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PROCESS FOR PREPARING SPRAY-DRIED BLENDED DETERGENTS

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ABSTRACT OF THE DISCLOSURE

Improved detergent compositions are prepared by addition of a portion of the total formulation to the remainder of the ingredients after these have been spray dried. Specifically it has been found that blending composites of hydrated alkali metal silicate glass and sequestering agents with the spray dried granules of the other detergent ingredients yield formulations with improved properties. Compared with conventional spray drying of complete detergent compositions, this process allows more uniform distribution of minor components throughout the formulation, higher alkali metal silicate levels, and decreases or even eliminates the formation of insolubles. An additional advantage of this process is that it permits increased production rates.

INTRODUCTION

Spray dried laundry detergents are usually prepared by combining the ingredients in a slurry of about 60% solids concentration in a blending device called a crutcher, followed by spray drying of the slurry to obtain a granular, free-flowing product of about 10% water content. A home laundry detergent typically includes 5 to 40% surfactant or combination of surfactants, 10 to 50% sequestering agent, such as sodium tripolyphosphate (STPP) or organic sequestering agents; 5 to 15% sodium silicate; 0 to 5% anti-redeposition agent, such as soduim carboxymethylcellulose (NaCMC); 0 to 25% sodium sulfate and 0 to 2% optical brightener. Other ingredients such as perfumes, dyes, enzymes, etc. are added after spray drying.

In general, such a spray dried detergent may be prepared as follows. The surfactant, which is generally a linear alkylaryl sulfonate, is processed by sulfonating the alkylate group in sulfuric acid and then neutralizing with 50% caustic. The result is a paste of 50% solids that contains the surfactant and sodium sulfate. The paste is placed in the crutcher and the other ingredients are added and blended to form a slurry of 60 to 70% solids which is then spray dried.

Generally, the sodium silicates, which are included in detergents for their detergent building, corrosion inhibiting and bead forming properties, are added to the crutcher mix as solution containing 40 to 60% water. Most of this water, along with the water added with the surfactant, must be removed in the drying tower to give a free-flowing, granular product.

The production or throughout of any spray drying tower is dependent upon the size of the spray tower; the conditions of operation, which are often fixed and the amount of water that must be removed to give an apparently dry product. Therefore, since the amount of water added with the surfactant paste is fixed, the amount of alkali metal silicate necessary for the best alkalinity, buffering, deflocculation and corrosion prevention, cannot be added as solutions without reducing the production of the spray tower. Also, the amount of alkali mteal silicate necessary to allow replacement or reduction of phosphates cannot be added as a solution. However, if the amount of water added with the alkali metal silicate can be reduced and the silicate added after spray drying, the silicate content of the detergents can be increased without a decrease in detergent production.

Sequestering agents have been added to detergents and cleaners to enhance performance, or to allow reduction or replacement of phosphates. It has been found that phosphate in detergents may be injurious to the environment and the levels of phosphate used should be reduced or eliminated from detergents entirely. Therefore, a greater variety of sequestering agents must be considered for use in detergents. Most currently available sequestering agents are very small in particle size and are inconvenient in storage, handling and processing; they cake, are subject to dusting, and form lumps when in contact with moisture or liquid components of detergent slurries. Many other sequestering agents are available as solutions of 40 to 60% so that only limited amounts of such sequestering agents can be added without decreasing the detergent production.

Also, it is generally known that insolubles are formed on drying of spray dried detergents containing alkali metal silicate. These materials, consisting mostly of silica, also form on storage. The formation of these insolubles is generally the result of the interaction of the alkali metal silicate with relatively acidic components in the detergent.

Some detergent ingredients, such as enzymes, dyes, perfumes, etc. cannot be spray dried with the detergent slurry because of their sensitivity to heat. Other ingredients such as colored particles cannot be added prior to spray drying because it is desired that they retain their individual character. Generally, these materials are present in very small quantities and obtaining uniform dosage and distribution of these ingredients is difficult.

It has been found that by changing the manufacturing process of making spray dried detergents, the alkali metal silicate content of the detergent can be increased without decreasing the production of detergent and in some cases even permitting an increase in the rate. Also the sequestering agent can be added to the detergent without lumping in the crutcher, insoluble formation is reduced, and more uniform distribution of minor ingredients is assured. This new process involves preparing a detergent slurry that is deficient in alkali metal silicate but does contain sufficient silicate to prevent corrosion. The slurry is also deficient or essentially free of sequestering agent. Such a slurry is dried in a spray tower to provide free-flowing surfactant containing granules. A post spray drying additive is formed by blending a composite of hydrated alkali silicate glass and sequestering agents with minor detergent ingredients that cannot be spray dried. This post additive is then blended with the surfactant containing granules to form a complete detergent.

THE INVENTION

The hydrated alkali metal silcate glass-sequestering agent composite employed in this invention, can most conveniently be prepared as described in a co-pending patent application S.N. 109,577. The composite is essentially a granular material with moisture content of 5 to 30%. The composite is readily and almost completely soluble when 5 g. is dissolved in 95 g. of water at 75° C. for 5 minutes. The bulk density of the composite can vary

from 20 to 70 lbs./cu. ft. The ratio of sequestering agent solids to alkali metal silicate solids can vary from 0.10:1.0 to 3.4:1.0.

The alkali metal silicate glass of the composite can have a mole ratio of SiO_2/M_2O that varies from 1.2/1 to 4.0/1.0 where M stands for an alkali metal or a combination of alkali metals. Commonly, sodium silicate glasses are used but potassium silicate glasses and sodium-potassium glasses are useful.

The sequestering agent used in the composite can be any chemical capable of reducing the activity of metal ions in solution and maintaining the ions in solution. The most important of such metal ions are Ca^{++} and Mg^{++} which precipitate soap and silicates, and decrease the efficiency of surfactants and, in general are responsible for the reactions of hard water. Many chemicals reduce the activity of metal ions in solution but do not maintain the ions in solution while the agents employed in this invention achieve both functions. Such agents include the following organic acids and their salts, especially sodium and potassium salts: nitrilotriacetic acid (NTA), ethylenediaminetetraacetic acid (EDTA), diethylenetriaminepentaacetic acid (DPTA) and citric acid. Polyacids, polyacid hydroxides and their salts are useful; examples of these materials are ascorbic acid, glycolic acid and lactic acid. Other useful materials are diethanol glycene, sodium gluconate, sodium glucoheptonate and sodium boroglucoheptonate.

The detergent slurries of this invention can be prepared in a crutcher by blending 10 to 85 p.b.w. of surfactant paste, such as 28% linear alkylaryl sulfonates, 22% Na_2SO_4 and 50% H_2O; 0 to 5 p.b.w. antiredeposition agent, such as sodium carboxymethylcellulose; 0 to 2% optical brightener and in some cases 5 to 35 p.b.w. of sodium tripolyphosphate (STPP). Some alkali metal silicate solution is added to prevent corrosion of equipment and to facilitate bead formation. Ingredients such as soda ash, borax and the like, generally classified as bulking agents, can also be included. This slurry is blended in the crutcher for a few minutes at 75 to 90° C. before it is pumped into the spray tower and dried with hot air to give a free-flowing granular, apparently dry product.

The surfactant containing granules are then blended with what we term a "post additive composite." This "post additive composite" consists of the hydrated alkali metal silicate glass-sequestering agent composite blended with minor ingredients that cannot be spray dried such as enzymes, colored granules, perfumes, dyes and the like. Almost any standard dry blending equipment such as ribbon blenders, twin shell blenders, etc. are useful for both blending operations. This method assures more uniform distribution of these minor ingredients. The ratio of spray dried surfactant containing granules to the "post additive composite" can vary from 10.0:1.0 to 0.5:1.0.

This process allows the amount of alkali metal silicate added to the detergent to be increased, while the amount of water that needs to be removed in the spray tower can be reduced or at least maintained at a constant level. If the surfactant level in the final detergent mixture can be lower than the level realized from present practice, the amount of solids produced by the spray tower can be higher than those realized from present practice. When the post additive is added the production rate is greatly increased. If the surfactant level in the final detergent mixture should be about equivalent to the levels realized from present practice, then the amount of solids realized from the spray dryer in this process may be somewhat lower than those realized from present practice. However, when the surfactant containing granules are combined with the post additive the production rate of complete detergent is higher than that of the usual process.

A very important advantage of this invention is the low levels of insoluble material that are formed on drying and aging of the detergent. There seems to be a number of reasons for this reduced tendency to form insolubles. The bulk of the silicate is not dried with whatever relatively acidic materials the detergent slurry may contain so that insoluble silica is not formed on drying. Also the silicate is not part of the detergent bead so that interaction of other detergent ingredients with the silicate is limited, decreasing the chances of insoluble formation. Also the composites of alkali metal silicate glass and alkali metal salts of organic sequestering agents appear to associate in such a way that they retard the formation of insolubles on aging. We believe that this behavior is the result of interaction between the 2 materials involving the alkali metal ions and rendering the silicate less prone to insoluble formation.

EXAMPLES

A further understanding of the invention can be obtained from the following illustrative examples that should not be considered restrictive. Examples 1 and 2 illustrate the practice of the industry prior to this invention while Examples 3–6 illustrate applicants' invention.

EXAMPLE 1

A slurry of the following composition was made in a crutcher using a sodium silicate solution (mole ratio 2.4 SiO_2/Na_2O, 50% solids) and anhydrous sodium nitrilotriacetic acid.

	P.b.w.
Surfactant paste (28% linear dodecylbenzene sulfonate, 22% Na_2SO_4 and 50% H_2O)	62.0
STPP	38.0
SNTA	10.0
Sodium silicate solution (2.4 SiO_2/Na_2O, 50% solids)	18.0
Na CMC	1.5
Blancophor RG (optical brightener, GAF Corp.)	0.3

This slurry contained 40 p.b.w. of water, of this 30 p.b.w. must be removed to produce a product of about 10% water content. The slurry was heated at 75° C. for 10 minutes and then pumped through a spray tower with full counter-current air flow and jet type spray. The inlet temperature was 320° C. and the outlet temperature was 93° C. The production rate was 5,000 p.b.w./hour so that 6500 p.b.w./hour of the slurry were pumped into the spray tower. Perfume (0.25 p.b.w.) and green particles (0.5 p.b.w.) were blended with 100 p.b.w. of the detergent but the uniformity of the blend as poor. The product was free flowing and granular with a density of 0.35 g./cc., a moisture content of 10.3% and a silicate solids content of 9.0%. The product was an efficient laundry compound and the insoluble content was 0.37%; after 6 months in a chipboard box at 70° F. and 50% R.H., the insoluble content was 1.22%.

EXAMPLE 2

A detergent slurry of the following composition was made in a crutcher.

	P.b.w.
Surfactant paste (28% linear dodecylbenzene sulfonate, 22% Na_2SO_4 and 50% H_2O)	62.0
STPP	38.0
Sodium boroglucoheptonate (NaBGH) (50% solution)	20.0
Sodium silicate solution (2.4 SiO_2/Na_2O, 50% solids)	18.0
Na CMC	1.5
Blancophor RG	0.3

This slurry contained 50 p.b.w. of water; of this 40 p.b.w. must be removed to produce a product of about 10% water content. The slurry was heated at 75° C. for 10 minutes and pumped in the spray tower of Example 1 at 5,250 p.b.w./hour. The production rate was 3,750 p.b.w./hour of a free-flowing granular product which was blended with 37.5 p.b.w./hour of blue dye. The uniformity of the blend was poor. The product had a density

of 0.33 g./cc., moisture content of 9.7% and a silicate solids content of 9.0%. The product was an efficient laundry compound and had an insoluble content of 0.28%. After 6 months aging in a chipboard box at 70° F. and 50% R.H. the insoluble content was 0.99%.

EXAMPLE 3

A slurry of the following composition was prepared in a crutcher.

	P.b.w.
Surfactant paste (28% linear dodecylbenzene sulfonate, 22% Na_2SO_4 and 50% H_2O)	62.0
STPP	38.0
Sodium silicate solution (2.4 SiO_2/Na_2O, 50% solids)	2.0
Na CMC	1.5
Blancophor RG	0.3

This slurry contained 32 p.b.w. of water, of this 27 p.b.w. must be removed to produce a composition of the proper water content. The slurry was heated at 70° C. for 10 minutes and then pumped into the spray tower of Example 1 at 5,300 p.b.w./hour and the production rate of surfactant containing granules was 3,800 p.b.w./hour.

The post additive was prepared by blending 97 p.b.w. of a composite of hydrated sodium silicate glass and NaBGH (40% sodium silicate, 2.0% SiO_2/Na_2O; 40% NaBGH and 20% H_2O) with 1.5 p.b.w. of Alkalase enzyme (Novo Industri) and 1.5 p.b.w. of blue crystals. The complete detergent was prepared by blending 75 p.b.w. of the surfactant containing granules with 25 p.b.w. of the post additive. Thus, the complete detergent was produced at 5,100 p.b.w. The product was free-flowing, granular and did not cake; the enzyme and blue crystals were distributed uniformly throughout the detergent. The bulk density was 0.34 g./cc., the moisture content was 9.8% and the silicate solids content was 10.9%. The product was an efficient laundry detergent with an insoluble content of 0.10%. After six months in a chipboard box the insoluble content was 0.42%.

EXAMPLE 4

A slurry of the following composition was made in a crutcher.

	P.b.w.
Surfactant paste (28% linear dodecylbenzene sulfonate, 22% Na_2SO_4 and 50% H_2O)	62.0
STPP	20.0
Sodium silicate solution (2.4 SiO_2/Na_2O, 50% solids)	2.0
Na CMC	1.5
Blancophor RG	0.3

This slurry contained 32 p.b.w. of water, of this 29 p.b.w. must be removed to provide a composition of the proper water content. The slurry was heated at 68° C. for 10 minutes and then pumped into the spray tower of Example 1 at the rate of 4,300 p.b.w./hour. The production rate of surfactant containing granules was 2,800 p.b.w./hour.

The post additive was prepared by blending 97 p.b.w. of a composite of sodium silicate, sodium glucoheptonate, (NaGH) and sodium citrate (40% sodium silicate, 2.0 SiO_2/Na_2O; 30% NaGH, 12% sodium citrate and 18% H_2O), 1.0 p.b.w. Alkalase enzyme and 2.0 p.b.w. of blue dye. The complete detergent was prepared by blending 56 p.b.w. of the surfactant containing granules with 44 p.b.w. of the post additive. Thus, the complete detergent was produced at 5,500 p.b.w./hour. The product was a uniform blue, free-flowing granular product that did not cake, with a density of 0.37 g./cc., a moisture content of 10.6% and a silicate solids content of 18.1%. The product was an efficient laundry compound with an insoluble content of 0.11%. After 6 months in a chipboard box at 70° F. and 50% R.H. the insoluble content was 0.47%.

EXAMPLE 5

A slurry of the following composition was made in a crutcher.

	P.b.w.
Surfactant paste (28% linear dodecylbenzene sulfonate, 22% Na_2SO_4 and 50% H_2O)	62.0
Na_2CO_3	20.0
Sodium silicate solution (2.4 SiO_2/Na_2O, 50% solids)	2.0
Na CMC	1.5
Blancophor RG	0.3

This slurry contained 32 p.b.w. of water, of this 29 p.b.w. must be removed to produce a composition of the proper water content. The slurry was heated for 10 minutes at 85° C. and pumped into the spray tower of Example 1 at the rate of 4450 p.b.w./hour. The production rate of surfactant containing granules was 2,950 p.b.w./hour.

The post additive composite was made by blending 97 p.b.w. of a composite of hydrated sodium silicate glass and EDTA (30% 1.6 SiO_2/Na_2O sodium silicate glass, 55% EDTA and 15% H_2O) with 1.5 p.b.w. Alkalase enzyme and 1.5 p.b.w. blue crystals. The complete detergent was prepared by blending 50 p.b.w. of the surfactant containing granules with 50 p.b.w. of the post additive composite. Thus, the complete detergent was produced at 5,900 p.b.w./hour. The product was a free-flowing, granular material with blue crystals distributed uniformly throughout. The density was 0.35 g./cc., the moisture content was 9.8% and the silicate solids content was 16.0%. The product was an efficient laundry compound with an insoluble content of 0.08%. After 6 months in a chipboard box at 70° F. and 50% R.H. the insoluble content was 0.48%.

EXAMPLE 6

A slurry of the following composition was prepared in a crutcher.

	P.b.w.
Surfactant paste (28% linear dodecylbenzene sulfonate, 22% Na_2SO_4 & 50% H_2O)	62.0
NaGH	20.0
Sodium silicate solution (2.4 SiO_2/Na_2O, 50% solids)	2.0
Na CMC	1.5
Blancophor RG	0.3

This slurry contained 32 p.b.w. of water, of this 29 p.b.w. must be removed to produce a composition of the proper water content. The slurry was heated for 10 minutes at 78° C. and pumped into the spray tower of Example 1 at the rate of 4,450 p.b.w./hour. The production rate of surfactant containing granules was 2,950 p.b.w./hour.

The post additive composite was made by blending 97 p.b.w. of a composite of hydrated sodium silicate glass and sodium citrate (65% 2.0 SiO_2/Na_2O sodium silicate glass, 20% Na citrate and 15% H_2O) with 1.5 p.b.w. Alkalase enzyme and 1.5 p.b.w. blue crystals. The complete detergent was prepared by blending 50 p.b.w. of the surfactant granules with 50 p.b.w. of the post additive composite. Thus, the complete detergent was produced at 5,900 p.b.w./hour. The product was a free-flowing granular material with blue crystals distributed uniformly throughout. The density was 0.38 g./cc., the moisture content was 9.9% and the silicate solids content was 33.0%. The product was an efficient laundry compound with an insoluble content of 0.18%. After 6 months in a chipboard box at 70° F. and 50% R.H. the insoluble content was 0.57%.

A more or less detailed claim will be presented hereinafter and even though such claim is rather specific in nature those skilled in the art to which this invention pertains will recognize that there are obvious equivalents for the specific materials recited therein. Some of these obbious equivalents are disclosed herein, other obvious equivalents will immediately occur to one skilled in the

art, and still other obvious equivalents could be readily ascertained upon rather simple, routine, noninventive experimentation. Certainly no invention would be involved in substituting one or more of such obvious equivalents for the materials specifically recited in the claim. It is intended that all such obvious equivalents be encompassed within the scope of this invention and patent grant in accordance with the well-known doctrine of equivalents, as well as changed proportions of the ingredients which do not render the composition unsuitable for the disclosed purposes. Therefore, this application for Letters Patent is intended to cover all such modifications, changes and substitutions as would reasonably fall within the scope of the appended claim.

What we claim is:

1. An improved process for preparing detergent compositions which comprises:

(a) blending together a detergent slurry consisting of
  (1) 10–85% of a surfactant paste composed of an alkylbenzene sulfonate, sodium sulfate and water,
  (2) 0–5% of an antiredeposition agent,
  (3) 0–2% by weight of an optical brightener, and
  (4) about 2% of a sodium silicate solution,

(b) spray drying the detergent slurry of step (a) and recovering surfactant containing granules,

(c) preparing a post additive consisting of a granular composite consisting essentially of hydrated sodium silicate glass and a sequestering agent selected from the group consisting of nitrilotriacetic acid, sodium nitrilotriacetic acid, ethylenediaminetetraacetic acid, sodium ethylenediaminetetraacetic acid, potassium ethylenediaminetetraacetic acid, sodium gluconate, sodium glucoheptonate, sodium boroglucoheptonate and sodium citrate, said granular composite having a moisture content of 5–30% and a bulk density of 20–70 lbs./cu. ft., the ratio of the sequestering agent to hydrated sodium silicate being within the range of 0.10:1.0 to 3.4:1.0, the sodium silicate having a mol ratio of SiO_2Na_2O of 1.2:1 and 4.0:1.

(d) blending said surfactant containing granules with said granular post additive in a ratio between 10.0:1.0 to 0.5:1.0 so as to obtain an improved free-flowing blended granular detergent composition.

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