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(54) **Granular detergent compositions containing sodium aluminosilicate or other phosphorus-free detergency builders.**

(57) Granular detergent compositions containing sodium aluminosilicate or other phosphorus-free detergency builders with reduced caking and hygroscopicity tendencies are disclosed. The compositions contain not more than 4.5% sodium silicate and from about 0.3% to about 6.0% of polyethylene glycol or other polymers containing at least about 50% ethylene oxide by weight, the polymer having a melting point not less than about 35°C.

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Technical Field

The present invention relates to granular detergent compositions containing a detergent surfactant, an aluminosilicate ion exchange material or other specified non-phosphorus containing detergency builders, and polyethylene glycol or other water-soluble polymers containing at least 50% ethylene oxide by weight. The compositions herein, which contain no phosphorus or reduced levels of phosphorus-containing materials and not more than about 4.5% by weight of alkali metal silicate materials, provide granules having superior free-flowing characteristics, solubility in the laundering solution and reduced hygroscopicity.

Granular detergent compositions often contain relatively high levels of phosphate builder materials, particularly sodium tripolyphosphate and sodium pyrophosphate. When a water slurry containing such phosphates is dried granules are produced which exhibit desirable physical properties, i.e., crisp, durable and free-flowing. Moreover, the phosphate material readily disintegrates in the laundering solution so that no insoluble residue is left on the fabrics.

Alkali metal silicates are usually included in granular detergents for corrosion inhibition and processing reasons. When phosphate builders are removed from detergents, the level of silicate has often been increased since silicates also dry to a glassy film capable of strengthening detergent granule walls and enhancing free-flowing characteristics. However, the increased silicate level enhances silicate polymerization during drying, resulting in the formation of insoluble silicates. The exposure of the silicate to carbon dioxide during drying and storage can also reduce its solubility, resulting in detergent granules with unacceptably high level of insoluble material being deposited on fabrics. The insolubles problem can be particularly severe when

the detergent composition also contains a water-insoluble aluminosilicate detergency builder.

Background Art

5 U.S. Patent 3,985,669, Krummel et al, issued October 12, 1976, describes the use of low levels of alkali metal silicates in granular detergent compositions containing aluminosilicate builder materials to provide both corrosion inhibition and crispness benefits without enhancing deposition of the aluminosilicates onto fabrics.

10 U.S. Patent 4,379,080, Murphy, issued April 5, 1983, discloses spray-dried detergent granules containing a film-forming polymer to provide physical property benefits in low silicate compositions containing aluminosilicate.

15 U.S. Patent 3,960,780, Murata et al, issued June 1, 1976 discloses granular detergent compositions containing an alkyl ether sulfate surfactant and polyethylene glycol at a level of from 10% to 100% of the surfactant.

Summary of The Invention

20 The present invention encompasses granular detergent compositions prepared by drying aqueous slurries which after drying provide compositions comprising:

- 25 (a) from about 5% to about 50% by weight of a detergent surfactant selected from the group consisting of anionic, nonionic, zwitterionic, ampholytic and cationic surfactants and mixtures thereof, provided that at least about 50% of the total surfactant by weight is a non-soap anionic surfactant;
- 30 (b) from about 10% to about 70% by weight of a non-phosphorus containing detergency builder selected from the group consisting of sodium aluminosilicate, sodium carbonate, polycarboxylic acids and water-soluble salts thereof, and mixtures thereof;
- 35 (c) from 0% to about 4.5% of a water-soluble silicate material;
- (d) from about 0.3% to about 6.0% by weight of a

water-soluble polymeric material containing at least about 50% ethylene oxide by weight, said polymer having a melting point not less than about 35°C.

Detailed Description of The Invention

5 The granular detergent compositions of the present invention contain, as essential components, a detergent surfactant, a non-phosphorus containing detergency builder and a water-soluble polymeric material containing at least about 50% ethylene oxide by weight. The compositions contain not more than about 4.5% by
10 weight of water-soluble silicate materials.

 The compositions herein are prepared by drying an aqueous slurry comprising the above components. Order of component addition to the slurry is not critical, but in a preferred
15 embodiment the polymeric material is mixed with surfactants and other organic materials before mixing with the remaining components. The slurry generally contains from about 25% to about 50% water, whereas the dried granules contain from about 3% to about 15% water. The drying operation can be accomplished
20 by any convenient means, for example, by using spray-drying towers, both counter-current and co-current, fluid beds, flash-drying towers, both counter-current and co-current, fluid beds, flash-drying equipment, or industrial microwave or oven drying equipment. Spray-drying in heated air is particularly
25 preferred, using a process such as disclosed in U.S. Patent 3,629,955, Davis et al issued December 28, 1971, incorporated herein by reference.

 The compositions of the invention quickly disintegrate in laundering solution and leave little or no insoluble residue on
30 fabrics. Moreover, the polymeric material does not enhance the deposition of the aluminosilicate material onto fabrics, as do the higher levels of water-soluble silicates necessary for granule crispness in the absence of the polymeric material.

Detergent Surfactant

35 The detergent compositions herein contain from about 5% to about 50% by weight of an organic surfactant selected from the

group consisting of anionic, nonionic, zwitterionic, ampholytic and cationic surfactants, and mixtures thereof. The surfactant preferably represents from about 10% to about 40%, and more preferably from about 15% to about 35%, by weight of the detergent composition. Surfactants useful herein are listed in U.S. Patent 3,664,961, Norris, issued May 23, 1972, and in U.S. Patent 3,919,678, Laughlin et al, issued December 30, 1975, both incorporated herein by reference. Useful cationic surfactants also include those described in U.S. Patent 4,222,905, Cockrell, issued September 16, 1980, and in U.S. Patent 4,239,659, Murphy, issued December 16, 1980, both incorporated herein by reference.

At least about 50%, and preferably at least about 75%, of the total surfactant by weight consists of non-soap anionic surfactants. This is necessary to provide desired physical and detergency characteristics.

Useful anionic surfactants include the water-soluble salts, preferably the alkali metal salts, of organic sulfuric reaction products having in their molecular structure an alkyl group containing from about 10 to about 20 carbon atoms and a sulfonic acid or sulfuric acid ester group. (Included in the term "alkyl" is the alkyl portion of acyl groups.) Examples of this group of synthetic surfactants are the sodium and potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols (C_8 - C_{18} carbon atoms) such as those produced by reducing tallow or coconut oil or their component fatty acids or methyl esters, and the sodium and potassium alkyl benzene sulfonates in which the alkyl group contains from about 9 to about 15 carbon atoms, in straight chain or branched chain configuration, e.g., those of the type described in United States Patents 2,220,099 and 2,477,383. Especially valuable are linear straight chain alkyl benzene sulfonates in which the average number of carbon atoms in the alkyl group is from about 11 to 13, abbreviated as C_{11-13} LAS.

Other anionic surfactants herein are the sodium alkyl

glyceryl ether sulfonates, especially those ethers of higher alcohols derived from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfonates and sulfates; sodium or potassium salts of alkyl phenol ethylene oxide ether sulfates
5 containing from about 1 to about 10 units of ethylene oxide per molecule and wherein the alkyl groups contain from about 8 to about 12 carbon atoms; and sodium or potassium salts of alkyl ethylene oxide ether sulfates containing about 1 to about 10 units of ethylene oxide per molecule and wherein the alkyl group
10 contains from about 10 to about 20 carbon atoms.

Other useful anionic surfactants herein include the water-soluble salts of esters of alpha-sulfonated fatty acids containing from about 6 to 20 carbon atoms in the fatty acid group and from about 1 to 10 carbon atoms in the ester group;
15 water-soluble salts of 2-acyloxy-alkane-1-sulfonic acids containing from about 2 to 9 carbon atoms in the acyl group and from about 9 to about 23 carbon atoms in the alkane moiety; water-soluble salts of paraffin or olefin sulfonates containing from about 12 to 24 carbon atoms; and beta-alkyloxy alkane sulfonates containing
20 from about 1 to 3 carbon atoms in the alkyl group and from about 8 to 20 carbon atoms in the alkane moiety.

Water-soluble salts of the higher fatty acids, i.e., "soaps", are useful anionic surfactants in the compositions herein. This includes alkali metal soaps of fatty acids containing from about 8
25 to about 24 carbon atoms, and preferably from about 12 to about 18 carbon atoms. Soaps can be made by direct saponification of fats and oils or by the neutralization of free fatty acids. Particularly useful are the sodium and potassium salts of the mixtures of fatty acids derived from coconut oil and tallow, i.e.,
30 sodium or potassium tallow and coconut soap.

Water-soluble nonionic surfactants, other than those which contain at least about 50% ethylene oxide by weight and also have a melting point of 35°C or greater, are suitable in the compositions of the invention, although not beneficial to physical
35 properties as the specified water-soluble polymeric materials

containing at least about 50% ethylene oxide by weight and having a melting point at least about 35°C. Such nonionic materials include compounds produced by the condensation of alkylene oxide groups with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. The length of the polyoxyalkylene group which is condensed with any particular hydrophobic group can be readily adjusted to yield a water-soluble compound having the desired degree of balance between hydrophilic and hydrophobic elements (HLB). Preferred nonionic surfactants of this type have HLB values of from about 9 to about 14.

Suitable nonionic surfactants include the polyethylene oxide condensates of alkyl phenols, e.g., the condensation products of alkyl phenols having an alkyl group containing from about 6 to 15 carbon atoms, in either a straight chain or branched chain configuration, with from about 3 to 12 moles of ethylene oxide per mole of alkyl phenol.

Preferred nonionics are the water-soluble condensation products of aliphatic alcohols containing from 8 to 22 carbon atoms, in either straight chain or branched configuration, with from 3 to 12 moles of ethylene oxide per mole of alcohol. Particularly preferred are the condensation products of alcohols having an alkyl group containing from about 9 to 15 carbon atoms with from about 4 to 8 moles of ethylene oxide per mole of alcohol.

Semi-polar nonionic surfactants include water-soluble amine oxides containing one alkyl moiety of from about 10 to 18 carbon atoms and 2 moieties selected from the group consisting of alkyl groups and hydroxyalkyl groups containing from 1 to about 3 carbon atoms; water-soluble phosphine oxides containing one alkyl moiety of about 10 to 18 carbon atoms and 2 moieties selected from the group consisting of alkyl groups and hydroxyalkyl groups containing from about 1 to 3 carbon atoms; and water-soluble sulfoxides containing one alkyl moiety of from about 10 to 18 carbon atoms and a moiety selected from the group consisting of

alkyl and hydroxyalkyl moieties of from about 1 to 3 carbon atoms.

Ampholytic surfactants include derivatives of aliphatic or aliphatic derivatives of heterocyclic secondary and tertiary amines in which the aliphatic moiety can be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to 18 carbon atoms and at least one aliphatic substituent contains an anionic water-solubilizing group.

Zwitterionic surfactants include derivatives of aliphatic quaternary ammonium, phosphonium, and sulfonium compounds in which one of the aliphatic substituents contains from about 8 to 18 carbon atoms.

Particularly preferred surfactants herein include linear alkylbenzene sulfonates containing from about 11 to 14 carbon atoms in the alkyl group; C_{12-18} alkyl sulfates, coconutalkyl glyceryl ether sulfonates; alkyl ether sulfates wherein the alkyl moiety contains from about 14 to 18 carbon atoms and wherein the average degree of ethoxylation is from about 1 to 4; olefin or paraffin sulfonates containing from about 14 to 16 carbon atoms; alkyldimethylammonio hydroxy propane sulfonates wherein the alkyl group contains from about 14 to 18 carbon atoms; soaps of higher fatty acids containing from about 12 to 18 carbon atoms; condensation products of C_9-C_{15} alcohols with from about 4 to 8 moles of ethylene oxide, and mixtures thereof.

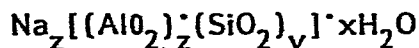
Specific preferred surfactants for use herein include: sodium linear C_{11-13} alkylbenzene sulfonate; triethanolamine C_{11-13} alkylbenzene sulfonate; sodium C_{12-18} alkyl sulfate; sodium coconut alkyl glyceryl ether sulfonate; the sodium salt of a sulfated condensation product of a tallow alcohol with about 4 moles of ethylene oxide; the condensation product of a coconut fatty alcohol with about 6 moles of ethylene oxide; the condensation product of tallow fatty alcohol with about 11 moles of ethylene oxide; 3-(N,N-dimethyl-N-coconutalkylammonio)-2--hydroxypropane-1-sulfonate; 3-(N,N-dimethyl-N-coconutalkylammonio)-propane-1-sulfonate; 6-(N-dodecylbenzyl-N,N-dimethylammonio)

hexanoate; dodecyl dimethyl amine oxide; C₁₂₋₁₅ alkyldimethyl amine oxide; and the sodium and potassium salts of coconut and tallow fatty acids.

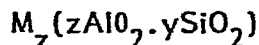
Non-Phosphorus-Containing Detergency Builder

5 The detergent compositions herein also contain from about 10% to about 70%, preferably from about 15% to about 50% by weight of a non-phosphorus-containing detergency builder selected from the group consisting of sodium aluminosilicate, sodium carbonate, polycarboxylic acids and water-soluble salts
10 thereof, and mixtures thereof.

An especially preferred detergency builder is crystalline aluminosilicate ion exchange material of the formula



wherein z and y are at least about 6, the molar ratio of z to y is
15 from about 1.0 to about 0.5 and x is from about 10 to about 264. Amorphous hydrated aluminosilicate materials useful herein have the empirical formula



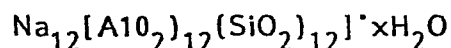
wherein M is sodium, z is from about 0.5 to about 2 and y is 1.

20 The aluminosilicate ion exchange builder materials herein are in hydrated form and contain from about 10% to about 28% of water by weight if crystalline, and potentially even higher amounts of water if amorphous. Highly preferred crystalline aluminosilicate ion exchange materials contain from about 18% to
25 about 22% water in their crystal matrix. The crystalline aluminosilicate ion exchange materials are further characterized by a particle size diameter of from about 0.1 micron to about 10 microns. Amorphous materials are often smaller, e.g., down to less than about 0.01 micron. Preferred ion exchange materials
30 have a particle size diameter of from about 0.2 micron to about 4 microns. The term "particle size diameter" herein represents the average particle size diameter of a given ion exchange material as determined by conventional analytical techniques such as, for example, microscopic determination utilizing a scanning electron
35 microscope. The crystalline aluminosilicate ion exchange materials

herein are usually further characterized by their calcium ion exchange capacity, which is at least about 200 mg. equivalent of CaCO_3 water hardness/g. of aluminosilicate, calculated on an anhydrous basis, and which generally is in the range of from about 300 mg. eq./g. to about 352 mg. eq./g. The aluminosilicate ion exchange materials herein are still further characterized by their calcium ion exchange rate which is at least about 2 grains Ca^{++} /gallon/minute/gram/gallon of aluminosilicate (anhydrous basis), and generally lies within the range of from about 2 grains/gallon/minute/gram/gallon to about 6 grains/gallon/minute/gram/gallon, based on calcium ion hardness. Optimum aluminosilicate for builder purposes exhibit a calcium ion exchange rate of at least about 4 grains/gallon/minute/gram/gallon.

The amorphous aluminosilicate ion exchange materials usually have a Mg^{++} exchange capacity of at least about 50 mg. eq. CaCO_3 /g. (12 mg. Mg^{++} /g.) and a Mg^{++} exchange rate of at least about 1 grain/gallon/minute/gram/gallon. Amorphous materials do not exhibit an observable diffraction pattern when examined by Cu radiation (1.54 Angstrom Units).

Aluminosilicate ion exchange materials useful in the practice of this invention are commercially available. The aluminosilicates useful in this invention can be crystalline or amorphous in structure and can be naturally-occurring aluminosilicates or synthetically derived. A method for producing aluminosilicate ion exchange materials is discussed in U.S. Patent 3,985,669, Krummel et al, issued October 12, 1976, incorporated herein by reference. Preferred synthetic crystalline aluminosilicate ion exchange materials useful herein are available under the designations Zeolite A, Zeolite B, and Zeolite X. In an especially preferred embodiment, the crystalline aluminosilicate ion exchange material in Zeolite A and has the formula



wherein x is from about 20 to about 30, especially about 27.

Sodium carbonate is a useful detergency builder within the scope of the invention because of its alkalinity in solution and its

ability to remove calcium and magnesium ions from washing solutions by precipitation. As disclosed hereinafter, sodium carbonate can also be added to the compositions of the invention in particulate dry form. The components of the compositions of the invention, however, are dried from a water slurry of the components.

Polycarboxylates in acid or soluble salt form are useful detergency builders within the scope of the invention. As used herein polycarboxylates includes polyacetates. Examples of polycarboxylates are the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylene diamine tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric acid.

One class of preferred polycarboxylate detergency builder herein is set forth in U.S. Patent No. 3,308,067, Diehl, issued March 7, 1967 incorporated herein by reference. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylenemalononic acid.

Other useful detergency builders are sodium and potassium carboxymethyloxymalonate, carboxymethyloxysuccinate, cis-cyclohexanehexacarboxylate, cis-cyclopentanetetra-carboxylate, and the copolymers of maleic anhydride with vinyl methyl ether or ethylene.

Other suitable polycarboxylates for use herein are the polyacetal carboxylates described in U.S. Patent 4,144,226, issued March 13, 1979 to Crutchfield et al, and U.S. Patent 4,246,495, issued March 27, 1979 to Crutchfield et al, both incorporated herein by reference. These polyacetal carboxylates can be prepared by bringing together under polymerization conditions an ester of glyoxylic acid and a polymerization initiator. The resulting polyacetal carboxylate ester is then attached to chemically stable end groups to stabilize the polyacetal carboxylate against rapid depolymerization in alkaline solution and

preferably converted to a salt.

Preferred compositions contain from about 10% to about 40% sodium aluminosilicate, from 0% to about 20% sodium carbonate and from 0% to about 20% polycarboxylate.

5 Polymeric Material Containing Ethylene Oxide

10 The compositions of the invention contain from about 0.3% to about 6.0%, preferably from about 0.5% to about 4.0%, and more preferably from about 0.7% to about 3.0% by weight of a water-soluble polymeric material, or mixtures thereof, containing at least about 50% ethylene oxide by weight, said polymer or mixtures thereof having a melting point not less than about 35°C. Preferably the polymeric material will have a melting point not less than about 45°C, more preferably not less than about 50°C and most preferably not less than about 55°C. Because the
15 polymeric materials useful in the practice of the invention are generally mixtures representing a range of molecular weights, the materials tend to soften and begin to become liquid over a range of temperatures of from about 3°C to about 7°C above their complete melting point. Mixtures of two or more polymric
20 materials can have an even wider range.

 Preferred polymers contain at least about 70% ethylene oxide by weight and more preferred polymers contain at least about 80% ethylene oxide by weight. Preferred polymeric materials have HLB values of at least about 15, and more preferably at least
25 about 17. Polyethylene glycol which can be said to contain essentially 100% ethylene oxide by weight is particularly preferred.

 Preferred polyethylene glycols have an average molecular weight at least about 1000, and more preferably from about 2500
30 to about 20,000 and most preferably from about 3000 to about 10,000.

 Other suitable polymeric materials are the condensation products of C₁₀₋₂₀ alcohols or C₈₋₁₈ alkyl phenols with sufficient ethylene oxide not less than 50% by weight of the polymer that
35 the resultant product has a melting point not below about 35°C.

Block and heteric polymers based on ethylene oxide and propylene oxide addition to a low molecular weight organic compound containing one or more active hydrogen atoms are suitable in the practice of the invention. Polymers based on the addition of ethylene oxide and propylene oxide to propylene glycol, ethylenediamine, and trimethylolpropane are commercially available under the names Pluronic®, Pluronic® R, Tetronics® and Pluradots® from the BASF Wyandotte Corporation of Wyandotte, Michigan. Corresponding nonproprietary names of the first three trade names are poloxamer, meroxapol and poloxamine, respectively.

Optional Ingredients

As described hereinbefore water-soluble silicate materials such as alkali metal silicates can be incorporated in the compositions of the invention at levels not greater than about 4.5% by weight. Preferably, silicate content is not greater than about 3.5% by weight. In a particularly preferred embodiment the silicate material is sodium silicate and comprises from about 0.5% to about 3% of the composition by weight. Preferred silicate materials have a weight ratio of SiO_2 to alkali metal oxide such as Na_2O of from about 0.5 to about 4.0, and preferably from about 1.0 to about 2.4.

Phosphorus-containing detergency builders such as phosphates and polyphosphonates can be used in the compositions of the inventions, but preferably at levels not greater than about 15%, and more preferably, at levels not greater than about 5% by weight. Most preferably, the compositions are substantially free of phosphates.

Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from about 6 to 21, and orthophosphate. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene diphosphonic acid, the sodium and potassium salts of ethane 1-hydroxy-1,1-diphosphonic acid and the sodium and potassium salts of ethane, 1,1,2-

triposphonic acid. Other phosphorus builder compounds are disclosed in U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148, incorporated herein by reference.

5 Other ingredients commonly used in detergent compositions can be included in the compositions of the present invention. These include bleach activators, suds boosters or suds suppressors, anti-tarnish and anti-corrosion agents, soil suspending agents, soil release agents, dyes, fillers, optical brighteners, 10 germicides, pH adjusting agents, non-builder alkalinity sources and hydrotropes.

Materials that are not heat stable or stable in aqueous slurries can be added to compositions of the invention after drying of the slurry and formation of granules. Examples are bleaching 15 agents, such as sodium perborate, enzymes and perfumes. Silicate material additional to the limited quantity in the dried granule may also be added to the compositions of the invention at this point without serious harmful effect, but are not to be 20 considered as components of the compositions of the invention unless dried from a common slurry.

The following non-limiting examples illustrate the detergent compositions of the present invention. The numerical values within parentheses are on a 100 parts/percentage basis and are the determinants of the scope of the invention for compositions 25 prepared by drying an aqueous slurry of the components of said composition.

All percentages, parts, and ratios used herein are by weight unless otherwise specified.

The granular detergent compositions of the examples were 30 evaluated using the indicated tests.

Compression Test

The granules are poured into a cylinder and compressed by applying a 10 pound weight for 4 minutes. The difference in height is measured and computed into percent compression. 35 Lower numbers are better. Numbers of less than about 30 are acceptable.

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Cake Strength Test

5 The compressed, unsupported cylinder of granules created by the compression test is fractured by applying a weight to the top until the cylinder fractures. The weight in pounds required to fracture the cylinder is the cake grade. In general, lower grades are more acceptable.

Solubility Grade

10 The detergent composition is dissolved in water and filtered with suction through a black fabric and graded against photographic standards. Grades of 7 to 10 are acceptable.

Pour/Lump Grade

15 The resistance to carton caking was measured after storage. Cartons are opened from the top according to the package instruction. The contents are poured out by tilting the cartons at a 45° angle, upside down, and at a height 10-12 inches above the receiving tray, using the necessary shaking and tapping needed to empty the cartons. A pour grade of 0-10 is given according to the ease with which the products flowed from the cartons. A lump grade of 0-10 is also given according to the
20 amount of lumps/cakes appearing on the tray. This is graded against photographic standards. The pour grade and the lump grade are averaged to form the pour/lump grade, with 10 being the best grade and 0 being the worst.

25

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EXAMPLE ICompositions of The Invention

	<u>Components</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
	<u>Mixed in 30-40% Water</u>				
5	<u>Slurry and Spray Dried</u>				
	Sodium C ₁₂ LAS	16.10(20.1)	16.10(20.1)	16.10(20.1)	16.10(20.1)
	Sodium C ₁₄₋₁₅ alkyl sulfate	6.90(8.6)	6.90(8.6)	6.90(8.6)	6.90 (8.6)
	Sodium tallow				
10	soap	0.75 (0.93)	0.75 (0.93)	0.75 (0.93)	0.75 (0.93)
	Polyethylene glycol (avg. M.W. 8000)	1.40 (1.75)	1.40 (1.75)	3.00 (3.75)	4.50 (5.6)
	Sodium silicate				
15	(2.0 ratio SiO ₂ .Na ₂ O)	2.00 (2.5)	2.00 (2.5)	2.00 (2.5)	2.00 (2.5)
	Sodium aluminosilicate				
	(Zeolite A)	15.00(18.75)	15.00(18.75)	15.00(18.75)	15.00(18.75)
20	Miscellaneous	0.42 (0.53)	0.42 (0.53)	0.42 (0.53)	0.42 (0.53)
	Water	4.40 (5.5)	6.80 (8.5)	4.40 (5.5)	4.60 (5.7)
	Sodium sulfate	-----Balance-----			
		80.02(100)	80.02(100)	80.02(100)	80.02(100)
25	<u>Components</u>				
	<u>Admixed after</u>				
	<u>Spray Drying</u>				
	Sodium Perborate				
	monohydrate	3.40	3.40	3.40	3.40
30	Sodium Carbonate	16.10	16.10	16.10	16.10
	Polyethylene glycol (avg. M.W. 8000)	--	--	--	--
	Miscellaneous	0.48	0.48	0.48	0.48
35		19.98	19.98	19.98	19.98
		100.00	100.00	100.00	100.00

<u>Comparative Compositions</u>				
<u>Components</u>	<u>E</u>	<u>F</u>	<u>G</u>	<u>H</u>
<u>Mixed in 30%-40% Water</u>				
<u>Slurry and Spray Dried</u>				
5 Sodium C ₁₂ LAS	16.10(19.0)	16.10(19.5)	16.10(19.5)	16.10(20.1)
Sodium C ₁₄₋₁₅				
alkyl sulfate	6.90(8.1)	6.90(8.4)	6.90(8.4)	6.90(8.6)
Sodium tallow				
soap	0.75 (0.89)	0.75 (0.91)	0.75 (0.91)	0.75 (0.93)
10 Polyethylene				
glycol (avg.				
M.W. 8000)	-- (---)	1.40 (1.70)	3.00 (3.64)	-- (---)
Sodium silicate				
(2.0 ratio				
15 SiO ₂ .Na ₂ O)	12.00(14.2)	5.00 (6.1)	5.00 (6.1)	2.00 (2.5)
Sodium aluminosilicate				
(Zeolite A)	15.00(17.71)	15.00(18.18)	15.00(18.18)	15.00(18.75)
Miscellaneous	0.42 (0.50)	0.42 (0.51)	0.42 (0.51)	0.42(0.53)
20 Water	3.90 (4.60)	4.40 (5.3)	4.40 (5.3)	4.80(6.0)
Sodium sulfate	-----Balance-----			
	84.72(100)	82.52(100)	82.52(100)	80.02(100)
<u>Components</u>				
25 <u>Admixed after</u>				
<u>Spray Drying</u>				
Sodium Perborate				
monohydrate	3.40	3.40	3.40	3.40
Sodium Carbonate	10.00	13.60	13.60	16.10
30 Polyethylene				
glycol (avg.				
M.W. 8000)	1.40	--	--	--
Miscellaneous	0.48	0.48	0.48	0.48
35	15.28	17.48	17.48	19.98
	100.00	100.00	100.00	100.00

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	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>	<u>G</u>	<u>H</u>
Physical properties after 2 weeks of aging under 90°F, 80% RH condition								
5 Pour/lump grade (10 max)	10.0	8.0	10.0	9.5	5.0	10.0	9.5	--
10 Cake strength (lbs.)	3.0	3.9	1.6	5.8	10.0	2.8	0.4	(10.3)*
Compression (%)	16.4	16.4	8.8	16.4	31.5	11.3	8.8	(31.5)*
Moisture absorbed (%)	0**	0**	0.4	1.4	5.5	1.0	2.1	--
15 Solubility Grade (10 max)	7.5	8.0	7.0	8.5	4.0	5.0	6.0	8.5
* These are the initial properties of the product which was not storage tested due to unacceptable compression and high cake strength.								
20 ** Not detected.								
Compositions of the invention A, B, C and D exhibit satisfactory solubility, physical properties and low moisture absorption relative to compositions outside of the scope of the invention which are deficient in one or more respects.								
25 The polyethylene glycol 8000 (PEG 8000) of Examples A through D is replaced by an equal weight of the condensation product of 1 mole of a C ₁₈ alcohol with 150 moles of ethylene oxide per mole of alcohol. Products with satisfactory solubility, physical properties and low moisture absorption are obtained.								
30								

EXAMPLE II

		High Silicate				
	<u>Components</u>	<u>Comparison</u>	<u>A</u>	<u>B</u>	<u>C</u>	
5	<u>Mixed in 30%-40% Water</u>					
	<u>Slurry and Spray-Dried</u>					
	Sodium C ₁₃ LAS	10.00(11.8)	10.00(12.3)	10.00(12.5)	10.00(12.5)	
	Sodium C ₁₄₋₁₅					
	alkyl sulfate	10.00(11.8)	10.00(12.3)	10.00(12.5)	10.00(12.5)	
10	Tallow soap	0.75 (0.88)	0.75 (0.93)	0.75 (0.93)	0.75 (0.93)	
	PEG 8000)	-- (--)	0.80 (.99)	1.40 (1.75)	3.00 (3.75)	
	Sodium silicate					
	(2.0r)	12.00(14.2)	2.00 (2.5)	2.00 (2.5)	2.00 (2.5)	
	Sodium aluminosilicate					
15	(Zeolite A)	15.00(17.7)	15.00(18.5)	15.00(18.7)	15.00(18.7)	
	Miscellaneous	0.42 (0.50)	0.42 (0.52)	0.42 (0.52)	0.42 (0.52)	
	H ₂ O	5.40 (6.4)	4.60 (5.7)	4.80 (6.0)	3.40 (4.2)	
	Sodium sulfate	-----Balance-----				
20		84.72(100)	81.12(100)	80.02(100)	80.02(100)	
	<u>Components Admixed</u>					
	<u>After Spray Drying</u>					
	Sodium perborate					
	monohydrate	3.40	3.40	3.40	3.40	
25	Sodium carbonate	10.00	15.00	16.10	16.10	
	PEG 8000	1.40	--	--	--	
	Miscellaneous	0.48	0.48	0.48	0.48	
		15.28	18.88	19.98	19.98	
		100.00	100.00	100.00	100.00	

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	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
Physical properties after 2 weeks of aging <u>under 90°F, 80% RH condition</u>				
5 Pour/lump grade				
(10 max)	5.5	9.0	9.0	9.5
Cake strength				
(lbs)	7.0	7.6	3.4	3.0
Compression (%)	26.5	18.9	16.4	6.3
10 Moisture				
absorbed (%)	3.0	0	0.6	1.8
<u>Solubility Grade (10 max)</u>				
Initial	5.0	8.5	8.0	7.0

15 Compositions B, C and D contain preferred levels of polyethylene glycol within the scope of the invention and exhibit superior solubility and physical properties.

EXAMPLE III

	<u>High Silicate</u>		<u>Composition of</u>	
	<u>Comparison</u>		<u>Invention</u>	
20 <u>Components</u>				
Mixed in 30%-40% Water				
<u>Slurry and Spray-Dried</u>				
Sodium NaC ₁₃ LAS	18.00	(20.9)	18.00	(20.9)
Sodium C ₁₄₋₁₅ alkyl sulfate	2.00	(2.3)	2.00	(2.3)
25 Tallow soap	1.15	(1.3)	1.15	(1.3)
Pluronic® F108*	-	(--)	2.00	(2.3)
Sodium silicate (2.0R)	12.00	(13.9)	2.00	(2.3)
Sodium aluminosilicate				
(Zeolite A)	15.00	(17.4)	15.00	(17.4)
30 Miscellaneous	0.42	(0.49)	0.42	(0.49)
Water	4.40	(5.1)	3.40	(4.0)
Sodium sulfate	-----Balance-----			
	86.12(100)		86.12(100)	

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Components AdmixedAfter Spray Drying

Sodium perborate mono-			
	hydrate	3.40	3.40
5	Sodium carbonate	10.00	10.00
	Miscellaneous	0.48	0.48
		13.88	13.88
		100.00	100.00

10

Physical Properties after

One-Week Aging Under 90°F,

80% R.H. Condition

	Pour/lump grade (10 max.)	4.0	9.5
15	Cake strength (lbs)	11.0	3.2
	Compression (%)	21.4	8.8
	Moisture Absorbed (%)	5.0	1.0
	<u>Solubility Grade (10 max.)</u>	4.5	7.5

20

* Pluronic F108 has an average molecular weight of about 14,000 and contains 80% polyoxyethylene (ethylene oxide) by weight. It is a block copolymer of ethylene oxide and propylene oxide.

CLAIMS

1. A granular detergent composition comprising:
 - 5 (a) from about 5% to about 50% by weight of a detergent surfactant selected from the group consisting of anionic, nonionic, zwitterionic, ampholytic and cationic surfactants and mixtures thereof, provided that at least about 50% of the total surfactant by weight is a non-soap anionic surfactant;
 - 10 (b) from about 10% to about 70% by weight of a non-phosphorus-containing detergency builder selected from the group consisting of sodium aluminosilicate, sodium carbonate, polycarboxylic acids and water-soluble salts thereof, and mixtures thereof;
 - (c) from 0% to about 4.5% of a water-soluble silicate material;
 - 15 (d) from about 0.3% to about 6.0% by weight of a water-soluble polymeric material containing at least about 50% ethylene oxide by weight, said polymer having a melting point not less than about 35°C, said composition prepared by drying an aqueous slurry of
20 the components of said composition.
2. The composition of Claim 1 wherein the slurry is spray-dried.
3. The composition of Claim 2 wherein the water-soluble polymeric material is a polyethylene glycol.
4. The composition of Claim 3 wherein the non-soap anionic surfactant comprises at least about 75% of the total surfactant by weight.
5. The composition of Claim 4 wherein the non-soap anionic surfactant is selected from the group consisting of the water-soluble salts of alkyl benzene sulfonates, alkyl sulfates, alkyl ethoxy ether sulfates and mixtures thereof.

6. The composition of Claim 3 containing from about 0.5% to about 4.0% by weight of a polyethylene glycol with a melting point not less than about 45°C.

7. The composition of Claim 3 containing from about 0.7% to about 3.0% by weight of a polyethylene glycol with a melting point not less than about 50°C.

8. The composition of Claim 2 containing not more than about 3.5% by weight of a water-soluble silicate material.

9. The composition of Claim 2 wherein the water soluble silicate material is sodium silicate with a weight ratio of $\text{SiO}_2 \cdot \text{Na}_2\text{O}$ of from about 1.0 to about 2.4.

10. The composition of Claim 9 containing from about 0.5% to about 3% sodium silicate by weight.

11. The composition of Claim 1 comprising from about 15% to about 50% by weight of a non-phosphorus-containing detergency builder.

12. The composition of Claim 11 comprising from about 10% to about 40% sodium aluminosilicate by weight.

EFG:sp(A45/A10)

