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PROCESS FOR PREPARING GRANULAR DETERGENT COMPOSITION

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ABSTRACT OF THE DISCLOSURE

A process for preparing a granular detergent composition which comprises spray drying a slurry of 70–40% by weight of a mixture of 10–40% by weight of an anionic surface active agent having an SO_3 group or an SO_4 group, 15–40% by weight of sodium tripolyphosphate, 15–40% by weight of sodium silicate having a ratio of Na_2O to SiO_2 of 0.5–3, 0.5–10% by weight of magnesium sulfate, and residual additives, in 30–60% by weight of water.

BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates to a process for preparing a granular detergent composition by the use of spray drying techniques. More particularly, this invention relates to a process for preparing a granular detergent composition containing particles of relatively high mechanical strength which are essentially non-caking.

Description of the Prior Art

Dry, granular form detergents are usually packaged for commercial or retail sales in cardboard boxes or cartons which are often subject to severe handling shocks before they reach the ultimate consumer. Since such package containers do not offer much resistance to such shocks, the forces are often transmitted to the detergent granules. One difficulty with many commercial detergent granular products is that they are readily crushable under the application of even moderate applied forces, so that during the package handling the detergent can powder into a fine particulate form. This causes a decrease in the apparent specific volume of the package and thereby decreases the commercial or retail value of the product.

A number of attempts have been made to increase the mechanical strength of the detergent granules, but heretofore, such attempts have resulted in either insufficient enhancement of mechanical strength, or have undesirably enhanced the tendency of the granules to "cake" or to coagulate into a more or less solid mass. Still other methods considered would have resulted in a decrease in productivity of the product, or would have adversely affected other properties of the detergent.

For instance, it was suggested to effect mechanical strengthening of the granules by altering the method of formation of the granules. It was found that mechanical strength could be improved by decreasing the rate of drying of the detergent composition, such as by the use of mild drying conditions. This method, however, decreased product productivity and hence could not find industrial acceptance.

It was also attempted to enhance mechanical strength by increasing the quantity of sodium tripolyphosphate. However, it was found that the degree of enhancement did not prove to be sufficiently high.

Another method of enhancing mechanical strength, suggested by the prior art was to add relatively large amounts

of sodium silicate to the composition. However, this severely increased the tendency of the granules to cake, particularly if the sodium silicate added exceeds 15% by weight of the total composition. Attempts at reducing the caking tendency, such as by the addition of such anti-caking agents as calcium halide, magnesium halide, or magnesium silicate, have not been effective in alleviating the caking tendency when large amounts of sodium silicate were present.

SUMMARY OF THE INVENTION

Accordingly, it is one object of this invention to provide a process for preparing a granular detergent wherein the granules are characterized by relatively high mechanical strength, but which do not have an increased tendency toward caking.

This and other objects, as will hereinafter become more readily understood, have now been attained by adding a combination of sodium silicate, to enhance the strength properties of the granules, and to add sodium tripolyphosphate and magnesium sulphate to reduce caking.

The process for preparing the granular detergent composition in accordance with this invention is characterized by spray drying a slurry of 70–40% by weight of a detergent composition containing 10–40% by weight of an anionic surface active agent having an SO_3 or SO_4 group, 15–40% by weight of sodium tripolyphosphate, 15–40% by weight of sodium silicate having a ratio of Na_2O to SiO_2 of 0.5–3, 0.5–10% by weight of magnesium sulfate and the remainder being other additives, in 30–60% by weight of water.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The granular detergent composition prepared in accordance with the process of this invention is characterized by a high mechanical strength and does not posses a high tendency to cake. Moreover, this detergent composition is characterized by a high degree of detergency and good perfume retention.

Although it is not completely understood why these desirable effects are obtained, it is theorized that the additives impart the following effect: Since the anionic surface active agent, the sodium tripolyphosphate, and the sodium silicate are all combined with the magnesium sulfate in a slurry, prior to spray drying, it is believed that magnesium salts of the anionic surface active agent of the tripolyphosphate, and of the silicate are formed. It is also believed that double salts of tripolyphosphoric acid and silicic acid with the magnesium ion, are formed. These magnesium compounds have a high degree of moisture retention and a high degree of hydration. A relatively large amount of water in the slurry is consequently retained by the salts.

During spray drying, the greater the solubility of the compound, the higher will be the concentration of the salts formed on the surface of the dried granules. Sodium silicate is normally one of the more soluble components of the composition, so that usually it will be formed in a high concentration at the surface of the granules. In the present invention, however, due to the formation of the magnesium salts and double salts, its solubility is decreased and the concentration of silicates formed at the surfaces of the granules is considerably reduced, while the concentration of the silicate within the granules, below the surfaces, is increased. Since caking tendency in the prior art compositions is enhanced by high silicate concentration on the surfaces of the granules, in the present composition, caking tendency is diminished while mechanical strength is either unaffected or is enhanced.

The fact that the concentration of silicate at the surface of the spray dried granules is quite small, can be confirmed

by chemical analysis of the components at the surface of a slurry containing magnesium sulfate as compared with the concentration on the surface of a large block prepared from the same detergent composition but without magnesium sulfate.

It has been found that magnesium chloride does not exhibit the same effect as magnesium sulfate, presumably because the rates of magnesium salt formation are substantially different, and because the solubilities of each is substantially different. Magnesium chloride, moreover, has a tendency of being double decomposed to form sodium chloride. Accordingly, magnesium chloride cannot be substituted for magnesium sulfate in the present invention.

It has also been found that the unique advantages of this invention are not obtainable if magnesium silicate or magnesium phosphate are used directly. The reason for this inadequacy is also not understood, but it can only be concluded that they do not contribute to the formation of the same type of salts.

Suitable anionic surface active agents which may be used herein are those having an SO_3 or an SO_4 group, such as:

sodium alkylaryl sulfonate (C_8–C_{18}),
sodium olefin sulfonate (C_8–C_{20}),
sodium alkyl sulfonate (C_8–C_{20}), and
sodium alkyl sulfate (C_8–C_{20}).

The sodium silicate should be used in amounts of from 15–40% by weight of total solids. Less than 15% results in a composition having inadequate strength properties. Greater than 40% can result in the formation of a large amount of water-insoluble material.

The sodium tripolyphosphate should also be used in amounts of from 15%–40% by weight. Greater than 40% is economically unadvantageous, whereas, less than 15% is generally ineffective.

It is difficult to obtain suitable detergency if less than 10% by weight of the anionic surface active agent to total solids is used, and it is difficult to provide a granular detergent if less than 40% by weight is used.

Magnesium sulfate should be used in an amount of from 0.5–10% by weight based on the total solid components in the slurry. If less than 0.5% is used, sufficient anti-caking is not obtained, whereas if greater than 10% is used, a large quantity of water-insoluble materials may be formed.

The granular detergent composition prepared in accordance with the process of this invention is characterized by a high degree of granule strength so that the granules are not easily crushed during package handling. Moreover, the granules do not suffer a tendency to cake even at relatively high temperatures and relatively high humidities.

Having now generally described the invention, a more complete understanding can be attained by reference to certain specific Examples which are provided herein for purposes of illustration only and are not intended to be construed as limiting unless otherwise so indicated. The term "parts" in these Examples is meant to indicate "parts by weight."

EXAMPLES 1–2

50 parts of sodium dodecylbenzene sulfonate (40% active ingredient and 60% water) was dissolved in 15.5 parts of water and the solution was admixed with 2.5 parts of carboxymethyl cellulose. 2 parts of sodium toluene sulfonate, 40 parts of sodium disilicate (45% active ingredient), 3 parts of polyoxyethylene nonylphenyl ether ($\overline{P}$=5), 10 parts of a 25% magnesium sulfate aqueous solution, 25 parts of sodium tripolyphosphate, and 22 parts of sodium sulfate, while stirring to prepare a slurry.

The slurry was spray dried using a disc type spray drier to prepare the granular detergent composition. Using a similar process, but using the compositions shown in Table I, the granular form detergents were prepared. All of the granular detergents respectively had a specific bulk density of 0.27.

In accordance with the comparative tests of strength of particles, caking property, detergency and perfume retention effect of the granular detergent compositions of Examples and References A–F, the following results were obtained:

TABLE I

Components	Example (percent)		Reference (percent)						
	1	2	A	B	C	D	E	F	G
Sodium dodecylbenzene sulfonate	20	20	20	20	20	20	20	20	20
Polyoxyethylene nonylphenyl ether ($\overline{P}$=5)	3	3	3	3	3	3	3	3	3
Sodium tripolyphosphate	25	25	25	25	25	10	25	25	25
Sodium disilicate	18	18	18	18	18	18	10	10	18
Carboxymethyl cellulose	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Sodium toluene sulfonate	2	2	2	2	2	2	2	2	2
Magnesium sulfate	2.5	5	0	0	0	2.5	0	2.5	0.2
Magnesium chloride	0	0	0	2.5	0	0	0	0	0
Magnesium disilicate	0	0	0	0	2.5	0	0	0	0
Sodium sulfate	22	19.5	24.5	22	22	37	32.5	30	24.3
Water	5	5	5	5	5	5	5	5	5
Perfume*	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

*The amount of perfume is shown in percent by weight to total components.

TABLE II

Item	Example		Reference						
	1	2	A	B	C	D	E	F	G
Strength of particles increasing rate (percent) 100 mesh pass	10	10	35	20	25	30	15	20	30
Caking property rate of cake (percent)	20	5	90	60	50	70	40	15	80
Detergency (percent)	95	93	87	92	89	70	80	85	87
Perfume retention effect (point)	6	6	3	4	3	5	5	5	3

The test methods were as follows:

(1) Caking property: 50 g. of sample was introduced into a cardboard (3 cm. x 8 cm.) box which had not been pre-treated for water-proofing. The packaged detergent was kept in a room of constant temperature and humidity, at 35° C. and 85% specific humidity, for 8 hours. The contents were then spread on a 12 mesh sieve. The residual caked detergent composition was measured. The caking rate was calculated by the percentage of weight of the residual composition to 50 g. of total composition. The lower the value, the higher the anti-caking property.

(2) Strength of granules: 50 g. of sample was introduced into a cardboard box (3 cm. x 10.5 cm. x 8 cm.) and was placed on a multi-purpose shaker (KM type manufactured by Iwaki Kagaku Co. RV–2 type) at 300 r.p.m. for 30 minutes. The contents were then spread on a 100 mesh sieve to weigh the amount of the sample passing through the sieve. The amount of sample passing through the 100 mesh sieve before shaking treatment was deducted from the quanity passing through the same sieve after shaking.

The strength of the granules was calculated as the percentage by weight of the deducted amount of the sample passing through the sieve per 50 grams of sample. The lower the value, the less crushing of granules occurred and hence the higher the strength.

(3) Detergency: A cotton cloth coated with natural soil (2% OWF) was washed in 0.2% of the sample detergent solution for 10 minutes using a Terg-O-Tometer at 120 r.p.m. at 25° C. with 3° DH hard water. The amount of soil removed by washing was measured and indicated as the measure of detergency.

(4) Perfume retention property: 50 g. of the sample was introduced into a cardboard box (3 cm. x 10.5 cm. x 8 cm.) which had not been pre-water-proofed, and kept in a room of constant temperature at 35° C. for 30 days. The intensity of perfume odor was measured sensually.

The intensity of perfume before the test is designated as 10 and the intensity at no perfume detection is designated as 0. The higher the number, the higher the perfume retention value.

Reference: Slurries of the formula as shown in Table III containing magnesium sulfate and not containing magnesium sulfate were prepared at about a 40% solid concentration. About 10 g. of each slurry was placed into a glass vessel and was kept in a constant temperature room at 130° C. for about 30 minutes to dry the slurry.

The resulting dry cake was in an unbroken semicircular configuration. This cake was carefully removed from the glass vessel and the surface was shaved off. A sample from the internal part of the dry cake was also taken and both samples were analyzed.

The results are shown in Table III.

TABLE III.—FORMULA OF DETERGENT

Component	Weight percent A	B
Sodium dodecylbenzene sulfonate	20	20
Sodium tripolyphosphate	30	30
Sodium disilicate	20	20
Magnesium sulfate	2.5	0
Sodium sulfate	27.5	30

TABLE IV.—RATIO OF EACH COMPONENT OF SURFACE LAYER TO INTERNAL LAYER

Component	Ratio ×100 A	B
Sodium disilicate	110	160
Sodium dodecylbenzene sulfonate	83	80
Sodium tripolyphosphate	105	90

As shown in Table IV, the amount of sodium disilicate in the surface layer was decreased by the addition of magnesium sulfate.

Having now fully described the invention, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without departing from the spirit or scope of the invention as set forth herein.

Accordingly, what is claimed and intended to be covered by Letters Patent is:

1. A process for preparing a granular detergent composition which comprises spray drying a slurry of 70–40% by weight of a mixture of 10–40% by weight of an anionic surface active agent selected from the group consisting of sodium alkylaryl sulfonate (C_8–C_{18}), sodium olefin sulfonate (C_8–C_{20}), sodium alkyl sulfonate (C_8–C_{20}), sodium alkyl sulfate (C_8–C_{20}) and mixtures thereof, 15–40% by weight of sodium tripolyphosphate, 15–40% by weight of sodium silicate having an Na_2O to SiO_2 ratio of 0.5–3, 0.5–10% by weight of magnesium sulfate in 30–60% by weight of water.

2. A granular detergent composition which comprises spray dried granules formed by spray drying a slurry consisting essentially of:

10–40% by weight of an anionic surface active agent selected from the group consisting of sodium alkylaryl sulfonate (C_8–C_{18}), sodium olefin sulfonate C_8–C_{20}), sodium alkyl sulfonate (C_8–C_{20}), sodium alkyl sulfate (C_8–C_{20}), and mixtures thereof,

15–40% by weight of sodium tripolyphosphate,

15–40% by weight of sodium silicate having an Na_2O to SiO_2 ratio of 0.5–3,

0.5–10% by weight magnesium sulfate wherein the concentration of silicate at the surface of the granules is less than the concentration within said granules.

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