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NON-CAKING DETERGENT COMPOSITION

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The invention described and claimed herein relates to detergent compositions and, more particularly, to those detergent compositions which contain alkyl benzene sulfonates as their active detergent ingredients.

Synthetic detergents of the alkyl benzene sulfonate type, by virtue of their excellent detergency, stability in hard water and in acid solutions, and because of their low manufacturing costs, represent at the present date a large proportion of the total volume of synthetic detergents sold in the United States. Particularly effective and consequently widely used detergents representative of this type are sodium salts produced by sulfonation of monoalkyl benzenes containing from 9 to 18 carbon atoms in the alkyl chain followed by neutralization with caustic. The above-mentioned monoalkyl benzenes suitable for the manufacture of detergent sulfonates are usually prepared by one of the following two procedures: (1) An olefin of about 9 to 18 carbon atoms such as a polymer of propylene or butylene is condensed with benzene in the presence of an alkylation catalyst such as hydrofluoric acid; or (2) a chlorinated kerosene fraction is condensed 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 mixtures of C_9–C_{18} alkyl benzene sulfonic acids and unreacted sulfuric acid are neutralized with sodium hydroxide or sodium carbonate to yield liquid dispersions or slurries of sodium alkyl benzene sulfonate and sodium sulfate. As a general practice sodium sulfate is allowed to remain in the slurry, since its presence in admixture with the sulfonate tends to extend its minimum effective concentration or "to build up" its detergent power. In fact, very frequently additional amounts of sodium sulfate or other 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 sulfate, the slurry is reduced to solid particulate form by an appropriate treatment such as spray- or drum-drying.

Notwithstanding the superiority of the particulate detergent formulations containing sodium alkyl benzene sulfonates as their active ingredients over other types of commercial detergents, these formulations possess a drawback of becoming caked when exposed to warm, humid climatic conditions. Since the more economical and most convenient type of container for household particulate detergents is the inexpensive carton permeable to the atmospheric moisture, the problem of preventing the caking of sodium alkyl benzene sulfonate detergent compositions packaged in cartons and stored in warehouses, on store shelves and in the home, in warm, humid, tropical or sub-tropical climates, is one of considerable practical importance.

It has now been found that the caking of detergent compositions containing in each 100 parts from about 10 to about 95 parts by weight of active sodium alkyl benzene sulfonate having from 9 to 18 carbon atoms in the alkyl chain, the remainder being essentially an inorganic sodium salt builder, may be substantially reduced by incorporating into said composition a small amount of from about 1.0% to about 10.0% by weight, based on said composition including detergent and builder ingredients, of a sodium salt of monochlorobenzene sulfonic acid (sodium p-chloro-benzene sulfonate) in such a manner as to produce ultimately a particulate solid detergent product, each particle of which contains the active detergent sulfonate, the corresponding inorganic builder or builders, and sodium monochlorobenzene sulfonate. The incorporation of these caking preventives into an alkyl benzene sulfonate detergent composition, particularly into one containing from about 20 to about 60 parts by weight of the active sulfonate in each 100 parts thereof, is observed to reduce markedly the extent of caking. At the same time, the detergency, wetting power and foaming ability of the composition are unaffected by the presence of the small amount of the caking preventive.

Addition of the caking preventive to the detergent composition may be carried out in a number of ways. One procedure is to add the parent compound of the sulfonate preventive, i. e., cholorbenzene, to the alkyl benzene material prior to its sulfonation and further conversion to sulfonate detergent. In this case the chlorobenzene is converted to the corresponding sodium chlorobenzene sulfonate simultaneously with the conversion of the C_9–C_{18} alkyl benzene to sodium alkyl benzene sulfonate detergent.

In another procedure chlorobenzene may be sulfonated separately and then admixed to the alkyl benzene prior to its sulfonation and conversion to the ultimate detergent sulfonate. Also, the sulfonated chlorobenzene may be added to the mixture of sulfonated C_9–C_{18} alkyl benzene and unreacted sulfuric acid prior to neutralization. Another procedure is to introduce chlorobenzene sulfonic acid or its sodium salt into the solution of caustic used for neutralizing the mixture of alkyl benzene sulfonic and unreacted sulfuric acid. Finally, one may also add sodium chlorobenzene sulfonate to the neutralized slurry of alkylbenzene sulfonic and sulfuric acid together with the desired builders prior to the drying treatment thereof, although the uniformity of particle composition in this instance is dependent upon the time and degree of slurry agitation. The presence in each 100 parts of the detergent composition of from 0 to about 89 parts by weight of sodium sulfate builder in no way impairs the beneficial caking-prevention effect of the addition of from about 1.0 to about 10.0% by weight of sodium monochlorobenzene sulfonate in accordance with our invention.

A series of tests was carried out in which the remarkable and unexpected anti-caking action of sodium chlorobenzene sulfonate preventive in the proportions indicated was firmly established. The following representative example of these tests illustrates the magnitude of the reduction in the amount of alkyl benzene sulfonate detergent caked, which can be achieved by the incorporation of from about 1.0 to about 10.0% by weight of the caking preventive of our invention.

In this example we employed a typical alkyl benzene sulfonate composition containing from about 40 to about 60 parts by weight of sodium C_{12}–C_{15} polypropylene benzene sulfonate as the active ingredient, the remainder to make up 100 parts being sodium sulfate builder. This composition has been prepared in accordance with the procedure followed in U. S. Patent No. 2,477,382 to A. H. Lewis, issued July 26, 1949.

In order to ascertain the extent of caking in detergent compositions containing the preventive agents in accordance with our invention, as compared with the caking

of the detergent compositions without such preventives, the following procedure was used: A sample of dried detergent formulation 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 1½" x 2½" 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 (¼") sieve. The sieve was then gently tipped and swirled to allow free-flowing materials 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 100 g. samples were exposed and screened.

Example

A mixture of 255 parts of C_{12}–C_{15} polypropylene benzene and 45 parts of chlorobenzene was sulfonated by adding slowly 361 parts of 20% fuming sulfuric acid while stirring. The acid was added to the mixture over a period of 45 minutes at a temperature of 85–90° F. At the end of this time the temperature was raised to 115° F., and stirring continued for 3 hours at that temperature. The acid mixture was then neutralized by pouring it slowly into a stirred solution of 250 parts of sodium hydroxide in 1000 parts of water while maintaining the temperature between 100–120° F. The ratio of sodium sulfate to sodium polypropylene benzene sulfonate was adjusted to equal 60:40 by the addition of 211 parts of sodium sulfate. The final slurry was then drum-dried. The dry detergent sample was tested for caking in accordance with the test procedure described hereinabove. At the completion of test, only 6% of the sample was observed to be caked. A control sample which did not contain any chlorobenzene sulfonate when subjected to the same test caked to the extent of 65% by weight.

The aforegiven representative example conclusively shows the improvement brought about by the introduction of sodium chlorobenzene sulfonate and into the detergent compositions having alkyl benzene sulfonates as their active detergent constituent.

Other inorganic salt builders than sodium sulfate, namely, various sodium phosphates such as trisodium phosphate, tetrasodium pyrophosphate and sodium tripolyphosphate as well as sodium carbonate, sodium bicarbonate and sodium chloride may equally well be employed as builders in the detergent compositions containing the effective amounts from about 10 to about 95 parts by weight of sodium alkyl benzene sulfonate detergent and 1 to 10% of the caking preventive, based on the combined weight of the detergent and builder ingredients.

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 containing sodium alkyl benzene sulfonate as the active ingredient.

We claim:

1. A particulate non-caking solid detergent composition in which the effective detergent ingredient is a sodium salt of a monoalkyl benzene sulfonic acid containing from 9 to 18 carbon atoms in the alkyl chain and present in each 100 parts by weight of said detergent composition in the amount from about 10 to about 95 parts by weight, the remainder of said 100 parts being essentially inorganic sodium salt builders, and from about 1.0 to about 10.0% by weight of sodium salt of monochlorobenzene sulfonic acid, based on the whole composition including detergent and builder ingredients.

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, and said inorganic builder is sodium sulfate.

4. The method of preventing caking in 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 from about 1.0 to about 10.0 percent by weight of sodium salt of monochlorobenzene sulfonic acid, based on the total solid content of said slurry, and spray-drying the slurry.

References Cited in the file of this patent

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