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[54] **METHOD FOR MAKING DETERGENT COMPOSITIONS**

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[58] Field of Search **252/99, 135, 539, 540**

[57] **ABSTRACT**

An improvement in a process of making detergent compositions in which water, organic detergents, hydrotrope, hydratable salts, inorganic salt filler and an oxygen-liberating percompound are mixed and oxygen is liberated to expand said paste wherein the hydratable salt is added prior to the addition of the filler.

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5 Claims, No Drawings

METHOD FOR MAKING DETERGENT COMPOSITIONS

This invention relates to an improvement in a process of making detergent compositions in the form of particles having low bulk density.

Prior to the present invention it was proposed to make a detergent composition comprising solid particles having an apparent density of less than 0.45 gram per cubic centimeter wherein the particles are in the form of irregularly shaped spongy coherent aggregates having a pock marked outer surface by mixing water, organic detergent, inorganic salt, hydrotrope and filler to form a slurry to which hydratable salt is then added, followed by the addition of an oxygen-liberating per-compound. In accordance with such proposals, this paste was thereafter immediately mixed very briefly and then permitted to stand quiescently at room temperature. During this period the composition expanded due to the evolution of oxygen. Subsequently the product was decomposed into granules which, if necessary, might be dried to improve the caking and flow characteristics of the composition.

In accordance with the present invention, a process for the preparation of a particulate detergent composition wherein water, organic detergent, hydrotrope, hydratable salt, inorganic salt filler and an aqueous solution of an oxygen-liberating per-compound are mixed to form a paste capable of retaining small oxygen bubbles without substantial coalescence thereof, liberating oxygen from said per-compound into said paste in an amount sufficient to bleach said paste and to expand said paste to a final volume at least two times the initial volume thereof, setting the expanded paste under quiescent conditions to a friable mass of substantially said final volume, and granulating said friable mass to form particles of a bleached detergent composition, the improvement comprising adding said hydratable salt prior to the addition of said filler whereby the hydratable salt is more effectively hydrated resulting in more uniform particles having less fines and having an apparent density of less than about 0.32 gram per cubic centimeter.

The addition of the hydratable salt before the filler in the above recited process results also in the consistent production of a finished product of improved texture, whose particles are chemically uniform throughout.

The particles of the detergent composition produced by the present invention are more readily soluble in water than those prepared by the prior method. Also, by employing the present invention, drying can be eliminated because the change in the order of addition improves the flowability of the feed composition which in turn makes possible the use of less moisture. By being able to use less moisture in the preparation, the final moisture content can be controlled to be 18.5% and there is no need to further dry the product since it has the desired flowing and non-caking characteristics at this moisture content. Aging time is also thereby reduced and less fines and dust are formed because of the elimination of the drying step. Furthermore, the hydratable salt is more effectively hydrated, apparently because some of it goes into solution and recrystallizes out as the hydrated salt.

It was discovered that to obtain the above desirable results, the critical point at which the hydratable salt should be added during the processing is after the water, detergent, hydrotrope and silicate have been added

and mixed for a short period. The filler, e.g., sodium sulfate, should be added after the hydratable salt, and finally the oxygen-liberating per-compound is added. It was found that, at the low moisture contents used, if the hydratable salt is added before the silicate is added, there would not be enough moisture present to hydrate the salt; whereas if the hydratable salt is added after the silicate, there is additional moisture for hydration provided because silicate is usually added in the form of an aqueous silicate solution.

The moisture content of the paste, the rate of hydration, and the degree of hydration of the hydratable salt should be carefully controlled during the preparation of the instant composition. There must be enough moisture present to sufficiently hydrate the salt at a suitable rate. This reaction liberates heat at a rate desirable for activating the liberation of O₂ from the expanding agent thereby controlling the foaming of the mixed mass produced by the process. The hydratable salt also serves to take up, as water of hydration, the water which is added to form a paste. If there is too much moisture the product density will be too high and the product will not swell properly but instead will remain a fluid paste which is unstable and which tends to collapse. If too little moisture is present, a thick or stiff, non-flowable, non-swelling paste results which is also highly undesirable. It is preferred that the moisture content of the paste be about 19 to 20%.

In proposed methods where the hydratable salt is added after the sodium sulfate, e.g., see South African Pat. No. 3895/60 more moisture must be present in order to dissolve the salt. However, it is now found that, in that process, not all of the salt becomes hydrated, and there is moisture remaining which is not bound to the salt. Some of this unbound moisture must therefore be removed by passing the material through a drier so that a desirable moisture content of between 18 and 19% is obtained. At this moisture content, the finished product has the proper flowing and noncaking properties. With the present discovery, less moisture can be formulated into the process since there is sufficient moisture after the silicates have been added to enable the hydratable salt to be better mixed in and dispersed in the mixture. In the proposed methods, the hydratable salt is added to a high solids containing slurry whereas in the present invention the hydratable salt is added to a low solids containing slurry (thin slurry). By this change in the order of addition, it has been found that the hydratable salt is more effectively hydrated thereby imparting more desirable qualities to the detergent composition. Since less moisture is required by the new process, the amount of water used in the formulation may be decreased. By reducing the amount of moisture incorporated into the mixture, the final moisture content at the end of the screening stage, including water of hydration, is approximately 18.5%. This is the desired final water content and there is therefore no need to further dehydrate the composition. Thus, the drier is eliminated and the dust and fines which are created during the drying and cooling process are also eliminated by the present invention.

The water content of the paste employed in carrying out the process of the present invention may vary upwards from about 15% of the paste composition, depending upon the other constituents present and the processing conditions employed. A preferred range for the water content of the paste is from about 18 to 22, most preferred being from 19 to 20%. In any event,

enough water must be used such that the mass is of a viscosity and fluidity that it can be stirred to form a uniform paste having a consistency which permits it to swell or expand but which prevents the rise of oxygen bubbles through the paste and therefore substantially preserves a structure of thoroughly dispersed fine gas bubbles, preventing substantial coalescence of the gas bubbles or loss of gas from the system.

Detergents which may be used in the process of the present invention are water-soluble and organic in nature and in general should have foaming properties. Examples of suitable anionic detergents include water-soluble soaps and sulphonated synthetic detergents. The soaps are generally water-soluble salts of fatty acids (including rosin acids) which are derived usually from fats, oils and waxes of animal, vegetable or marine origin, e.g., tallow coconut oil, tall oil and palm kernel oil. It is preferred to employ an alkyl aryl sulphonate such as an alkylbenzene sulphonate wherein the alkyl group has 8 to 16 carbon atoms. Suitable examples are sodium decyl, dodecyl and pentadecyl benzene sulphonates. Other suitable anionic detergents are surface-active sulphated or sulphonated aliphatic compounds, preferably having 8 to 22 carbon atoms, for example: long-chain pure or mixed alkyl sulphates (e.g., sodium lauryl sulphate); fatty acid ethanolamide sulphates (e.g., sodium coconut fatty acid ethanolamide sulphate); fatty acid amides of amino alkyl sulphonic acids (e.g., sodium lauric acid amide of taurine); and fatty acid esters of isethionic acid. These anionic detergents are used generally in the form of the water-soluble salts, such as the alkali metal (e.g., sodium, potassium) salts, though other water-soluble salts such as ammonium, alkylamine and alkaline earth metal salts may be used if desired, depending upon the particular detergent composition.

Also, the organic detergent may be, in whole or in part, a nonionic detergent such as the nonionic polyalkylene oxide condensates with an aliphatic or aromatic hydrophobic group. The hydrophobic organic group contains usually at least 8 carbon atoms and is condensed with at least 5 and usually up to 50 alkylene oxide groups. Examples are polyethylene oxide condensates with alkyl phenols having 6 to 20 carbon atoms in the alkyl group; polyethylene oxide esters of fatty acids such as tall oil acids or lauric acid with 16 to 20 ethylene oxide groups per molecule; polyethylene oxide condensates of aliphatic alcohols such as lauryl, myristyl, oleyl or stearyl alcohol with 6 to 30 moles of ethylene oxide per mole of the alcohol; and polyoxyethylene oxide condensates of fatty acid amides such as coconut fatty acid amide with 10 to 50 moles of ethylene oxide per mole of the amide. Water-soluble polyoxyethylene condensates with hydrophobic polyoxypropylene glycols may be employed also. Suitable nonionic detergents may be, for example, the condensate of ethylene oxide with polypropylene glycol, which condensate contains 80% ethylene oxide and has a molecular weight of about 1700, and iso-octyl phenoxy polyoxyethylene ethanol having about 8.5 ethanoxyl groups per molecule.

A cationic surface-active agent may be incorporated in the product also. It may be admixed in powdered or liquid form with the other ingredients in any suitable manner. Where it is desired to use a cationic agent, it is preferred to admix it in minor proportion with an acidic ingredient and coat this mixture with a coating agent. Suitable cationic detergents are alkyl quaternary am-

monium compounds such as cetyl quaternary ammonium salts. Specific examples of such materials are cetyl trimethyl ammonium chloride and cetyl pyridinium chloride. Similarly, ampholytic detergents such as salts of the N-alkyl compounds of beta amino propionic acid wherein the alkyl group is derived from a fatty acid such as a mixture of coconut oil fatty acids may be employed in compatible amounts. An example of such an ampholytic detergent is sodium dodecyl beta-alanine.

The detergent may in whole or in part be a fatty acid soap such as kettle soap. The presence of kettle soap facilitates the processing by imparting more rapid setting and acration characteristics to the slurry. The soap itself lends some structural strength to the product and thereby reduces breakdown of the product particles. Suitable soaps include the water-soluble alkali metal, amine and ammonium salts of fatty acids, such as those containing from 10 to 18 carbon atoms, e.g., sodium laurate, sodium myristate, potassium palmitate, triethanolamine stearate, and mixtures thereof with each other and with soaps of unsaturated fatty acids such as sodium oleate (e.g., the sodium soap of tallow fatty acids and the sodium soap of between 65:35 and 85:15 mixture of tallow and Palm Kernel Oil or coconut oil fatty acids).

A suitable range of proportions of the organic detergents is from 2 to 65% of the finished product, and preferably 10 to 40% where the detergent is a synthetic material and 20 to 65% where a soap is the active material.

A water-soluble hydrotropic alkyl aryl sulphonate is employed as an aid in the successful entrainment of gas released during the process. As much as 20% of this material can be used; however, it is usually desirable to include not more than 6% of the hydrotropic alkyl aryl sulphonate, while up to 2% is preferred. The hydrotropic alkyl aryl sulphonates used may be water-soluble hydrotropic agents characterized by a hydrophilelipophile balance such that they are highly water-soluble (more so for example, than related organic detergents), and while exhibiting considerable surface activity, are substantially non-detergent in nature. Examples of suitable hydrotropes are sodium toluene sulphonate, sodium xylene sulphonate, sodium dibutyl naphthalene sulphonate, sodium (mono-) dodecyl oxy dibenzene disulphonate, and the corresponding potassium and lithium salts and mixtures thereof, including commercially available isomeric mixtures. Preferably the alkyl substituent contains from 1 to 6 carbon atoms for each sulphonate group in the molecule, and the aryl nucleus desirable is benzene or naphthalene. The hydrotropic agent may be singly or plurally sulphonated or alkylated or both. Although reference has been made to alkali metal salts of these compounds, other water-soluble salts such as the alkaline earth metal, e.g., magnesium, salts and ammonium and substituted ammonium, e.g., the triethanolamine salts may be used totally or partially in lieu thereof.

In operation, the hydrotropic alkyl aryl sulphonate may be added to the slurry as such, or it may be added in the form of the sulphonic acid and neutralized in the presence of other constituents. Likewise, the hydrotropic alkyl aryl sulphonic acid may be prepared by sulphonation of the appropriate hydrocarbon in the presence of other constituents if desired, for example, by sulphonation with the same sulphonating agent used to prepare other sulphated sulphonated constituents of

the composition, such as an alkyl aryl sulphonate detergent. Such co-sulphonation may be either simultaneous or tandem as appropriate, it being preferred to sulphonate the hydrotropic alkyl aryl hydrocarbon as the last material to be sulphonated in the latter case.

The inorganic salts, e.g., silicates, employed in the present invention are present in the detergent composition in amounts ranging from 3 to 14% as silicate solids. Normally, the silicates are introduced as an aqueous solution having a $\text{Na}_2\text{O}:\text{SiO}_2$ ratio of from about 1:1 to about 1:3.3, preferably 1:1.5 to 1:2.5, most preferably 1:2 and a solids content of 35 to 55% and preferably 40 to 50% of the aqueous solution. The amount of silicate solution employed in the process ranges from about 9 to about 26% and preferably 10 to 20% and more preferably 11 to 15% by weight of the detergent composition.

Fillers, for example, sodium sulfate, are employed in the present invention usually in amounts ranging from 2 to 20% and preferably 5 to 12% by weight of the composition. Other suitable fillers are sodium carbonate.

The product may contain other ingredients which impart desirable properties to the paste during processing, to the final product, or to both. Among such ingredients are organic colloidal materials with soil suspending properties, foam boosters, builders, coloring agents (in the event a colored product is desired), and the like.

Sodium carboxymethyl cellulose is a preferred example of an organic colloidal material with soil-suspending properties for use in the invention. In addition to its known functions as a soil-suspending agent when the finished product is used in laundering, sodium carboxymethyl cellulose appears to affect favorably the hydration of the tripolyphosphate preferred for the purposes of this invention where the formation of the hydrate, and consequent setting of the paste into the friable mass, should be delayed or the rate of hydration slowed to permit expansion of the paste before the paste becomes too rigid in order to yield a product having the desired low bulk density. It may be present from about 0.5 to 1.5%.

Since the hydratable salt is added to a thin slurry, it must be one which hydrates at a slow enough rate, so that it will not form a thick, unmixable paste in the mixer. The hydratable salt used in the process of the present invention preferably is one which forms a stable hydrate at room temperature. The most highly preferred inorganic salt is Form II pentasodium tripolyphosphate. Form II is a slowly hydrating salt. Other slowly hydrating salts are also useful such as seeded or wetted Form I pentasodium tripolyphosphate.

A suitable range of proportions of the hydratable salt is from about 10 to about 75% by weight of the finished product, more preferred is 25 to 50% and most preferred is 30 to 40% by weight of the finished product.

A preferred oxygen-liberating per-compound for use in accordance with the present invention is hydrogen peroxide, which forms oxygen and water on decomposition, providing effective bleaching and swelling. It is easy to incorporate hydrogen peroxide in, and to disperse it thoroughly throughout the paste so that the oxygen is liberated substantially uniformly throughout the paste to bleach it and to cause it to expand to a desired low bulk density of less than 0.32 gram per cubic centimeter, preferably from 0.2 to 0.30 gram per cubic centimeter. Under the alkaline conditions and at the elevated temperatures which are preferred at the

mixing stage of this process (about pH 9.5 to 11 and a temperature of about 35° to 60°C.), hydrogen peroxide does not require the presence of any additional reactant but rather decomposes at a suitable steady rate effectively to bleach the detergent composition and to yield small, uniform bubbles of gas. The hydrogen peroxide may not completely decompose. Sometimes up to 15% hydrogen peroxide remains in the final product either as such or possibly as a complex hydrate of the phosphate. Commercial grade hydrogen peroxide of about 20 to 55% concentration, which may contain a stabilizer, is satisfactory for the purposes of the present invention in which it is used in a suitable proportion, e.g., from about 0.25 to about 1.0% by weight of the paste, to confer the desired bleaching effect on, and low bulk density to the final product.

The detergent particles produced by the instant process have numerous convex faces meeting at acute angles, a structure which presumably derives from the fact that the rigid mass from which these particles are prepared by mechanical action tends to fracture along planes passing through the fine bubbles contained therein. The spongy, foraminous character of the instant particles, which extends throughout their structure, is believed to form the basis of their high rate of solubility in water. A substantial majority of the spherical voids therein, which represent the gas bubbles formed in the original paste, are of a diameter of about 0.2 millimeter or less, and virtually none of them are larger than about 0.6 millimeter. Thus, substantially all of the particles, which typically have an average diameter of less than about 2 millimeters inasmuch as they have been screened through a sieve with openings of 2 millimeters, exhibit a cross sectional diameter of at least three times the diameter of the largest single gas bubble or void within the particle, and in most instances of at least five times.

A foam booster, e.g., cocodiethanolamide or the monoethanolamide of coconut oil fatty acids which is a waxy solid at temperatures below about 65°C., may be used advantageously in the products of the invention, typically in a proportion of from about 2 to 7% of the final product.

Malamine may be incorporated to inhibit the attack of washing solutions on copper and copper-bearing alloys.

Optical brighteners, pigments, preservatives and the like may also be present in the compositions of the present invention in the amounts commonly used in detergent compositions, e.g., 0.05 to 5% by weight of the composition.

In order for the desired results of the present invention to be obtained, the above additional ingredients are added prior to the addition of the tripolyphosphate. The hydrogen peroxide is the last constituent to add to the previously mixed components. After it is added, mixing of the final paste is continued only for the minimum period of time necessary to accomplish thorough mixing of all constituents. This may be less than one minute and preferably is on the order of about 30 seconds.

The following examples illustrate the invention but are not to be construed as limiting it. The term "apparent density" in this example and elsewhere throughout the present specification and claims refers to the untamped weight per unit volume of the particulate material as it is charged into a container.

EXAMPLE I

In this example, a batch process is illustrated in which 12.0 parts of water, 12.4 parts of aqueous sodium silicate containing 44.1% of solids having a $\text{Na}_2\text{O}:\text{SiO}_2$ ratio of 1:2.0, 1.9 parts of commercial sodium toluene

EXAMPLE II

The following tabulation indicates the ingredients and parts by weight of a variety of suitable formulations which may be prepared in accordance with the process set forth in Example I.

TABLE

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
Total water in paste	18	18.5	19	19.5	20	20.5	21	21.5	22	19	20
Sodium silicate	6.0	6.0	6.0	—	6.0	6.0	6.0	6.0	6.0	6.0	6.0
Sodium carboxymethyl cellulose	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	—
Fluorescent dye	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Tridecyl benzene sulfonate	2.4	—	19.0	27.0	27.0	27.0	27.0	19.0	27.0	27.0	—
Ethylene oxide condensate	—	10.0	—	—	—	—	—	—	—	—	7.0
Total sodium sulfate	9.1	26.1	4.1	15.1	9.1	9.1	9.1	4.1	9.1	11.1	9.9
Pentasodium tripolyphosphate	61.6	37.0	—	37.0	37.0	37.0	37.0	50.0	37.0	37.0	7.0
Hydrogen Peroxide:											
(As 35% solution)	0.6	0.6	0.6	0.6	—	0.3	0.9	0.6	0.6	0.6	0.6
(As 20% solution)	—	—	—	—	0.6	—	—	—	—	—	—
Sodium toluene sulfonate	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	—	2.0
Tetrasodium pyrophosphate	—	—	50.0	—	—	—	—	—	—	—	—
Moisture Content of Screened Product, percent	16.5	17.0	17.5	18	18.5	19	19.5	20	20.5	17.5	8.5
Apparent Density, Grams/cc	0.31	.25	.30	.21	.27	.21	.29	.20	.25	.22	.21

sulfonate, 1.0 parts of commercial sodium carboxymethyl cellulose (74% organic active ingredient), and 0.1 parts of fluorescent brightener are thoroughly mixed in a tilting bowl, sigma blade type mixer, and 27.2 parts of commercial sodium tridecylbenzene sulfonate are added to the mixer. These materials are mixed to form a uniform slurry, which is heated to a temperature of 60°C. while mixing. To this mixture which has a total moisture content of 34.71%, 31.5 parts of Form II pentasodium tripolyphosphate dry powder are added and thoroughly mixed within less than about one minute. After the tripolyphosphate has been mixed into the slurry, 5.8 parts of sodium sulfate are added and then 1.7 parts of a 50% solution of hydrogen peroxide are added to the mixer. The final paste is then mixed for 30 seconds. A total of 20.3 % water has been added during this process although some of the free water has already been eliminated through the hydration process. Immediately after the mixing is discontinued, the paste is dumped from the mixer into a tub having a volume approximately three times that of the paste (prior to expansion). The material in the tub is permitted to stand quiescently in a room at 25°C. During the first 15 minutes it is in the tub, the paste swells to its final volume, the temperature of the paste rising to about 65°C. in about 30 minutes. The material in the tub is permitted to cool to about 35°C., which, in the case of a 230 kilogram batch exposed to an ambient temperature of 25°C., requires about 48 hours. The cooled, rigid material in the tub is then removed therefrom in the form of large lumps which are passed through a rotating cage mill and then screened through a sieve having square apertures 2 millimeters on a side. The moisture content of the solidified mass is approximately 20% initially, but during the mixing and aging steps there is a loss of moisture such that the final particles contain only 18.5% moisture.

The product produced has an apparent bulk density of 0.28, has improved texture, and is rapidly soluble in water.

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications of this invention can be made and that equivalents can be substituted therefor without departing from the principles and true spirit of the invention.

What is claimed is:

1. In a process for the preparation of a particulate detergent composition wherein about 15-40% water, about 2-65% organic detergent, less than about 20% hydrotrope, about 9-26% of aqueous inorganic silicate solution, about 2-20% of inorganic salt filler, about 10-75% of a hydratable salt selected from the group consisting of Form II pentasodium tripolyphosphate and Form I pentasodium tripolyphosphate and about 0.25-1% of an oxygen-liberating percompound are mixed to form a paste capable of retaining small oxygen bubbles without substantial coalescence thereof, liberating oxygen from said percompound into said paste in an amount sufficient to bleach said paste and to expand said paste to a final volume at least two times the initial volume thereof, setting the expanded paste under quiescent conditions to a friable mass of substantially said final volume, and granulating said friable mass to form particles of a bleached detergent composition, the improvement comprising adding said hydratable salt prior to the addition of said filler whereby the hydratable salt is more effectively hydrated resulting in more uniform particles having less fines and having an apparent density of less than about 0.32 gram per cubic centimeter.

2. A process in accordance with claim 1 wherein the filler material is sodium sulfate and the hydratable salt is Form II pentasodium tripolyphosphate.

3. A process in accordance with claim 2 wherein the hydrotrope is sodium toluene sulfonate.

4. A process in accordance with claim 3 wherein the water content is maintained between 18 to 19% by weight of the formulation so that further dehydration in a rotary drier is eliminated.

5. In a batch process for the preparation of a bleached particulate detergent composition which comprises the steps of mixing in the following order:

9

12.0 parts of water, 12.4 parts of aqueous sodium silicate containing 44.1% of solids having a $\text{Na}_2\text{O}:\text{SiO}_2$ ratio of 1:2.0, 1.9 parts of commercial sodium toluene sulfonate, 1.0 parts of commercial sodium carboxymethyl cellulose (74% organic active ingredient), 27.2 parts of commercial sodium tridecylbenzene sulfonate, and 0.1 parts of fluorescent brightener in a tilting bowl, sigma blade type mixer, to form a uniform thin solids slurry, heating said slurry to a temperature of 60°C. while mixing, adding 31.5 parts of Form II pentasodium tripolyphosphate dry powder to the slurry and thoroughly blending it within the space of about one minute, adding 5.8 parts of sodium sulfate and 1.7 parts of a 35% solution of hydrogen peroxide to the mixer and mixing the final paste for 30 seconds, immediately

10

dumping the paste from the mixer into a tub having a volume approximately three times that of the paste (prior to expansion), allowing the material in the tub to stand quiescently in a room at 25°C., so that during the first 15 minutes it is in the tub, the paste swells to its final volume, and the temperature of the paste rises to about 65°C. in about 30 minutes, permitting the material in the tub to cool to about 35°C., removing the cooled, rigid material in the tub in the form of large lumps and screening them through a sieve having square apertures 2 millimeters on a side, the final moisture content of the solidified mass is about 18.5% by weight and the mass has a bulk density of 0.28 gram per cubic centimeter.

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