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2,709,686

NON-CAKING DETERGENT COMPOSITIONS

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No Drawing. Application September 14, 1951,
Serial No. 246,706

5 Claims. (Cl. 252—161)

The present invention relates to particulate detergent compositions and, more particularly, to those particulate detergent compositions which contain sodium salts of C_9 – C_{18} monoalkyl benzene sulfonic acids as their primary detergent ingredient.

Despite spectacular advances made during the last decade by synthetic detergents of the alkyl benzene sulfonate type in replacing soap for numerous household uses, their acceptance by the residents of humid subtropical and tropical areas has been handicapped by the occurrence of caking of the detergent particles in the containers, when stored under the adverse climatic conditions prevailing in such areas. This caking is particularly severe when the particulate detergent products are packaged in the commonly employed cardboard containers.

It is, therefore, highly desirable to find ways of reducing the occurrence of undesirable caking in order to make the alkyl benzene sulfonate type detergents more attractive to the users in hot, humid climatic areas.

We have discovered that the extent of caking of the particulate alkyl benzene sulfonate detergent compositions containing in each 100 parts thereof from about 10 to about 95 parts of sodium C_9 – C_{18} monoalkyl benzene sulfonate detergent, the remainder of said compositions being essentially an inorganic sodium salt builder, can be effectively and substantially reduced by incorporating into these compositions a small amount of from about 1.0 to about 10.0% by weight of a sodium 2-hydroxy alkane sulfonate containing from 2 to 6 carbon atoms in the alkane portion thereof, based on the weight of the detergent and builder ingredients in such a composition. The additions of small amounts from about 1.0 to about 10.0% by weight of these sodium 2-hydroxy alkane sulfonates are particularly effective when employed to reduce caking of those sulfonate detergent compositions, which contain in each 100 parts thereof, from about 20 to about 60 parts of sodium C_9 – C_{18} monoalkyl benzene sulfonate, the remainder being inorganic sodium salt builders.

Sodium 2-hydroxy alkane sulfonates containing from 2 to 6 carbon atoms such as sodium 2-hydroxyethane-sulfonate, sodium 2-hydroxy propane sulfonate, 2-hydroxy butane sulfonate, 2-hydroxy pentane sulfonate, 2-hydroxy hexane sulfonate and 2-hydroxy cyclohexane sulfonate may be prepared in a known manner, for example, by reacting a corresponding epoxy-alkane with sodium bisulfite. Such 2-hydroxy alkane sulfonates, when added to an alkyl benzene sulfonate detergent composition in the aforementioned amounts ranging from about 1.0 to about 10.0% by weight of the whole detergent composition, reduce the degree of caking significantly.

Alkyl benzene sulfonate detergents which show pronounced caking tendency and require the use of caking preventives of our invention may be produced by sulfonation of alkyl benzenes containing from 9 to 18 carbon atoms in the alkyl chain. These alkyl benzenes may be prepared by one of the following two procedures: (1) Condensation of an olefin of about 9 to 18 carbon atoms such as a polymer of propylene or butylene with

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benzene in the presence of a suitable catalyst, e. g., hydrofluoric acid; or (2) condensation of a chlorinated kerosene fraction with benzene in the presence of an aluminum chloride catalyst. Alkyl benzenes produced by either of these two condensations are subsequently sulfonated by conventional sulfonation techniques, for instance, with the aid of fuming sulfuric acid or sulfur trioxide. After sulfonation, the resulting mixture of C_9 – C_{18} alkyl benzene sulfonic acid and unreacted sulfuric acid is neutralized with a solution of sodium hydroxide or sodium carbonate to yield a liquid aqueous solution or slurry of sodium alkyl benzene sulfonate and sodium sulfate. As a general practice, the sodium sulfate is allowed to remain in the slurry, since its presence in admixture with the sulfonate tends to extend the minimum effective concentration or "to build up" the detergent power of this latter. In fact, very frequently additional amounts of sodium sulfate and/or of other effective inorganic sodium salt builders are introduced into the neutralized slurry to assure the desired cleansing properties in the ultimate product. Following the neutralization and the addition of the inorganic builder, the slurry is reduced to the solid particulate form by an appropriate treatment such as spray- or drum-drying.

Various techniques may be employed to add sodium 2-hydroxy alkane sulfonates to the detergent alkyl benzene sulfonate compositions in the amounts from about 1.0 to about 10.0% by weight, effective to reduce the undesirable caking.

One procedure is to add sodium 2-hydroxy alkane sulfonates to the neutralized slurry of sulfonic and sulfuric acid together with the desired builders prior to their drying treatment. The more preferred procedure, however, is to introduce them into the caustic solution used for neutralizing the mixture of alkyl benzene sulfonic acid and unreacted sulfuric acid.

In order to ascertain the extent of caking of detergent compositions into which sodium 2-hydroxy alkane sulfonates have been introduced in accordance with our invention, as compared with the caking of detergent compositions without such additives, the following testing procedure was used:

A typical alkyl benzene sulfonate particulate composition containing 40% by weight of sodium C_{12} – C_{15} polypropylene benzene sulfonate as the active ingredient and about 50 to 60% by weight of sodium sulfate, and prepared in accordance with the procedure taught in U. S. Patent No. 2,477,382 issued to A. H. Lewis, was employed for the tests. A sample of this typical dried detergent composition was screened through a #16 sieve and collected on a #200 sieve, whereupon weighed amounts of about 100 g. were placed in untreated cardboard boxes measuring $1\frac{1}{2}$ " x $2\frac{1}{2}$ " x 4". These boxes were sealed and placed into a standard humidity cabinet or chamber in which the temperature and humidity were continuously controlled at 90° F. and 80% relative humidity. At the end of seven days, the boxes were taken out and the entire side of each box was cut away, placing the sample very carefully on a #4 sieve. The sieve was then gently tipped and swirled to allow free-flowing material to pass through the screen, but so as to avoid the breaking of any lumps which may have formed during the test. The weight in per cent of the material which would not pass through the screen was determined and recorded as the "per cent caked." In each test three samples of 100 g. containing the preventive were tested with three samples containing no preventive.

Example 1

A slurry composed of 267 parts of the aforementioned composition (60% by weight of sodium C_{12} – C_{15} polypropylene benzene sulfonate and 40% by weight of so-

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dium sulfate), 113 parts of sodium sulfate, 20 parts of sodium 2-hydroxyoctadecane sulfonate and 400 parts of water was mixed thoroughly and then drum-dried to give a product which had the following composition:

	Per cent
Sodium C ₁₂ -C ₁₅ polypropylene benzene sulfonate	40
Sodium 2-hydroxyoctadecane sulfonate	5
Sodium sulfate	55

When subjected to the 7-day caking test in the humidity cabinet, the product caked badly to the extent of 94% by weight.

Example 2

285 parts of C₁₂-C₁₅ polypropylene benzene sulfonic acids containing about 10% of sulfuric acid was added to a solution of 94 parts of sodium hydroxide and 27.4 parts of sodium 2-hydroxyethane sulfonate in 430 parts of water. To the resulting neutral slurry were added 206 parts of sodium sulfate and 200 parts of water. The final slurry was drum dried and gave a product of the following composition:

	Per cent
Sodium C ₁₂ -C ₁₅ polypropylene benzene sulfonate	35
Sodium 2-hydroxyethane sulfonate	5
Sodium sulfate	60

After testing this product for 7 days in the humidity cabinet, the caking was observed to amount to a mere 10% by weight.

Example 3

In this instance, 27.4 parts by weight of sodium hydroxymethane sulfonate was incorporated in the detergent composition of Example 2. The ultimate drum-dried product had the following composition:

	Per cent
Sodium C ₁₂ -C ₁₅ polypropylene benzene sulfonate	35
Sodium hydroxymethane sulfonate	5
Sodium sulfate	60

On exposure in the humidity cabinet (7-day test) the product was found to cake very badly. As much as 87% by weight of the product became caked.

Example 4

In this test the alkyl benzene sulfonate detergent composition was employed without incorporating therein any of the sodium 2-hydroxy alkane sulfonates as caking preventives—to 90 parts of sodium hydroxide in 430 parts of water were added 270 parts of the alkyl benzene sulfonic acid of Example 2 (C₁₂-C₁₅ polypropylene benzene sulfonic acids), 160 parts of sodium sulfate and 116 parts of water. Upon drum-drying of the resulting slurry, the ultimate particulate product was found to contain 40% of sodium C₁₂-C₁₅ polypropylene benzene sulfonate and 60% sodium sulfate. The caking test (7 days) of this product showed that the average extent of caking was equal to 65% by weight.

The foregoing representative examples demonstrate the remarkable improvement brought about by the incorporation of from about 1.0 to about 10.0% by weight of sodium 2-hydroxyalkane sulfonate containing from 2 to 6 carbon atoms in the alkane portion into the alkyl benzene sulfonate detergent compositions with respect to the reduction of caking tendency of these latter.

As indicated above, other inorganic salt builders than sodium sulfate, namely, various sodium phosphates such

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as trisodium phosphate, tetrasodium pyrophosphate and sodium tripolyphosphate as well as sodium carbonate, sodium bicarbonate and sodium chloride may be present in the detergent compositions without significantly interfering with the action of the caking preventives of our invention, provided each 100 parts by weight of the detergent compositions contains from 10 to 95 parts by weight of the active sulfonate ingredient, the remainder being essentially the inorganic builder.

Other modes of carrying into practice the principle of our invention than the one described hereinabove may be employed, provided that the critical proportions of ingredients recited in the appended claims are employed in the detergent formulations.

We claim:

1. A particulate non-caking solid detergent composition, each 100 parts by weight whereof contains from about 10 to about 95 parts by weight of sodium salts of monoalkyl benzene sulfonic acid containing from 9 to 18 carbon atoms in the alkyl chain as the effective detergent ingredient, the remainder of said 100 parts being essentially an inorganic sodium salt builder and from about 1.0 to about 10.0% by weight of a sodium 2-hydroxyalkane sulfonate having from 2 to 6 carbon atoms in the alkane portion, based on the combined weight of said detergent and builder.

2. A particulate non-caking solid detergent composition as defined in claim 1, wherein the sodium salt of monoalkyl benzene sulfonic acid contains from 12 to 15 carbon atoms in the alkyl chain.

3. A particulate non-caking solid detergent composition as defined in claim 1, wherein the alkyl chain of the sodium salt of monoalkyl benzene sulfonic acid is a polypropylene chain containing from 12 to 15 carbon atoms.

4. A particulate non-caking solid detergent composition in which each 100 parts by weight of said composition contains from about 10 to about 95 parts by weight of a sodium salt of a monoalkyl benzene sulfonic acid containing from 9 to 18 carbon atoms in the alkyl chain as the effective detergent ingredient, said salt normally tending to cake on storage, the remainder of said 100 parts being essentially sodium sulfate builder and from about 1.0 to about 10% by weight, based on the combined weight of said detergent and builder, of a sodium 2-hydroxyalkane sulfonate having from 2 to 6 carbon atoms in the alkane portion.

5. The method of inhibiting caking of solid, particulate detergent compositions comprising in each 100 parts by weight thereof from about 10 to about 95 parts by weight of sodium salts of monoalkyl benzene sulfonic acids having from 9 to 18 carbon atoms in the alkyl chain, the remainder to make up 100 parts by weight being essentially inorganic sodium salt builders, said composition normally exhibiting a tendency to cake when stored in a warm, humid atmosphere, said method comprising forming an aqueous slurry of said sodium alkyl benzene sulfonates and said inorganic sodium salt builders, having intimately mixed therein about 1.0 to about 10.0 per cent by weight of a sodium 2-hydroxy alkane sulfonate having from 2 to 6 carbon atoms in the alkane portion, based on the total solid content of said slurry, and spray-drying this slurry.

References Cited in the file of this patent

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2,477,383 Lewis July 26, 1949