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(54) **Spray-dried granular detergent compositions and making thereof.**

(57) Detergent compositions comprising a mixture of a nonionic surfactant, a polyethylene glycol and polyacrylate of specified molecular weight for improved physical properties and cold water dispersion are disclosed.

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Croydon Printing Company Ltd.

SPRAY-DRIED GRANULAR DETERGENT COMPOSITIONS AND MAKING THEREOF

Mark Edward Cushman

Technical Field

The present invention relates to spray-dried, granular detergent compositions.

Summary of the Invention

The present invention encompasses a spray-dried granular detergent composition comprising:

(a) from about 5% to about 50% by weight of a nonsoap anionic detergent surfactant; and

(b) from about 1% to about 10% of a mixture of nonionic surfactant, comprising an ethoxylated alcohol or alkyl phenol, polyethylene glycol and a polyacrylate, said nonionic surfactant comprising from about 10% to about 80% of the mixture, said polyethylene glycol having a weight average molecular weight of from about 1,000 to about 20,000, and said polyacrylate having a weight average molecular weight of from about 1,000 to about 20,000 said mixture having a polyethylene glycol:polyacrylate weight ratio of from about 1:10 to about 10:1, and wherein such mixture is added in the crutcher.

Detailed Description of the Invention

The detergent compositions of the present invention contain a nonsoap anionic detergent surfactant, and a mixture of a nonionic surfactant, a polyethylene glycol of selected molecular weight and a polyacrylate polymer of selected molecular weight. The nonionic surfactant/polyethylene glycol/polyacrylate mixtures herein provide a surprising boost to dispersion rates and improvement in physical properties.

The compositions of the present invention are prepared by spray drying and have superior physical characteristics.

Anionic Surfactant

The detergent compositions herein contain from about 5% to about 50%, preferably from about 10% to about 30% of a nonsoap anionic surfactant, or mixtures thereof. 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 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 9 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 the glycerides of tallow or coconut oil; 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 U.S. Patents 2,220,099 and 2,477,383 both of which are incorporated herein by reference. 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 suitable for use 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 containing from about 1 to about 10 units of ethylene oxide per molecule and from about 8 to about 12 carbon atoms in the alkyl group; and sodium or potassium salts of alkyl ethylene oxide ether sulfates containing from about 1 to about 10 units of ethylene oxide per molecule and from about 10 to about 20 carbon atoms in the alkyl group.

Other useful anionic surfactants 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; 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; alkyl ether sulfates containing from about 10 to 20 carbon atoms in the alkyl group and from about 1 to 30 moles of ethylene oxide; water-soluble salts of olefin or paraffin sulfonates containing from about 12 to 18 carbon atoms; and beta-alkyloxy alkane sulfonates containing from about 1 to about 3 carbon atoms in the alkyloxy group and from about 8 to about 20 carbon atoms in the alkane moiety.

Particularly preferred surfactants for use herein include sodium C_{11-13}LAS, C_{14-18} alkyl sulfates, C_{14-18} alkyl linear polyethoxy sulfates containing from about 1 to about 4 moles of ethylene oxide, and mixtures thereof.

Nonionic Surfactant/Polyethylene Glycol/Polyacrylate Mixture

The compositions of the present invention contain from about 1% to about 10%, preferably from about 1% to about 5%, more preferably from about 2% to about 4%, of a mixture of a polyethoxylate nonionic surfactant, a polyethylene glycol and a polyacrylate.

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 about 15, preferably from about 9 to about 13, carbon atoms, in either a straight chain or branched chain configuration, with from about 3 to about 12, preferably from about 4 to about 8, moles of ethylene oxide per mole of alkyl phenol.

Preferred nonionics are the water-soluble condensation products of aliphatic alcohols containing from about 8 to about 20, preferably from about 10 to about 18, carbon atoms, in either straight chain or branched configuration, with from about 3 to about 12, preferably from about 4 to about 8, moles of ethylene oxide per mole of alcohol. Particularly preferred are the condensation products of alcohols having an alkyl group containing from about 10 to about 16 carbon atoms with from about 4 to about 8 moles of ethylene oxide per mole of alcohol.

The nonionic surfactant comprises by weight from about 10% to about 80%, preferably from about 25% to about 70%, more preferably from about 35% to about 60% of the mixture.

The polyethylene glycol has a weight average molecular weight of from about 1,000 to about 20,000, preferably from about 2,000 to about 12,000, more preferably from about 4,000 to about 10,000. The polyacrylate has a weight average molecular weight of from about 1,000 to about 20,000, preferably from about 2,000 to about 10,000, more preferably from about 3,000 to about 8,000.

The polyethylene glycol and the polyacrylate are present in a weight ratio of from about 1:10 to about 10:1, preferably from about 1:3 to about 3:1, more preferably from about 2:1 to about 1:2.

While polyethylene glycols are preferred, other suitable polymeric materials are the condensation products of C_{10-20} alcohols or C_{8-18} alkyl phenols with sufficient ethylene oxide, i.e., more than 50% by weight of the polymer, so that the resultant product has a melting point above about 35°C.

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 about 17. 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 Pluronics®, 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.

Optimum solubility of the polyacrylate is obtained when it is in the form of an at least partially neutralized alkali metal salts. The sodium salts are most preferred.

Suitable polyacrylates herein are the partially or fully neutralized salts of polymers of acrylic acid. One can also use copolymers formed with small amounts of other copolymerizable monomers. The percentage by weight of the polyacrylate units which is derived from acrylic acid is preferably greater than about 80%. Suitable copolymerizable monomers include, for example, methacrylic acid, hydroxyacrylic acid, vinyl chloride, vinyl alcohol, furan, acrylonitrile, methacrylonitrile, vinyl acetate, methyl acrylate, methyl methacrylate, styrene, alpha-methylstyrene, vinyl methyl ether, vinyl ethyl ether, vinyl propyl ether, acrylamide, ethylene, propylene and 3-butenoic acid. Mixtures of these polymers can also be used. The polyacrylate may also be added in the acid form and neutralized by various bases present.

Preferred copolymers of the above group contain at least about 90% by weight of units derived from the acrylic acid. Preferably essentially all of the polymer is derived from acrylic acid. Particularly preferred is sodium polyacrylate, especially when it has an average molecular weight of from about 3,000 to about 8,000.

It is essential that this mixture be added in the crutcher rather than post dosed for the benefits of the invention to be seen.

Optional Ingredients

The compositions of the invention can additionally contain up to 10%, preferably about 5% of an organic surfactant selected from the group consisting of zwitterionic, ampholytic, and cationic surfactants and mixtures thereof. The compositions can also contain other conventional ingredients, such as nonphosphorous builders, either organic or inorganic in nature.

Optional 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 about 18 carbon atoms and at least one aliphatic substituent contains an anionic water-solubilizing group.

Useful cationic surfactants 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.

Optional zwitterionic surfactants include derivatives of aliphatic quaternary ammonium or phosphonium or ternary sulfonium compounds in which one of the aliphatic substituents contains from about 8 to about 18 carbon atoms.

Also useful in the compositions of the invention are alkylpolysaccharide surfactants. The preferred alkylpolyglycosides have the formula $RO(C_nH_{2n}O)_t(glycosyl)_x$ wherein R is selected from the group consisting of alkyl, alkylphenyl, hydroxyalkyl, hydroxyalkylphenyl and mixtures thereof, in which said alkyl groups contain from about 10 to about 18, preferably from about 12 to about 14 carbon atoms, n is 2 or 3, preferably 2, t is from 0 to about 10, preferably 0, and x is from about 1-1/4 to about 10, preferably from about 1-1/3 to about 3, most preferably from about 1-1/3 to about 2. The glycosyl is preferably derived from glucose.

Detergency builders are preferred optional ingredients when included, the level of detergency builder is from 0% to about 60%, preferably from about 10% to about 60%, more preferably from about 20% to about 60% of the composition.

Preferred detergency builders include the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, silicates and borates.

Especially preferred for use in compositions of the invention are the inorganic phosphate builders.

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-1, 1-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-triphosphonic acid. Other phosphorus builder compounds are disclosed in U.S. Pat. Nos. 3,159,581, 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148 (all incorporated herein by reference). Sodium tripolyphosphate and pyrophosphate are particularly preferred.

The detergent compositions of the invention can also optionally contain water-insoluble aluminosilicate ion exchange material of the formula

$$Na_z[(AlO_2)_z \cdot (SiO_2)_y] \cdot xH_2O$$

wherein z and y are at least about 6, the molar ratio of z to y is 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

$$M_z(zAlO_2 \cdot ySiO_2)$$

wherein M is sodium, potassium, ammonium or substituted ammonium, z is from about 0.5 to about 2 and y is 1, said material having a magnesium ion exchange capacity of at least about 50 milligram equivalents of $CaCO_3$ hardness per gram of anhydrous aluminosilicate.

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 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 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 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. $CaCO_2$/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 has the formula

$$Na_{12}[AlO_2)_{12}(SiO_2)_{12}]\cdot xH_2O$$

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

Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium, carboxylates, nonpolymeric polycarboxylates and polyhydroxysulfonates. Examples of nonpolymeric polycarboxylate builders are the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric acid. The compositions of this invention only contain the limited amount of polyacrylate defined hereinafter.

Other useful builders herein are sodium and potassium carboxymethyloxymalonate, carboxymethyloxysuccinate, cis-cyclohexanehexacarboxylate, cis-cyclopentanetetracarboxylate, and phloroglucinol trisulfonate.

Other suitable nonpolymeric polycarboxylates 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 converted to the corresponding.

The compositions herein preferably contain from about 0% to about 10%, preferably from about 0.5% to about 8%, and most preferably from about 1% to about 7%, by weight of an alkali metal silicate having a molar ratio of SiO_2 to alkali metal oxide of from about 1.0 to about 3.2, preferably from about 1.4 to about 2.4.

Sodium silicate, particularly one having a molar ratio of about 1.6 to about 2.2 is preferred.

The alkali metal silicates can be purchased in either liquid or granular form. Silicate slurries can conveniently be used to avoid having to dissolve the dried form in the crutcher mix of the components herein.

Other ingredients commonly used in detergent compositions can be included in the compositions of the present invention. These include color speckles, bleaching agents such as perborates and percarbonates and bleach activators, suds boosters or suds suppressors, antitarnish and anticorrosion agents, soil suspending agents, soil release agents, dyes, fillers, optical brighteners, germicides, pH adjusting agents, nonbuilder alkalinity sources, hydrotropes such as toluene sulfonates and xylene sulfonates, enzymes, enzyme-stabilizing agents, perfumes and water.

The detergent compositions of the present invention can comprise a portion of compositions containing a wide variety of materials suitable for detergent or other uses.

The following nonlimiting examples illustrate the detergent compositions of the present invention.

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

COMPARATIVE EXAMPLE I

A base product was prepared by spray drying according to the following formula.

Na C_{13} linear alkyl sulfonate (LAS)	10.3
Na C_{14-15} alkyl sulfate (AS)	10.3
Na tripolyphosphate	43.3
Na silicate solids (1.6r)	6.9
Na sulfate	17.0
Na carbonate (dry mixed)	15.5
Diethylenetriamine pentacetate	0.7
Minor ingredients and water	balance

EXAMPLE II

The base product was produced according to Example I with varying ratios of polyethylene glycol (PEG) with a weight average molecular weight of 8,000 and sodium polyacrylate with weight average molecular weight of 4,500, and nonionic surfactant ($C_{12-13}E_{6.5}$ topped) as shown below added in the crutcher mix. Seventy-two grams (72 g.) of product were sewn into black fabric pockets and agitated on delicate agitation at 60°F (15.5°C) in a Kenmore washer. Pockets were removed and cut at 3 minutes, 5 minutes, 7 minutes, and 10 minutes and graded on a 1-7 scale where 1 is poorly dispersed with most of the product remaining caked in the pocket and 7 is completely dispersed. Grades reported are averages of two replicate tests.

				Solubility grade at Minutes			
Product	% PEG	% 4500 MW Poly-acrylate	% $C_{23}E_{6.5T}$ Nonionic	3	5	7	10
A	0	0	0	1	2	6.5	7
B	0	1.03	0	1	6	7	4
C	0.69	1.03	0	1	6	7	7
D	0.69	1.03	1.37	7	7	7	7

As can be seen, the products containing all three ingredients (PEG, polyacrylate, and nonionic surfactant) of the invention have the most improved cold water dispersion.

CLAIMS

1. A spray-dried granular detergent composition comprising:
   (a) from about 5% to about 50% of a non-soap anionic surfactant or mixtures thereof; and
   (b) from about 1% to about 10% of a mixture of nonionic surfactant, polyethylene glycol and a polyacrylate, said mixture being added in the crutcher, said nonionic surfactant comprising a polyethylene condensation product of an aliphatic alcohol containing from about 10 to about 20 carbon atoms or an alkyl phenol,

wherein the alkyl group contains from about 6 to about 15 carbon atoms, condensed with from about 3 to about 12 moles of ethylene oxide per mole of alcohol or alkyl phenol, said polyethylene glycol having a weight average molecular weight of from about 1000 to about 20,000, and said polyacrylate having a weight average molecular weight of from about 1,000 to about 20,000; the ratio of polyethylene glycol to polyacrylate being from about 10:1 to 1:1, and said nonionic surfactant comprising from about 10% to about 80% of the mixture.

2. The composition of Claim 1 wherein the mixture of nonionic surfactant, polyethylene glycol and polyacrylate comprises from about 1% to about 5% of the composition.

3. The composition of Claim 2 wherein the nonionic surfactant comprises from about 25% to about 70% of the mixture and the ratio of polyethylene glycol to polyacrylate is from about 3:1 to about 1:3.

4. The composition of Claim 3 wherein the polyethylene glycol has a weight average molecular weight of from about 2000 to about 12,000, and the polyacrylate has a weight average molecular weight of from about 2,000 to about 10,000.

5. A composition according to Claim 3 wherein the polyacrylate is sodium polyacrylate.

6. The composition of Claim 3 further comprising:
   (a) from about 20 to about 60% of a detergency builder; and
   (b) from about 1% to about 10% of an alkali metal silicate having a ratio of from about 1.6 to about 2.4.

7. The composition of Claim 1 comprising:
   (a) from about 10% to about 30% of non-soap anionic surfactant selected from the group consisting of alkali metal salts of C_{11-13} alkylbenzene sulfonates, C_{14-18} alkyl sulfates, C_{14-18} alkyl polyethoxysulfates containing from about 1 to about 4 moles of ethylene oxide and mixtures thereof;
   (b) from about 10% to about 60% of inorganic phosphate detergency builder selected from the group consisting of alkali metal phosphates and polyphosphates; and
   (c) from about 1% to about 5% of said mixture of nonionic surfactant, polyethylene glycol, and polyacrylate wherein said nonionic surfactant is an ethylene oxide condensate of an aliphatic alcohol containing from about 10 to about 18 carbon atoms or alkyl phenol, wherein the alkyl group contains from about 9 to about 13 carbon atoms, condensed with an average of from about 4 to about 8 moles of ethylene oxide per mole of alcohol or alkyl phenol, and

wherein said polyethylene glycol has a weight average molecular weight of from about 2,000 to about 12,000, said polyacrylate is sodium polyacrylate having a weight average molecular weight of from about 3,000 to about 8,000, the nonionic surfactant comprises from about 35% to about 60% of the mixture, the weight ratio of polyethylene glycol to polyacylate is from about 1:3 to about 3:1, and wherein said mixture is added in the crutcher.

8. The composition of Claim 7 further comprising:
   (a) at least about 20% of the detergency builder; and
   (b) from about 0.5% to about 8% of an alkali metal silicate having a ratio of from about 1.4 to about 2.4.

9. The spray-dried granular detergent composition of Claim 1 comprising:
   (a) from about 10% to about 30% of non-soap anionic surfactant comprising a mixture of alkali metal salts of C_{11-13} alkyl benzene sulfonates, and C_{14-18} alkyl sulfates wherein the ratio of the alkyl sulfonate to the alkyl sulfate is from about 3:1 to about 1:3,
   (b) from about 20% to about 60% of an alkali metal tripolyphosphate, pyrophosphate or mixtures thereof; and
   (c) from about 1% to about 5% of a mixture of:
      (1) nonionic surfactant comprising an ethylene oxide condensate of an aliphatic alcohol, wherein the alkyl group contains from about 10 to about 18 carbon atoms, and is condensed with from about 4 to about 8 moles of ethylene oxide per mole of alcohol;
      (2) a polyethylene glycol with a weight average molecular weight of from about 4,000 to about 10,000; and
      (3) a sodium polyacrylate with a weight average molecular weight of from about 3,000 to about 8,000,

   wherein the nonionic comprises from about 35% to about 60% of the mixture and the ratio by weight of polyethylene glycol to sodium polyacrylate is from about 1:3 to about 3:1.

10. A composition according to Claim 9 also comprising from about 0.5% to about 8% of sodium silicate solids having a molar ratio of SiO_2:Na_2O of from about 1.6 to about 2.2.

11. A composition according to Claim 9 wherein the mixture of anionic surfactants has about a 1:1 ratio on a weight basis.

12. A process for spray drying a granular detergent composition according to Claim 1 wherein the components are mixed in the crutcher along with enough additional water so that the water content of the crutcher paste is from about 25% to about 50% and then spray dried with an inlet air temperature of from about 400°F (204°C) to about 800°F (427°C).

13. The process of Claim 12 wherein the water content of the crutcher paste is from about 28% to about 40% and the inlet air temperature in the spray tower is from about 500°F (260°C) to about 700°F (371°C).