be included in the present concentrated, non-liquid compositions if an antimicrobial active, as discussed hereinafter in Section II.E, is either not present or not sufficient to effectively preserve the composition.

It can also be desirable to provide optional ingredients such as an antimicrobial active, as described in Section II.E, that provides substantial kill of organisms that cause, e.g., odor, infections, etc. It is also further desirable that the compositions contain an optional cyclodextrin-compatible surfactant to promote spreading of the odor controlling composition on hydrophobic surfaces such as polyester, nylon, etc. as well as to penetrate any oily, hydrophobic soil for improved odor control, especially control of malodor. It is more preferable that the concentrated, non-liquid compositions of the present invention contain both an antibacterial active and a cyclodextrin-compatible surfactant.

For controlling odor on fabrics in a laundry process, the concentrated, non-liquid compositions herein are preferably used in concentrated form and added to the wash or rinse cycle of a laundry process, either alone or in combination with a conventional laundry detergent composition, to maximize the malodor control and to take advantage of the cleaning benefit that can be achieved by the use of high levels of cyclodextrin. Specifically, soils that contain high levels of hydrophobic, oily soils, can be removed more completely by the addition of cyclodextrin. This more complete removal is partly due to solubilization of the soils from the fabrics and partly due to the suspension of the soils. Surprisingly, the interaction of the cyclodextrin and surfactants is minimal when the cyclodextrin is added during the wash or rinse cycle of the laundry process in combination with a conventional laundry detergent composition due to the lack of time and/or concentration required to form complexes.

#### 2. ODOR BLOCKERS

Although not as preferred, "odor blockers" can be used as an odor control agent to mitigate the effects of malodors. In order to be effective, the odor blockers normally have to be present at all times. If the odor blocker evaporates before the source of the odor is gone, it is less

likely to control the odor. Also, the odor blockers tend to adversely affect aesthetics by blocking desirable odors like perfumes.

Suitable odor blockers are disclosed in U.S. Pat. Nos. 4,009,253; 4,187,251, 4,719,105; 5,441,727; and 5,861,371, said patents being incorporated herein by reference.

#### 3. ALDEHYDES

As an optional odor control agent, aldehydes can be used to mitigate the effects of malodors. Suitable aldehydes are class I aldehydes, class II aldehydes, and mixtures thereof, that are disclosed in U.S. Patent No. 5,676,163, said patent being incorporated herein by reference.

#### 4. FLAVANOIDS

Another optional odor control agent are flavanoids, which are ingredients found in typical essential oils. Such oils include essential oil extracted by dry distillation from needle leaf trees and grasses such as cedar, Japanese cypress, eucalyptus, Japanese red pine, dandelion, low striped bamboo and cranesbill and it contains terpenic material such as alpha-pinene, beta-pinene, myrcene, phencone and camphene. The terpene type substance is homogeneously dispersed in the finishing agent by the action of nonionic surfactant and is attached to fibres constituting the cloth. Also included are extracts from tea leaf. Descriptions of such materials can be found in JP6219157, JP 02284997, JP04030855, etc. said references being incorporated herein by reference.

#### 5. METALLIC SALTS

The odor control agent of the present invention can include metallic salts for added odor absorption and/or antimicrobial benefit, especially where cyclodextrin is also present as an odor control agent in the composition. The metallic salts are selected from the group consisting of copper salts, zinc salts, and mixtures thereof.

The preferred zinc salts possess malodor control abilities. Zinc has been used most often for its ability to ameliorate malodor, e.g., in mouth wash products, as disclosed in U.S. Pat. Nos. 4,325,939, issued Apr. 20, 1982 and 4,469,674, issued Sept. 4, 1983, to N. B. Shah, et al., all of which are incorporated herein by reference. Highly-ionized and soluble zinc salts such as zinc chloride, provide the best source of zinc ions. Zinc borate can function as a fungistat and a mildew inhibitor, zinc caprylate functions as a fungicide, zinc chloride provides antiseptic and deodorant benefits, zinc ricinoleate functions as a fungicide, zinc sulfate heptahydrate functions as a fungicide and zinc undecylenate functions as a fungistat.

Preferably the metallic salts are water-soluble zinc salts, copper salts or mixtures thereof, and more preferably zinc salts, especially ZnCl<sub>2</sub>. These salts are preferably present in the present

invention as an odor control agent primarily to absorb amine and sulfur-containing compounds. These compounds have molecular sizes too small to be effectively complexed with a cyclodextrin odor control agent. Low molecular weight sulfur-containing materials, e.g., sulfide and mercaptans, are components of many types of malodors, e.g., food odors (garlic, onion), body/perspiration odor, breath odor, etc. Low molecular weight amines are also components of many malodors, e.g., food odors, body odors, urine, etc.

Copper salts possess some odor control abilities. See U. S. Pat. No. 3,172,817, Leupold, et al., which discloses deodorizing compositions for treating disposable articles, comprising at least slightly water-soluble salts of acylacetone, including copper salts and zinc salts; said patent being incorporated herein by reference. Copper salts also have some antimicrobial benefits. Specifically, cupric abietate acts as a fungicide, copper acetate acts as a mildew inhibitor, cupric chloride acts as a fungicide, copper lactate acts as a fungicide, and copper sulfate acts as a germicide.

When metallic salts are added to the composition of the present invention as an odor control agent, they are typically present at a level of from about 0.1% to an effective amount to provide a saturated salt solution, preferably from about 0.2% to about 90%, more preferably from about 2% to about 80%, still more preferably from about 4% to about 50% by weight of the usage composition.

#### B. <u>UNCOMPLEXED PERFUME</u>

The concentrated, non-liquid compositions of the present invention can also comprise uncomplexed perfume to provide a "scent signal," or freshness impression, in the form of a pleasant odor which signals the removal of malodor from fabrics. Also, uncomplexed perfume can enhance the aesthetic experience of consumers and provide a "scent signal" to indicate to the consumer that the malodor has been "cleaned" from the surface. To the extent any perfume ingredients can be considered odor control agents as described herein, such materials shall be considered odor control agents and not perfume ingredients in accordance with the present invention. As such, the uncomplexed perfume herein is in addition to perfume ingredients that fulfill the role of odor control agent (i.e. malodor counteractant) and perfume ingredients contained in perfume carrier complexes (as described in Section II.C, *infra*), and are designed to provide, at least in part, a lasting perfume scent. Perfume is added at levels of from about 0.0001% to about 5%, preferably from about 0.003% to about 3%, more preferably from about 0.005% to about 1%, by weight of the concentrated, non-liquid composition.

Uncomplexed perfume is added to provide a more lasting odor on fabrics. When stronger levels of perfume are preferred, relatively higher levels of perfume can be added. Any type of uncomplexed perfume can be incorporated into the composition of the present invention so long as the preferred hydrophobic perfume that will complex with the cyclodextrin is formed into a miscelle or vessicle with a droplet size that will not readily interact with the cyclodextrin in the concentrated, non-liquid composition. The perfume ingredients can be either hydrophilic or hydrophobic.

Hydrophilic perfumes are composed predominantly of ingredients having a ClogP of less than about 3.5, more preferably less than about 3 and, preferably, lower molecular weights, e.g., below about 220, preferably below about 210. If longer lasting perfume effects are desired, the hydrophobic perfumes disclosed below are used.

#### a. Hydrophobic Perfume Ingredients

In order to provide long lasting effects, the uncomplexed perfume is at least partially hydrophobic and has a relatively high boiling point. I.e., it is composed predominantly of ingredients selected from two groups of ingredients, namely, (a) hydrophilic ingredients having a ClogP of more than about 3, more preferably more than about 3.5, and (b) ingredients having a molecular weight above about 210, preferably above about 220. Typically, at least about 50%, preferably at least about 60%, more preferably at least about 70%, and most preferably at least about 80% by weight of the perfume is composed of perfume ingredients of the above groups (a) and (b). For these preferred perfumes, the cyclodextrin to perfume weight ratio is typically of from about 2:1 to about 200:1; preferably from about 4:1 to about 100:1, more preferably from about 6:1 to about 50:1, and even more preferably from about 8:1 to about 30:1.

Hydrophobic perfume ingredients have a tendency to complex with the cyclodextrins. The degree of hydrophobicity of a perfume ingredient can be correlated with its octanol/water partition coefficient P. The octanol/water partition coefficient of a perfume ingredient is the ratio between its equilibrium concentration in octanol and in water. A perfume ingredient with a greater partition coefficient P is considered to be more hydrophobic. Conversely, a perfume ingredient with a smaller partition coefficient P is considered to be more hydrophobic. Since the partition coefficients of the perfume ingredients normally have high values, they are more conveniently given in the form of their logarithm to the base 10, logP. Thus the preferred perfume hydrophobic perfume ingredients of this invention have a logP of about 3 or higher, preferably of about 3.5 or higher. Such hydrophobic perfume ingredients are described in detail

in co-pending U.S. Application Serial No. 09/571,131, filed May 15, 2000 by R. A. Woo et al., at page 18, line 11 to page 21, line32, which is incorporated herein by reference.

#### b. <u>Low Odor Detection Threshold Perfume Ingredients</u>

The composition can also contain low to moderate levels of low odor detection threshold materials. The odor detection threshold is the lowest vapor concentration of that material which can be olfactorily detected. The odor detection threshold and some odor detection threshold values are discussed in, e.g., "Standardized Human Olfactory Thresholds", M. Devos et al, IRL Press at Oxford University Press, 1990, and "Compilation of Odor and Taste Threshold Values Data", F. A. Fazzalari, editor, ASTM Data Series DS 48A, American Society for Testing and Materials, 1978, both of said publications being incorporated by reference. Examples of Low Odor Detection Threshold Perfume Ingredients are described in co-pending U.S. Application Serial No. 09/571,131 filed May 15, 2000 by R. A. Woo et al., at page 21, line 42 to page 23, line 13, which is incorporated herein by reference.

#### C. PERFUME CARRIER COMPLEX

The concentrated, non-liquid compositions of this invention contain an effective amount of various moisture-activated encapsulated perfume particles, as an optional, but highly preferred component. The encapsulated perfume particles herein are preferably water-soluble. The encapsulated particles act as protective carriers and reduce the loss of perfume prior to use. Such materials include, for example, cyclodextrin/perfume inclusion complexes, polysaccharide cellular matrix perfume microcapsules, silica perfume carriers, and the like. Encapsulation of perfume minimizes the diffusion and loss of the volatile blooming perfume ingredients. Perfume is released when the materials are wetted, to provide a pleasant odor signal in use. Especially preferred are cyclodextrin/perfume inclusion complexes.

The optional water-activated protective perfume carriers are very useful in the present invention. They allow the use of lower total level of perfume in the concentrated, non-liquid compositions herein because of the reduced loss of the perfume during manufacturing, storage, and use.

Due to the minimal loss of the volatile ingredients of the blooming perfume compositions provided by the water activated protective perfume carrier, the perfume compositions that incorporate perfume carrier complex can generally contain less blooming perfume ingredients than those used in the free, uncomplexed form, as described hereinbefore in Section II.B, *supra*. The encapsulated and/or complexed perfume compositions typically contain at least about 20%, preferably at least about 30%, and more preferably at least about 40% of hydrophilic perfume

ingredients, i.e. perfume ingredients having a ClogP of less than about 3.0. Optionally, but preferably, compositions that contain encapsulated and/or complexed perfume also comprise uncomplexed perfume in order to provide consumers with a positive scent signal before the composition is used.

#### 1. CYCLODEXTRIN / PERFUME INCLUSION COMPLEXES

As used herein, the term "cyclodextrin" includes any of the known cyclodextrins such as unsubstituted cyclodextrins containing from six to twelve glucose units, especially, alpha-, beta-, and gamma-cyclodextrins, and/or their derivatives, and/or mixtures thereof. Suitable cyclodextrins for forming perfume/cyclodextrin complexes have been described hereinbefore in Section II.A.1, *supra*.

The preferred cyclodextrin is a beta-cyclodextrin or derivatives thereof, such as hydroxyalkyl or alkylated beta-cyclodextrin. It is also desirable to use mixtures of cyclodextrins. Preferably at least a major portion of the cyclodextrins are alpha-, beta- and/or gamma-cyclodextrins, more preferably alpha- and beta-cyclodextrins. Some cyclodextrin mixtures are commercially available from, e.g., Ensuiko Sugar Refining Company, Yokohama, Japan.

The perfume/cyclodextrin inclusion complexes of this invention are formed in any of the ways known in the art. Typically, the complexes are formed either by bringing the perfume and the cyclodextrin together in a suitable solvent, e.g., water, or, preferably, by kneading/slurrying the ingredients together in the presence of a suitable, preferably minimal, amount of solvent, preferably water. The kneading/slurrying method is particularly desirable because it produces smaller complex particles and requires the use of less solvent, eliminating or reducing the need to further reduce particle size and separate excess solvent. Disclosures of complex formation can be found in Atwood, J.L., J.E.D. Davies & D.D. MacNichol, (Ed.): Inclusion Compounds. Vol. III, Academic Press (1984), especially Chapter 11, Atwood, J.L. and J.E.D. Davies (Ed.): Proceedings of the Second International Symposium of Cyclodextrins Tokyo, Japan, (July, 1984), and J. Szejtli, Cyclodextrin Technology, Kluwer Academic Publishers (1988), said publications incorporated herein by reference.

In general, perfume/cyclodextrin complexes have a molar ratio of perfume compound to cyclodextrin of about 1:1. However, the molar ratio can be either higher or lower, depending on the size of the perfume compound and the identity of the cyclodextrin compound. The molar ratio can be determined by forming a saturated solution of the cyclodextrin and adding the perfume to form the complex. In general the complex will precipitate readily. If not, the

complex can usually be precipitated by the addition of electrolyte, change of pH, cooling, etc. The complex can then be analyzed to determine the ratio of perfume to cyclodextrin.

As stated hereinbefore, the actual complexes are determined by the size of the cavity in the cyclodextrin and the size of the perfume molecule. Desirable complexes can be formed using mixtures of cyclodextrins since perfumes are normally mixtures of materials that vary widely in size. It is usually desirable that at least a majority of the material be alpha-, beta-, and/or gamma-cyclodextrin, more preferably beta-cyclodextrin. The content of the perfume in the beta-cyclodextrin complex is typically from about 5% to about 15%, more normally from about 7% to about 12%.

Continuous complexation operation usually involves the use of supersaturated solutions, kneading/slurrying method, and/or temperature manipulation, e.g., heating and then either cooling, freeze-drying, etc. The complexes are dried to a dry powder to make the desired composition. In general, the fewest possible process steps are preferred to avoid loss of perfume.

#### 2. MATRIX PERFUME MICROCAPSULES

Water-soluble cellular matrix perfume microcapsules are solid particles containing perfume stably held in the cells. The water-soluble matrix material comprises mainly polysaccharide and polyhydroxy compounds. The polysaccharides are preferably higher polysaccharides of the non-sweet, colloidally-soluble types, such as natural gums, e.g., gum arabic, starch derivatives, dextrinized and hydrolyzed starches, and the like. The polyhydroxy compounds are preferably alcohols, plant-type sugars, lactones, monoethers, and acetals. The cellular matrix microcapsules useful in the present invention are prepared by, e.g., (1) forming an aqueous phase of the polysaccharide and polyhydroxy compound in proper proportions, with added emulsifier if necessary or desirable; (2) emulsifying the perfumes in the aqueous phase; and (3) removing moisture while the mass is plastic or flowable, e.g., by spray drying droplets of the emulsion. The matrix materials and process details are disclosed in, e.g., U.S. Pat. No. 3,971,852, Brenner et al., issued July 27, 1976, which is incorporated herein by reference.

Moisture-activated perfume microcapsules can be obtained commercially, e.g., as IN-CAP 
® from Polak's Frutal Works, Inc., Middletown, New York; and as Optilok System® encapsulated perfumes from Encapsulated Technology, Inc., Nyack, New York.

Water-soluble matrix perfume microcapsules preferably have size of from about 0.5 micron to about 300 microns, more preferably from about 1 micron to about 200 microns, most preferably from about 2 microns to about 100 microns.

#### 3. SILICA PERFUME CARRIERS

Another type of perfume carrier complex suitable for use in the concentrated, non-liquid compositions of the present invention include amorphous silica, precipitated silica, fumed silica and aluminosilicates such as zeolite and alumina with a pore volume of at least 0.1 ml/g consisting of pores with a diameter between 4 and 100 A, which by their nature are hydrophilic. Perfume is incorporated in such carriers to form complexes by mixing the perfume and the carrier under shear conditions to provide a homogeneous mixture. Preferably, amorphous silica gel is used because of its high oil absorbency. Silica gel particles include SyloidR silicas such as Numbers: 72; 74; 221; 234; 235; 244; etc. SyloidR silicas are available from W. R. Grace & Co., Davison Chemical Division, P.O. Box 2117, Baltimore, Md. 21203. Such particles have surface areas of from about 250 to about 340 m<sup>2</sup>/g; pore volumes of from about 1.1 to about 1.7 cc/g; and average particle sizes of from about 2.5 to about 6 microns. Fumed silica particles have primary particle diameters of from about 0.007 to about 0.025 micron and include Cab-O-SilR Numbers: L-90; LM-130; LM-5; M-5; PTG; MS-55; HS-5; and EH-5. Cab-O-SilR silicas are available from Cabot Corp., P.O. Box 188, Tuscola, Ill. 61953. It is preferred that there be only minimal amounts of other materials present when the perfume is added to the silica particles to maximize adsorption. It is especially preferred that only small amounts, e.g., less than about 10% of organic materials, including waxes, be present.

Such silica perfume carriers are further described in U.S. Patent No. 5,723,420 issued March 3, 1998 to K. S. Wei et al., which is incorporated herein by reference.

#### D. CYCLODEXTRIN-COMPATIBLE SURFACTANT

The optional, but preferred, cyclodextrin-compatible surfactant, provides a low surface tension that permits the composition to spread readily and more uniformly on hydrophobic fabric surfaces, like polyester and nylon, during the washing and/or rinsing laundry cycles. It has been found that in aqueous solution, without such a surfactant, the present compositions tend not to spread satisfactorily. Furthermore, the composition containing a cyclodextrin-compatible surfactant can penetrate hydrophobic, oily soil better for improved odor control. Surprisingly, the combination of cyclodextrin-compatible surfactant and cyclodextrin significantly boosts the cleaning performance of conventional powder or liquid laundry detergents on greasy stains as well. The composition containing a cyclodextrin-compatible surfactant can also provide improved "in-wear" electrostatic control.

When cyclodextrin is present, the surfactant for use in providing the required low surface tension in the wash and/or rinse laundry solutions containing the composition of the present invention should be cyclodextrin-compatible, that is it should not substantially form a complex with the cyclodextrin so as to diminish performance of the cyclodextrin and/or the surfactant.

Complex formation diminishes both the ability of the cyclodextrin to absorb odors and the ability of the surfactant to lower the surface tension of the aqueous wash and/or rinse solutions.

Suitable cyclodextrin-compatible surfactants can be readily identified by the absence of effect of cyclodextrin on the surface tension provided by the surfactant. This is achieved by determining the surface tension (in dyne/cm) of aqueous solutions of the surfactant in the presence and in the absence of about 1% of a specific cyclodextrin in the solutions. The aqueous solutions contain surfactant at concentrations of approximately 0.5%, 0.1%, 0.01%, and 0.005%. The cyclodextrin can affect the surface activity of a surfactant by elevating the surface tension of the surfactant solution. If the surface tension at a given concentration in water differs by more than about 10% from the surface tension of the same surfactant in the 1% solution of the cyclodextrin, that is an indication of a strong interaction between the surfactant and the cyclodextrin. The preferred surfactants herein should have a surface tension in an aqueous solution that is different (lower) by less than about 10%, preferably less than about 5%, and more preferably less than about 1% from that of the same concentration solution containing 1% cyclodextrin.

#### 1. BLOCK COPOLYMERS

Nonlimiting examples of cyclodextrin-compatible nonionic surfactants include block copolymers of ethylene oxide and propylene oxide. Suitable block polyoxyethylene-polyoxypropylene polymeric surfactants, that are compatible with most cyclodextrins, include those based on ethylene glycol, propylene glycol, glycerol, trimethylolpropane and ethylenediamine as the initial reactive hydrogen compound. Polymeric compounds made from a sequential ethoxylation and propoxylation of initial compounds with a single reactive hydrogen atom, such as  $C_{12-18}$  aliphatic alcohols, are not generally compatible with the cyclodextrin. Certain of the block polymer surfactant compounds designated Pluronic<sup>®</sup> and Tetronic<sup>®</sup> by the BASF-Wyandotte Corp., Wyandotte, Michigan, are readily available.

Nonlimiting examples of cyclodextrin-compatible surfactants of this type include: <u>Pluronic Surfactants</u> with the general formula  $H(EO)_n(PO)_m(EO)_nH$ ,

wherein EO is an ethylene oxide group, PO is a propylene oxide group, and n and m are numbers that indicate the average number of the groups in the surfactants. Typical examples of cyclodextrin-compatible Pluronic surfactants are:

Name	Average MW	Average n	Average m
L-101	3,800	4	59
L-81	2,750	3	42
L-44	2,200	10	23
L-43	1,850	6	22
F-38	4,700	43	16
		-22-	

and mixtures thereof.

<u>Tetronic Surfactants</u> with the general formula:

wherein EO, PO, n, and m have the same meanings as above. Typical examples of cyclodextrin-compatible Tetronic surfactants are:

Name	Average MW	Average n	Average m
901	4,700	3	18
908	25,000	114	22,

and mixtures thereof.

Reverse Tetronic Surfactants

wherein EO, PO, n, and m have the same meanings as above. Typical examples of cyclodextrin-compatible Reverse Pluronic and Reverse Tetronic surfactants are:

#### Reverse Pluronic surfactants:

Name	Average MW	Average n	Average m
10 R5	1,950	8	22
25 R1	2,700	21	6
Reverse Tetro	onic surfactants		
Name	Average MW	Average n	Average m
Name 130 R2	Average MW 7,740	Average n 9	Average m 26
	•		•

#### 2. SILOXANE SURFACTANTS

A preferred class of cyclodextrin-compatible nonionic surfactants are the polyalkyleneoxide polysiloxanes having a dimethyl polysiloxane hydrophobic moiety and one or more hydrophilic polyalkylene side chains and have the general formula:

$$R^1$$
— $(CH_3)_2SiO$ — $[(CH_3)_2SiO]_a$ — $[(CH_3)(R^1)SiO]_b$ — $Si(CH_3)_2$ — $R^1$ 

wherein a + b are from about 1 to about 50, preferably from about 3 to about 30, more preferably from about 10 to about 25, and each  $R^1$  is the same or different and is selected from the group consisting of methyl and a poly(ethyleneoxide/propyleneoxide) copolymer group having the general formula:

$$-(CH_2)_n O(C_2 H_4 O)_c (C_3 H_6 O)_d R^2$$

with at least one R<sup>1</sup> being a poly(ethyleneoxide/propyleneoxide) copolymer group, and wherein n is 3 or 4, preferably 3; total c (for all polyalkyleneoxy side groups) has a value of from 1 to about 100, preferably from about 6 to about 100; total d is from 0 to about 14, preferably from 0 to about 3; and more preferably d is 0; total c+d has a value of from about 5 to about 150, preferably from about 9 to about 100 and each R<sup>2</sup> is the same or different and is selected from the group consisting of hydrogen, an alkyl having 1 to 4 carbon atoms, and an acetyl group, preferably hydrogen and methyl group.

Examples of this type of surfactants are the Silwet<sup>®</sup> surfactants which are available OSi Specialties, Inc., Danbury, Connecticut. Representative Silwet surfactants are as follows.

Name	Average MW	Average a+b	Average total c
L-7608	600	1	9
L-7607	1,000	2	17
L-77	600	1	9
L-7605	6,000	20	99
L-7604	4,000	21	53
L-7600	4,000	11	68
L-7657	5,000	20	76
L-7602	3,000	20	29

The molecular weight of the polyalkyleneoxy group  $(R^1)$  is less than or equal to about 10,000. Preferably, the molecular weight of the polyalkyleneoxy group is less than or equal to about 8,000, and most preferably ranges from about 300 to about 5,000. Thus, the values of c and d can be those numbers which provide molecular weights within these ranges. However, the number of ethyleneoxy units  $(-C_2H_4O)$  in the polyether chain  $(R^1)$  must be sufficient to render the polyalkyleneoxide polysiloxane water dispersible or water soluble. If propyleneoxy groups are present in the polyalkylenoxy chain, they can be distributed randomly in the chain or exist as

blocks. Preferred Silwet surfactants are L-7600, L-7602, L-7604, L-7605, L-7657, and mixtures thereof. Besides surface activity, polyalkyleneoxide polysiloxane surfactants can also provide other benefits, such as antistatic benefits, lubricity and softness to fabrics.

The preparation of polyalkyleneoxide polysiloxanes is well known in the art. Polyalkyleneoxide polysiloxanes of the present invention can be prepared according to the procedure set forth in U.S. Pat. No. 3,299,112, incorporated herein by reference. Typically, polyalkyleneoxide polysiloxanes of the surfactant blend of the present invention are readily prepared by an addition reaction between a hydrosiloxane (i.e., a siloxane containing siliconbonded hydrogen) and an alkenyl ether (e.g., a vinyl, allyl, or methallyl ether) of an alkoxy or hydroxy end-blocked polyalkylene oxide). The reaction conditions employed in addition reactions of this type are well known in the art and in general involve heating the reactants (e.g., at a temperature of from about 85° C. to 110° C.) in the presence of a platinum catalyst (e.g., chloroplatinic acid) and a solvent (e.g., toluene).

#### 3. ANIONIC SURFACTANTS

Nonlimiting examples of cyclodextrin-compatible anionic surfactants are the alkyldiphenyl oxide disulfonate, having the general formula:

wherein R is an alkyl group. Examples of this type of surfactants are available from the Dow Chemical Company under the trade name  $\operatorname{Dowfax}^{\circledR}$  wherein R is a linear or branched  $\operatorname{C}_6\text{-C}_{16}$  alkyl group. An example of these cyclodextrin-compatible anionic surfactant is  $\operatorname{Dowfax}$  3B2 with R being approximately a linear  $\operatorname{C}_{10}$  group. These anionic surfactants are preferably not used when the antimicrobial active or preservative, etc., is cationic to minimize the interaction with the cationic actives, since the effect of both surfactant and active are diminished.

#### 4. CASTOR OIL SURFACTANTS

The cyclodextrin-compatible surfactants useful in the present invention to form molecular aggregates, such as micelles or vesicles, with the cyclodextrin-incompatible materials of the present invention further include polyoxyethylene castor oil ethers or polyoxyethylene hardened castor oil ethers or mixtures thereof, which are either partially or fully hydrogenated. These ethoxylates have the following general formulae:

- or-

-or-

-or-

-or-

-or-

These ethoxylates can be used alone or in any mixture thereof. The average ethylene oxide addition mole number (i.e., l+m+n+x+y+z in the above formula) of these ethoxylates is generally from about 7 to about 100, and preferably from about 20 to about 80. Castor oil surfactants are commercially available from Nikko under the trade names HCO 40 and HCO 60 and from BASF under the trade names Cremphor<sup>TM</sup> RH 40, RH 60, and CO 60.

#### 5. SORBITAN ESTER SURFACTANTS

The sorbitan esters of long-chain fatty acids usable as cyclodextrin-compatible surfactants to form molecular aggregates with cyclodextrin-incompatible materials of the present invention include those having long-chain fatty acid residues with 14 to 18 carbon atoms, desirably 16 to 18 carbon atoms. Furthermore, the esterification degree of the sorbitan polyesters of long-chain fatty acids is desirably 2.5 to 3.5, especially 2.8 to 3.2. Typical examples of these sorbitan polyesters of long-chain fatty acids are sorbitan tripalmitate, sorbitan trioleate, and sorbitan tallow fatty acid triesters.

Other suitable sorbitan ester surfactants include sorbitan fatty acid esters, particularly the mono-and tri-esters of the formula:

HO 
$$\longrightarrow$$
 OR  $^{1}$  CHOR  $^{1}$  CH<sub>2</sub>OR  $^{2}$ 

Further suitable sorbitan ester surfactants include polyethoxylated sorbitan fatty acid esters, particularly those of the formula:

## 6. POLYETHOXYLATED FATTY ALCOHOL SURFACTANTS

Cyclodextrin-compatible surfactants further include polyethoxylated fatty alcohol surfactants having the formula:

wherein w is from about 0 to about 100, preferably from about 0 to about 80; y is 0 or 1; x is from about 1 to about 10; z is from about 1 to about 10; x+z+y=11 to 25, preferably 11 to 23.

Branched (polyethoxylated) fatty alcohols having the following formula are also suitable as cyclodextrin-compatible surfactants in the present compositions:

$$R-(OCH_2CH_2)_w-OH$$

wherein R is a branched alkyl group of from about 10 to about 26 carbon atoms and w is as specified above.

#### 7. GLYCEROL MONO-FATTY ACID ESTER SURFACTANTS

Further cyclodextrin-compatible surfactants include glycerol mono-fatty acid esters, particularly glycerol mono-stearate, oleate, palmitate or laurate.

#### 8. POLYETHYLENE GLYCOL FATTY ACID ESTER SURFACTANTS

Fatty acid esters of polyethylene glycol, particularly those of the following formula, are cyclodextrin-compatible surfactants useful herein:

$$R^1$$
-(OCH<sub>2</sub>CH<sub>2</sub>)<sub>w</sub> --OH

-or-

$$R^{1}$$
-(OCH<sub>2</sub>CH<sub>2</sub>)<sub>w</sub>-OR<sup>1</sup>

wherein R<sup>1</sup> is a stearoyl, lauroyl, oleoyl or palmitoyl residue; w is from about 2 to about 20, preferably from about 2 to about 8.

#### 9. FLUOROCARBON SURFACTANTS

Further cyclodextrin-compatible surfactants useful in the present compositions include fluorocarbon surfactants. Fluorocarbon surfactants are a class of surfactants wherein the hydrophobic part of the amphiphile comprises at least in part some portion of a carbon-based linear or cyclic moiety having fluorines attached to the carbon where typically hydrogens would be attached to the carbons together with a hydrophilic head group. Some typical nonlimiting fluorocarbon surfactants include fluorinated alkyl polyoxyalkylene, and fluorinated alkyl esters as well as ionic surfactants. Representative structures for these compounds are given below:

- (1)  $R_f R(R_1 O)_x R_2$
- (2)  $R_fR-OC(O)R_3$
- (3)  $R_fR-Y-Z$

#### (4) $R_fRZ$

wherein  $R_{\rm f}$  contains from about 6 to about 18 carbons each having from about 0 to about 3 fluorines attached. R is either an alkyl or alkylene oxide group which, when present, has from about 1 to about 10 carbons and R1 represents an alkylene radical having from about 1 to about 4 carbons. R2 is either a hydrogen or a small alkyl capping group having from about 1 to about 3 carbons. R<sub>3</sub> represents a hydrocarbon moiety comprising from about 2 to about 22 including the carbon on the ester group. This hydrocarbon can be linear, branched or cyclic saturated or unsaturated and contained moieties based on oxygen, nitrogen, and sulfur including, but not limited to ethers, alcohols, esters, carboxylates, amides, amines, thio-esters, and thiols; these oxygen, nitrogen, and sulfur moieties can either interrupt the hydrocabon chain or be pendant on the hydrocarbon chain. In structure 3, Y represents a hydrocarbon group that can be an alkyl, pyridine group, amidopropyl, etc. that acts as a linking group between the fluorinated chain and the hydrophilic head group. In structures 3 and 4, Z represents a cationic, anionic, and amphoteric hydrophilic head groups including, but not limited to carboxylates, sulfates, sulfonates, quaternary ammonium groups, and betaines. Nonlimiting commercially available examples of these structures include Zonyl® 9075, FSO, FSN, FS-300, FS-310, FSN-100, FSO-100, FTS, TBC from DuPont and Fluorad™ surfactants FC-430, FC-431, FC-740, FC-99, FC-120, FC-754, FC170C, and FC-171 from the 3M<sup>™</sup> company in St. Paul, Minnesota.

The cyclodextrin-compatible surfactants described above are either weakly interactive with cyclodextrin (less than 5% elevation in surface tension), or non-interactive (less than 1% elevation in surface tension). Conventional surfactants like sodium dodecyl sulfate and dodecanolpoly(6)ethoxylate are strongly interactive, with more than a 10% elevation in surface tension in the presence of a typical cyclodextrin like hydroxypropyl beta-cyclodextrin and methylated beta-cyclodextrin.

Typical levels of cyclodextrin-compatible surfactants in the present concentrated, non-liquid compositions are from about 0.1% to about 20%, preferably from about 3% to about 10%, more preferably from about 5% to about 9%, by weight of the concentrated, non-liquid composition.

#### E. ANTIMICROBIAL ACTIVES

A water-soluble antimicrobial active is useful in providing protection against organisms that become attached to the treated fabrics. The antimicrobial active is preferably cyclodextrin-compatible, e.g., not substantially forming complexes with the cyclodextrin in the concentrated, non-liquid composition. The free, uncomplexed antimicrobial active provides an optimum antibacterial performance.

Sanitization of fabrics can be achieved by the compositions of the present invention containing, antimicrobial actives, e.g., biguanides, quaternary compounds, peroxygen bleach / bleach activator system, and mixtures thereof.

#### 1. **BIGUANIDES**

Some of the more robust cyclodextrin-compatible antimicrobial halogenated compounds which can function as disinfectants/sanitizers as well as finish product preservatives, and are useful in the compositions of the present invention include 1,1'-hexamethylene bis(5-(p-chlorophenyl)biguanide), commonly known as chlorhexidine, and its salts, e.g., with hydrochloric, acetic and gluconic acids. The digluconate salt is highly water-soluble, about 70% in water, and the diacetate salt has a solubility of about 1.8% in water. When chlorhexidine is used as a sanitizer in the present invention it is typically present at a level of from about 0.001% to about 0.4%, preferably from about 0.002% to about 0.3%, and more preferably from about 0.05% to about 0.2%, by weight of the usage composition. In some cases, a level of from about 1% to about 2% may be needed for virucidal activity.

Other useful biguanide compounds include Cosmoci<sup>®</sup> CQ<sup>®</sup>, Vantocil<sup>®</sup> IB, including poly (hexamethylene biguanide) hydrochloride. Other useful cationic antimicrobial agents include the bis-biguanide alkanes. Usable water soluble salts of the above are chlorides, bromides, sulfates, alkyl sulfonates such as methyl sulfonate and ethyl sulfonate, phenylsulfonates such as p-methylphenyl sulfonates, nitrates, acetates, gluconates, and the like.

Examples of suitable bis biguanide compounds are chlorhexidine; 1,6-bis-(2-1,6-di- $(N_1,N_1)$ -phenyldiguanido- $N_5.N_5$ )-hexane ethylhexylbiguanidohexane)dihydrochloride; 1,6-di-(N<sub>1</sub>,N<sub>1</sub>'-phenyl-N<sub>1</sub>,N<sub>1</sub>'-methyldiguanido-N<sub>5</sub>,N<sub>5</sub>')-hexane tetrahydrochloride; dihydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-o-chlorophenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')-hexane dihydrochloride: 1.6 $di(N_1,N_1'-2,6-dichlorophenyldiguanido-N_5,N_5') hexane \ dihydrochloride; \ 1,6-di[N_1,N_1'-.beta.-(p-1)] hexane \ dihydrochlori$ methoxyphenyl) diguanido-N5,N5']-hexane dihydrochloride; 1,6-di(N1,N1'-.alpha.-methyl-.beta.-1,6-di(N<sub>1</sub>,N<sub>1</sub>'-p-nitrophenyldiguanidodihydrochloride; phenyldiguanido-N5,N5')-hexane dihydrochloride;.omega.:.omega.'-di-(N1,N1'-phenyldiguanido-N5,N5')-di-n-N<sub>5</sub>,N<sub>5</sub>')hexane  $dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega:omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5')-di-n-dihydrochloride;.omega'-di(N_1,N_1'-p-chlorophenyldiguanido-N_5,N_5'-q-chlorophenyldiguanido-N_5,N_5'-q-chlorophenyldiguanido-N_5,N_5'-q-chlorophenyldiguanido-N$ propylether 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-2,4-dichlorophenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')hexane tetrahydrochloride; propylether tetrahydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-p-methylphenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')hexane dihydrochloride; 1,6 $di(N_1,N_1'-2,4,5-trichlorophenyldiguanido-N_5,N_5') hexane \\ tetrahydrochloride; \\ 1,6-di[N_1,N_1'-2,4,5-trichlorophenyldiguanido-N_5,N_5'] hexane \\ 1,6-di[N_1,N_1'-2,4,5-trichlorophenyldiguanido$ N<sub>1</sub>'-p-chlorophenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')m-xylene dihydrochloride;  $1,12-di(N_1,N_1'-p$  $chlorophenyldiguanido-N_5,N_5') \quad dodecane \quad dihydrochloride; \quad 1,10-di(N_1,N_1'-phenyldiguanido-n_5,N_5')$ tetrahydrochloride; 1,12-di(N<sub>1</sub>,N<sub>1</sub>'-phenyldiguanido-N<sub>5</sub>,N<sub>5</sub>') N<sub>5</sub>,N<sub>5</sub>')-decane tetrahydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-o-chlorophenyldiguanido-N<sub>5</sub>,N<sub>5</sub>') hexane dihydrochloride; 1,6-

 $di(N_1,N_1'\text{-p-chlorophenyldiguanido-}N_5,N_5')\text{-hexane} \quad tetrahydrochloride}; \quad ethylene \quad bis \quad (1\text{-tolylorophenyldiguanido-}N_5,N_5')\text{-hexane} \quad tetrahydrochloride}; \quad ethylene \quad e$ biguanide); ethylene bis (p-tolyl biguanide); ethylene bis(3,5-dimethylphenyl biguanide); ethylene bis(p-tert-amylphenyl biguanide); ethylene bis(nonylphenyl biguanide); ethylene bis (phenyl biguanide); ethylene bis (N-butylphenyl biguanide); ethylene bis (2,5-diethoxyphenyl biguanide); ethylene bis(2,4-dimethylphenyl biguanide); ethylene bis(o-diphenylbiguanide); ethylene bis(mixed amyl naphthyl biguanide); N-butyl ethylene bis(phenylbiguanide); trimethylene bis(o-tolyl biguanide); N-butyl trimethylene bis(phenyl biguanide); and the corresponding pharmaceutically acceptable salts of all of the above such as the acetates; gluconates; hydrochlorides; hydrobromides; citrates; bisulfites; fluorides; polymaleates; Ncoconutalkylsarcosinates; phosphites; hypophosphites; perfluorooctanoates; silicates; sorbates; salicylates; maleates; tartrates; fumarates; ethylenediaminetetraacetates; iminodiacetates; cinnamates; thiocyanates; arginates; pyromellitates; tetracarboxybutyrates; benzoates; glutarates; monofluorophosphates; and perfluoropropionates, and mixtures thereof. Preferred antimicrobials from this group are 1,6-di-(N<sub>1</sub>,N<sub>1</sub>'-phenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')-hexane tetrahydrochloride; 1,6dihydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-2,6di(N<sub>1</sub>,N<sub>1</sub>'-o-chlorophenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')-hexane dihydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-2,4dichlorophenyldiguanido-N5,N5')hexane dichlorophenyldiguanido-N5,N5')hexane tetrahydrochloride; 1,6-di[N<sub>1</sub>,N<sub>1</sub>'-.alpha.-(pchlorophenyl) ethyldiguanido-N5,N5'] hexane dihydrochloride;.omega.:.omega.'di(N1, N1'-p- $1,12-di(N_1,N_1'-p$ dihydrochloride; chlorophenyldiguanido-N5,N5')m-xylene 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-odihydrochloride; dodecane chlorophenyldiguanido-N5,N5') dihydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-phexane chlorophenyldiguanido-N5,N5') chlorophenyldiguanido-N5,N5')-hexane tetrahydrochloride; and mixtures thereof; more 1,6-di( $N_1,N_1$ '-o-chlorophenyldiguanido- $N_5,N_5$ ')-hexane dihydrochloride; 1,6di(N<sub>1</sub>,N<sub>1</sub>'-2,6-dichlorophenyldiguanido-N<sub>5</sub>,N<sub>5</sub>')hexane dihydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-2,4-1,6-di[N<sub>1</sub>,N<sub>1</sub>'-.alpha.-(ptetrahydrochloride; dichlorophenyldiguanido-N5,N5')hexane chlorophenyl) ethyldiguanido-N5,N5'] hexane dihydrochloride;.omega.:.omega.'di(N1, N1'-p-1,12-di(N<sub>1</sub>,N<sub>1</sub>'-pdihydrochloride; chlorophenyldiguanido-N5,N5')m-xylene dodecane dihydrochloride; 1,6-di(N<sub>1</sub>,N<sub>1</sub>'-ochlorophenyldiguanido-N5,N5')  $1,6-di(N_1,N_1'-p$ chlorophenyldiguanido-N5,N5') hexane dihydrochloride; chlorophenyldiguanido-N5,N5')-hexane tetrahydrochloride; and mixtures thereof. As stated hereinbefore, the bis biguanide of choice is chlorhexidine its salts, e.g., digluconate, dihydrochloride, diacetate, and mixtures thereof.

#### 2. OUATERNARY COMPOUNDS

A wide range of quaternary compounds can also be used as antimicrobial actives, especially in conjunction with the preferred surfactants, and especially for compositions of the

present invention that do not contain cyclodextrin as the odor control agent. Non-limiting examples of useful quaternary compounds include: (1) benzalkonium chlorides and/or substituted benzalkonium chlorides such as commercially available Barquat® (available from Lonza), Maquat® (available from Mason), Variquat® (available from Witco/Sherex), and Hyamine® (available from Lonza); (2) di(C<sub>6</sub>-C<sub>14</sub>)alkyl di short chain (C<sub>1-4</sub> alkyl and/or hydroxyalkl) quaternary such as Bardac® products of Lonza, (3) N-(3-chloroallyl) hexaminium chlorides such as Dowicide<sup>®</sup> and Dowicil<sup>®</sup> available from Dow; (4) benzethonium chloride such as Hyamine<sup>®</sup> 1622 from Rohm & Haas; (5) methylbenzethonium chloride represented by Hyamine® 10X supplied by Rohm & Haas, (6) cetylpyridinium chloride such as Cepacol chloride available from of Merrell Labs. Examples of the preferred dialkyl quaternary compounds are di(C<sub>8</sub>-C<sub>12</sub>)dialkyl dimethyl ammonium chloride, such as didecyldimethylammonium chloride (Bardac 22), and dioctyldimethylammonium chloride (Bardac 2050). Typical concentrations for biocidal effectiveness of these quaternary compounds range from about 0.003% to about 2%, preferably from about 0.006% to about 1.2%, and more preferably from about 0.1% to about 0.8% by weight of the concentrated, non-liquid compositions.

The cyclodextrin-compatible surfactants, when added to the concentrated, non-liquid compositions tend to provide improved antimicrobial action. This is especially true for the siloxane surfactants, and especially when the siloxane surfactants are combined with the chlorhexidine or Bardac<sup>®</sup> antimicrobial actives.

#### 3. PEROXYGEN BLEACH / BLEACH ACTIVATOR SYSTEM

A peroxygen bleach / bleach activator system can also be incorporated in the present compositions as an antimicrobial active, especially in solid compositions such as granules. tablets, laundry bars, substrates/sheets, and the like. The combined peroxygen bleach and bleach activator function to kill or reduce microorganisms on fabrics in laundry process. The peroxygen bleach / bleach activator system useful herein comprises a peroxygen bleaching compound and a bleach activator.

# a. PEROXYGEN BLEACHING COMPOUND

The peroxygen bleaching compounds useful herein are those capable of yielding hydrogen peroxide in an aqueous solution. These compounds are well known in the art and include hydrogen peroxide and the alkali metal peroxides, organic peroxide bleaching compounds such as urea peroxide, and inorganic persalt bleaching compounds, such as the alkali metal

perborates, percarbonates, perphosphates, and the like. Mixtures of two or more such bleaching compounds can also be used, if desired.

Preferred peroxygen bleaching compounds include sodium perborate, commercially available in the form of mono- and tetra-hydrate, sodium carbonate peroxyhydrate, sodium pyrophosphate peroxyhydrate, urea peroxyhydrate, and sodium peroxide. Particularly preferred are sodium perborate tetrahydrate and, especially, sodium perborate monohydrate. Sodium perborate monohydrate is especially preferred because it is very stable during storage and yet still dissolves very quickly in the bleaching solution. It is believed that such rapid dissolution results in the formation of higher levels of percarboxylic acid and, thus, enhanced surface bleaching performance.

The level of peroxygen bleach within compositions of the invention is from about 0.1% to about 95% preferably from about 1% to about 60%, and more preferably from about 1% to about 20%.

#### b. BLEACH ACTIVATOR

The bleach activators within the invention include those described in U.S. Patent No. 4,412,934, at col. 5, line 40 to col. 7, line 55; which is incorporated herein by reference. A particularly preferred bleach activator herein is nononyloxybenzene sulfonate.

The level of bleach activator within the compositions of the invention is from about 0.1% to about 60%, preferably from about 0.5% to about 40%, and more preferably from about 0.5% to about 20%.

#### F. FILLER MATERIALS

Preferred filler materials are alkaline earth metals such as sodium, magnesium, calcium and even sometimes aluminum salts of carbonates, bicarbonates, sulfates, chlorides and mixtures thereof. The water-soluble fillers are more preferred given that they do not interfere with the cleaning of a conventional laundry detergent, and as such, the sodium salts are most preferred.

Another suitable, but less preferred, filler material belong to the silicate-containing materials such as powder sodium silicates, alumino-silicates such as zeolites, and even clay. Additional, but least preferred, types belong to cellulosic filler materials such as starch and flour.

Additional suitable filler materials, especially for laundry detergent bars, are described in Trajano et al., WO 00/017,312 A1 (P&G Case AA-337); and Trajano et al., WO 97/044,434 A1 (P&G Case JA-146); which are both incorporated herein by reference.

#### G. OTHER OPTIONAL INGREDIENTS

The concentrated, non-liquid compositions of the present invention can further comprise other optional ingredients. Such additional ingredients will generally depend upon the form of the concentrated, non-liquid compositions. For example, gel or paste compositions will generally comprise thickening agents as optional materials. Suitable thickening agents in the present compositions include guar gum, hydrophobically-modified cellulose, polyvinyl alcohol, and the like, including those disclosed in U.S. Patent No. 4,260,528, which is incorporated herein by reference. Most of the thickening agents work in the range of from about 0.1 to about 10%, most preferably not more than about 5%. Moreover, the gel compositions would have water as a major component, but still have a non-liquid viscosity according to the present invention. The thickening agents can cross-linked the whole matrix by forming hydrogen bonds with water molecules.

Concentrated, tablet compositions herein will typically comprise an effervescing agent including carboxylic acids such as citric acid, maleic acid, and the like, and alkali metal carbonates and/or bicarbonates, such as sodium bicarbonate. Tablet compositions can optionally further comprise tablet coating agents, such as sebacic acid and Nymcel<sup>®</sup>. Sebacic acid is a dicarboxylic acid, wax-like and is solid at room temperature. It is somewhat brittle, yet hard and is used to protect the tablet from breakage during handling and transport. Nymcel<sup>®</sup> on the other hand is a hydrophobically-modified cellulose that is insoluble in water. It swells when in contact with water and it pushes against the sebacic acid coating, breaking it and consequently releasing the actives in the tablet.

Binders are also optional, but preferred, ingredients herein, especially for tablet compositions, water-soluble sheets, and bar compositions. The binder is preferably an organic compound having a melting point or a softening point of from about 40°C to about 160°C. Examples of such binders include polyethylene glycol having a molecular weight of from about 1,500 to about 20,000, preferably from about 1,500 to about 8,000; a polyethylene glycol alkyl ether having an alkyl group of from about 8 to about 22 carbon atoms; polyethylene glycol alkyl phenol ether having an alkyl group of from about 8 to about 22 carbon atoms; an aliphatic acid having an alkyl group of from about 12 to about 37 carbon atoms; an ethylene oxide / propylene oxide block copolymers; and mixtures thereof. Binders with much lower molecular weights are also acceptable in tablet compositions.

# **EXAMPLES**

The following Examples I to III are non-limiting examples of concentrated, powder (or granular) compositions of the present invention.

Ingredient	Ī	ĪĪ	ĪII
HPBCD <sup>a</sup>	90.0	45.0	45.0
HCO-60 <sup>b</sup>	9.0	9.0	9.0
Perfume	1.0	1.8	1.8
Na <sub>2</sub> CO <sub>3</sub>			24.2
Na <sub>2</sub> SO <sub>4</sub>		44.2	
Perborate			10.0
NOBS <sup>c</sup>			10.0
TOTAL	100.0%	100.0%	100.0%

<sup>&</sup>lt;sup>a</sup> Hydroxypropyl beta-cyclodextrin.

The following Examples IV to VIII are non-limiting examples of concentrated, tablet compositions of the present invention. Examples IV, VI, and VIII are uncoated tablet compositions, whereas Examples V and VII are coating tablet compositions.

For the non-coated tablet, its effervescence source (i.e., citric acid/alkali) can also act as a disintegrant. However, for the coated tablets, disintegrants like Nymcel are typically needed. The coating is a waxy but brittle material, e.g., sebacic acid. Nymcel swells when coated with water, and as it does, it breaks apart the sebacic coating, releasing the tablet core which contains the cyclodextrin.

Processes for making such tablets are known in the art and are described, for example, in U.S. Patent Nos. 6,083,895; 4,219,435; and 6,087,311.

Ingredient	<u>IV</u>	<u>V</u>	<u>VI</u>	VII	VIII
HPBCD <sup>a</sup>	45.0	40.0	45.0	40.0	40.0
HCO-60 <sup>b</sup>	9.0	9.0	9.0	9.0	9.0
Perfume	1.0	1.0	1.0	1.0	1.0
Na <sub>2</sub> CO <sub>3</sub>	15.0	12.0	0.00	18.0	12.0
NaHCO <sub>3</sub>	0.00	0.00	15.0	4.0	10.0
Perborate	10.0	10.0	10.0		
NOBS <sup>c</sup>	10.0	10.0	10.0		

<sup>&</sup>lt;sup>b</sup> Hydrogenated castor oil surfactant having an average ethlyene oxide mole addition number of 60 and is commercially available from Nikko.

<sup>&</sup>lt;sup>c</sup> Nononyloxybenzene Sulfonate.

Citric acid	7.0	5.0	7.0	5.0	5.0
PEG 400 (binder)	3.0	3.0	3.0	3.0	3.0
Zeolite <sup>d</sup>					20.0
(Tablet Coating)		(10%)		(10%)	
Sebacic Acid		8.0		8.0	
Nymcel <sup>e</sup>		2.0		2.0	
TOTAL	100.0%	100.0%	100.0%	100.0%	100.0%

<sup>&</sup>lt;sup>a</sup> Hydroxypropyl beta-cyclodextrin.

The following Examples IX to XII are non-limiting examples of concentrated, gel (or paste) compositions of the present invention. Processes for making such gels are known in the art and are described, for example, in U.S. Patent Nos. 5,415,801; 5,269,974; and 5,320,783.

<u>Ingredient</u>	<u>IX</u>	<u>X</u>	<u>XI</u>	XII
HPBCD <sup>a</sup>	45.0	45.0	45.0	45.0
HCO-60 <sup>b</sup>	9.0	9.0	9.0	9.0
Perfume	1.0	1.0	1.0	1.0
Carbopol 934 °	0.50			
Guar gum		1.0		
PVA <sup>d</sup>			15.0	
Cetylhydroxyethyl				2.0
cellulose				
Preservative (Kathon)	0.003			0.003
Bacteriocide				
Bardac®		2.5	2.5	

<sup>&</sup>lt;sup>b</sup> Hydrogenated castor oil surfactant having an average ethlyene oxide mole addition number of 60 and is commercially available from Nikko.

<sup>&</sup>lt;sup>c</sup> Nononyloxybenzene Sulfonate.

<sup>&</sup>lt;sup>d</sup> Zeolite is used as a moisture sink to prevent possible hydration of the tablets given that Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> are hygroscopic materials.

<sup>&</sup>lt;sup>e</sup> Nymcel™ is a hydrophobically modified carboxy methyl cellulose used as a disintegrant in the tablet coating.

NaOH/HCl for pH	as needed	as needed	as needed	as needed
control				
H2O	to make	to make 100%	to make	to make 100%
	100%		100%	•
Gel pH <sup>e</sup>	6.0-10.0	5.0-7.0	5.0-7.0	6.0-7.0

<sup>&</sup>lt;sup>a</sup> Hydroxypropyl beta-cyclodextrin.

<sup>e</sup> The pH of the gel should be controlled carefully given that some of the thickeners' viscosity is pH-dependent.

The following Examples XIII to XVI are non-limiting examples of articles of manufacture of the present invention. Examples XIII and XIV are water-insoluble sheets impregnated with a powder or granular composition similar to Examples I to III; whereas Examples XV and XVI are water-soluble sheets composed of a PVA film. A process for manufacturing water-insoluble sheets herein is described in U.S. Patent No. 5,348,667. A process for manufacturing water-soluble sheets herein is described in U.S. Patent Nos. 5,224,601; and 4,806,261.

Ingredient	XIII	XIV	XV	XVI
HPBCD <sup>a</sup>	45.0	45.0	35.0	35.0
HCO-60 <sup>b</sup>	4.5	4.5	4.5	4.5
Perfume	0.5	0.5	0.5	0.5
H <sub>2</sub> O	< 1.00	< 1.00	20.0	10.0
Non-woven sheet c	~ 50.00	~ 50.00		
Calcium Bentonite clay d		5.00		
PVA <sup>e</sup>			40.0	40.0
PEG-4000 <sup>f</sup>				10.0
TOTAL	100.0%	100.0%	100.0%	100.0%

<sup>&</sup>lt;sup>b</sup> Hydrogenated castor oil surfactant having an average ethlyene oxide mole addition number of 60 and is commercially available from Nikko.

<sup>&</sup>lt;sup>c</sup> Carbopol® 934 is a crosslinked carboxyvinyl polymer thickening agent.

<sup>&</sup>lt;sup>d</sup> Polyvinyl alcohol; it is preferred that a mixture of different MW's of PVA be used to have good film strength and solubility. It is further preferred to use PVA's which are more than 80% hydrolyzed, and most preferably with 4 acyl groups.

- <sup>a</sup> Hydroxypropyl beta-cyclodextrin.
- <sup>b</sup> Hydrogenated castor oil surfactant having an average ethlyene oxide mole addition number of 60 and is commercially available from Nikko.
- <sup>c</sup> The non-woven sheet is 70% 3-denier with 30% polyvinyl acetate; 1 <sup>9</sup>/16 inches in thickness; impregnated with a 2:1 ratio of "lotion" (CD/perfume/HCO-60/water, with CD and water in equal proportions): non-woven substrate. Other suitable non-woven sheets are described in U.S. Patent Nos. 5,929,026 and 5,470,492, which are incorporated herein by reference.
- <sup>d</sup> Calcium bentonite clay is added to control the viscosity of the CD/perfume/HCO-60/water mixture.
- e Polyvinyl alcohol; it is preferred that a mixture of different MW's of PVA be used to have good film strength and solubility. It is further preferred to use PVA's which are more than 80% hydrolyzed, and most preferably with 4 acyl groups.

The following Examples XVII to XIX are non-limiting examples of laundry bar compositions of the present invention. A process for manufacturing laundry bar compositions includes: (a) premix perfume and CD and water (CD:H2O at 1:1 ratio); (b) add HCO-60 while continually mixing in a KM-mixer (high-shear mixer); (c) add in the fillers (Na<sub>2</sub>CO<sub>3</sub>, CaCO<sub>3</sub>, etc.); (d) add the binders (glycerin, PEG 4000) and continue mixing; (e) add Perborate/NOBS last, if any; and (f) dump the mixture in a Duplex Plodder; plod and cut evenly into bars.

<u>Ingredient</u>	XVII	XVIII	XIX
HPBCD <sup>a</sup>	45.0	45.0	40.0
HCO-60 <sup>b</sup>	9.0	9.0	9.0
Perfume	1.0	1.0	1.0
Na <sub>2</sub> CO <sub>3</sub>	20.5		10.0
CaCO <sub>3</sub>	<u></u>	39.5	14.5
Perborate	10.0		10.0
NOBS	10.0		10.0
Glycerin d	5.0	5.0	3.0
PEG 4000			2.0
Moisture	0.5	0.5	0.5
TOTAL	100.0%	100.0%	100.0%

<sup>&</sup>lt;sup>a</sup> Hydroxypropyl beta-cyclodextrin.

<sup>&</sup>lt;sup>f</sup> Polyethylene glycol having a MW of 4,000 is used as a plasticizer.

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b Hydrogenated castor oil surfactant having an average ethlyene oxide mole addition number of 60 and is commercially available from Nikko.

- <sup>c</sup> Tallow/coconut soap is a functional filler and the formulation as a whole can be used to aid the user during rinsing. The soap will depress sudsing and impart some sort of fabric softening - on fabrics washed with anionic detergent.
- d Glycerin is a binder.
- e Starch is a cross-linking agent for soap. Hydrophobically-modified cellulose can also be used as a starch-substitute.
- f Sodium tripolyphosphate (STPP) is a calcium sequestrant. This formulation can supplement the overall cleaning.

#### WHAT IS CLAIMED IS:

1. A method of reducing odor on fabrics in a wash or rinse solution of a laundry process, said method comprising the step of contacting said fabrics with an effective amount to reduce odor on said fabrics of a odor control agent selected from the group consisting of: uncomplexed cyclodextrin; odor blocker; class I and/or class II aldehydes; flavanoid; metallic salt; and mixtures thereof.

- 2. The method of Claim 1 wherein said odor control agent is uncomplexed cyclodextrin, said uncomplexed cyclodextrin provided at a level of at least about 20 ppm, by weight of said wash or rinse solution.
- 3. The method of Claim 2 wherein said uncomplexed cyclodextrin is provided at a level of at least about 30 ppm, by weight of said wash or rinse solution.
- 4. The method of Claim 2 wherein said uncomplexed cyclodextrin is beta-cyclodextrin.
- 5. The method of Claim 4 wherein said beta-cyclodextrin is hydroxyalkyl beta-cyclodextrin.
- 6. The method of Claim 4 wherein said beta-cyclodextrin is alkylated beta-cyclodextrin.
- 7. The method of Claim 1 wherein said odor control agent is odor blocker, said odor blocker provided at a level of at least about 0.004 ppm, by weight of said wash or rinse solution.
- 8. The method of Claim 1 wherein said odor control agent is class I and/or class II aldehydes, said class I and/or class II aldehydes provided at a level of at least about 0.05 ppm, by weight of said wash or rinse solution.
- 9. The method of Claim 1 wherein said odor control agent is flavanoid, said flavanoid provided at a level of at least about 0.1 ppm, by weight of said wash or rinse solution.
- 10. The method of Claim 1 wherein said odor control agent is metallic salt, said metallic salt provided at a level of at least about 0.2% by weight of said wash or rinse solution.

11. A method of reducing odor on fabrics in a laundry process, said method comprising the steps of:

- (a) placing said fabrics in an aqueous laundry wash or rinse solution;
  - (b) optionally, adding a laundry detergent composition to said wash or adding a fabric softening composition to said rinse solution;
- (c) adding an effective amount to reduce malodor on said fabric of a concentrated, non-liquid composition to said wash or rinse solution, said concentrated, non-liquid composition comprising:
  - (i) a odor control agent selected from the group consisting of:
    - (aa) uncomplexed cyclodextrin;
    - (bb) odor blocker;
    - (cc) class I and/or class II aldehydes;
    - (dd) flavanoid;
    - (ee) metallic salt; and
    - (ff) mixtures thereof;
  - (ii) an additional component selected from the group consisting of:
    - (aa) uncomplexed perfume;
    - (bb) perfume carrier complex;
    - (cc) cyclodextrin-compatible surfactant;
    - (dd) antimicrobial active;
    - (ee) filler materials; and
    - (ff) mixtures thereof;
  - (d) agitating said fabrics in said wash or rinse solution;
  - (e) optionally, rinsing said fabrics with a final rinse solution comprising water; and
- (f) drying said fabrics.
- 12. A concentrated, non-liquid composition for reducing malodor on fabrics in a laundry process, said composition comprising:
  - (a) a odor control agent selected from the group consisting of:
    - (i) uncomplexed cyclodextrin;
    - (ii) odor blocker;
    - (iii) class I and/or class II aldehydes;
    - (iv) flavanoid;

- (v) metallic salt; and
- (vi) mixtures thereof;
- (b) an additional component selected from the group consisting of:
  - (i) uncomplexed perfume;
  - (ii) perfume carrier complex;
  - (iii) cyclodextrin-compatible surfactant;
  - (iv) antimicrobial active;
  - (v) filler materials; and
  - (vi) mixtures thereof;

wherein said composition is essentially free of surfactant that is not cyclodextrin-compatible.

- 13. The composition of Claim 12, wherein said composition is a powder or granular composition; and said odor control agent is uncomplexed cyclodextrin.
- 14. The composition of Claim 13, wherein said powder or granular composition further comprises uncomplexed perfume.
- 15. The composition of Claim 14, wherein said powder or granular composition further comprises perfume carrier complex.
- 16. The composition of Claim 15, wherein said powder or granular composition further comprises a cyclodextrin-compatible surfactant.
- 17. The composition of Claim 16, wherein said cyclodextrin-compatible surfactant is a castor oil surfactant.
- 18. The composition of Claim 13, wherein said cyclodextrin is present at a level of at least about 20%, by weight of the composition.
- 19. The composition of Claim 18, wherein said cyclodextrin is hydroxyalkyl betacyclodextrin.

20. The composition of Claim 12, wherein said composition is a composition form selected from the group consisting of powder, granular, tablet, gel, paste, substrate, sheet, laundry bar, and combinations thereof.