

UNITED STATES PATENT OFFICE

2,333,443

METHOD OF PREPARING DETERGENT COMPOSITIONS

Edward A. Robinson, Mount Lebanon, Pa., assignor to Diamond Alkali Company, Pittsburgh, Pa., a corporation of Delaware

No Drawing. Application December 23, 1940, Serial No. 371,406

5 Claims. (Cl. 252—135)

This invention relates to alkali metal silicate compositions and to a method of preparing them. The products of the invention are adapted particularly for detergent purposes such as in cleansing, washing, degreasing, and scouring operations.

One of the principal objects of the present invention has been to provide detergent compositions comprised of alkali metal silicates which are adapted to prevent the formation of lime and magnesium deposits and precipitates when they are used in unsoftened water. It has been understood in the past by those skilled in the art that the use of alkali metal phosphates is helpful in obtaining this result. For instance, a composition comprised of anhydrous tetrasodium pyrophosphate and sodium metasilicate mixed together in a dry state is adapted for the purpose, but such a mixture is slow to dissolve in water, because the pyrophosphate, in picking up water to form its decahydrate, cements the particles of metasilicate together; and of itself the anhydrous pyrophosphate is very slow to dissolve in water. For that reason, as well as others, when an alkali metal phosphate of this type is to be used commercially to supplement a silicate in a detergent bath, the practice has been to dissolve the two components separately or use the more soluble decahydrate of tetrasodium pyrophosphate in mechanical mixes. The first procedure is obviously tedious and becomes a substantial nuisance when detergent baths are made up day after day, as in typical industrial operations, while the use of the decahydrate is more expensive because of its higher cost per unit of P_2O_5 . These difficulties are also encountered when mechanical mixes containing anhydrous sodium tripolyphosphate are made. Moreover, in addition to slow solubility, the components of dry mechanical mixes tend to separate, through sifting, especially if one of them is not of the same particle size or degree of granulation or fineness as the other ingredients. It is the frequent experience that the composition at the top of the barrel of the material will be different from the composition at the bottom of the barrel from this cause.

The objective of the present invention has been to provide a dry and granular detergent composition comprising an alkali metal silicate and an anhydrous alkali metal phosphate which, in addition to providing the desired water softening as well as detergent properties, is readily soluble in water and, therefore easy to use in the industrial operations for which it is intended. Otherwise expressed, the objective has been to obtain

a material exhibiting all of the advantages which each component is capable of providing, but none of the disadvantages exhibited by a dry mixture of the same ingredients.

Briefly, the present invention is predicated upon the discovery that, while a dry mixture of an alkali metal silicate and an anhydrous alkali metal phosphate is a composition difficult to dissolve in water, the addition of the phosphate component while the alkali metal silicate is in a liquid condition provides a product which is of greatly improved solubility. The present invention contemplates a process for improving the water solubility of a silicate-anhydrous phosphate composition by incorporating the two with one another while the silicate is in a liquid state, that is, before the silicate is crystallized. In this process the two components are stirred together until a composition of uniform consistency is obtained, then the silicate is permitted to exert its crystallitic characteristic and harden. The resultant composition is adapted to be granulated or reduced to a marketable form in the usual manner as readily as the ordinary forms of crystallized alkali metal silicates.

The precise chemical explanations accounting for the fact that an admixture of the two components mechanically is not readily soluble in water while an admixture of the two components is freely soluble if made while one is in a liquid state, are not fully appreciated or explained by the present knowledge concerning the behavior of the chemicals. However, the fact is that the products made by the latter method dissolve in one-third to one-half the time required to dissolve dry-mix compositions, and are therefore especially suitable for industrial usage.

One of the characteristics of the alkali metal phosphates other than the orthophosphates is their tendency to revert to orthophosphates in the presence of strong alkalies and water at elevated temperatures. This reversion greatly reduces their water softening power. It has been discovered that under the manufacturing conditions of the present process, anhydrous tetrasodium pyrophosphate does not revert to orthophosphate to any marked degree when added to hot liquid alkali metal metasilicates or to more alkaline silicate materials. It has also been discovered that if at least 15% of sodium tripolyphosphate is used in the compositions, there is very little tendency for this compound to revert to pyrophosphate and orthophosphate. As the quantity of tripolyphosphate is increased the reversion is still further lessened, and products

containing about 30-40% sodium tripolyphosphate are stable and have the same water softening power as equivalent mechanical mixes. This stability remains undiminished for long periods after the compositions are prepared. The other complex phosphates of the kind heretofore available are much less stable under the conditions of this process and must ordinarily be dry mixed with alkali metal silicates to preserve their water softening power. Such mixes, of course, have the disadvantage of separation during shipment in barrels and other containers.

In the practice of the method of the present invention it is preferable that the silicate be in a hydrated form, that is, with approximately 2 to approximately 9 molecules water of hydration. Water may be present in excess of this amount, provided it does not detract from the ability of the silicate subsequently to crystallize.

The amount of phosphate which it is desirable to use for the prevention of the deposition of calcium and magnesium compounds is usually quite small, for example, in the neighborhood of 5 to 15%, sometimes less and sometimes considerably more, in instances where the composition is to be used for a special purpose.

The process of the present invention enables supplementary components besides phosphates to be incorporated, for example, sodium carbonate or caustic soda. When sodium carbonate is used it, like the phosphate, is preferably in the anhydrous condition.

The following examples illustrate typical compositions of the present invention:

Example A

	Per cent
Sodium metasilicate pentahydrate.....	80.00
Tetrasodium pyrophosphate anhydrous....	15.00
Sodium carbonate anhydrous.....	5.00

Example B

	Per cent
Sodium metasilicate pentahydrate.....	55.00
Tetrasodium pyrophosphate anhydrous....	40.00
Sodium carbonate anhydrous.....	5.00

This composition represents about the maximum amount of phosphate which is desirable to use.

Example C

	Per cent
Sodium silicate ($1.6\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot 5.5\text{H}_2\text{O}$).....	76.90
Tetrasodium pyrophosphate anhydrous....	12.60
Sodium carbonate anhydrous.....	10.50

Example D

	Per cent
Sodium orthosilicate ($2\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot 5.4\text{H}_2\text{O}$).....	85.00
Tetrasodium pyrophosphate anhydrous....	10.00
Sodium carbonate anhydrous.....	5.00

Example E

	Per cent
Sodium metasilicate pentahydrate.....	75.00
Sodium tripolyphosphate anhydrous.....	20.00
Sodium carbonate anhydrous.....	5.00

Example F

	Per cent
Sodium metasilicate pentahydrate.....	55.00
Sodium tripolyphosphate anhydrous.....	40.00
Sodium carbonate anhydrous.....	5.00

This composition represents about the maximum amount of phosphate which it is desirable to use. All percentages in the above examples are by weight.

All of these products exhibit pronounced ability to prevent the deposition of calcium and mag-

nesium compounds, especially when they are used in conjunction with fatty acid soaps. This characteristic is displayed even after the materials have been maintained in aqueous solution at elevated temperatures for substantial periods of time. In operations where soap is not used they prevent the formation of calcium, magnesium, and iron phosphates and carbonates.

In the practice of the method, the phosphate component and any supplementary ingredients, such as sodium carbonate or the like, are added to the silicate component while it is in the liquid state, in the proportions desired, and the components are then stirred together so that the mass becomes of uniform composition over a period of time during which the temperature is maintained to prevent crystallization of the mass from taking place. After the incorporating procedure the temperature of the mass is lowered and it crystallizes to a hard, grindable condition. For example, after the admixture, a given batch of the material, may be cast in pans and there permitted to cool, and the cakes so obtained can then be ground. In cases where the product is incorporated in the orthosilicates as disclosed in the McDaniel Patents Nos. 2,131,718 and 2,223,293, the final product is a flaked material similar to the products made by the McDaniel process.

The silicates which are adapted to be used in the practice of the present invention are the metasilicate, in which the ratio of Na_2O to SiO_2 is 1 to 1; the orthosilicate, in which the ratio is 2 to 1; (orthosilicates as disclosed in the McDaniel Patents Nos. 2,131,718 and 2,223,293 may be usefully employed); intermediate products in which the ratios may be 1.3 to 1; 1.5 to 1; 1.6 to 1 or thereabouts, and ratios up to $25\text{Na}_2\text{O}$ to 1SiO_2 . These products differ from one another as to the molecular quantity of sodium oxide which they contain and are therefore of differing alkaline activity. The alkali metal pyrophosphates and tripolyphosphates are preferred in the practice of the present invention because of their stability during the manufacturing process and their capacity to suppress the deposition or precipitation of calcium and magnesium compounds even at elevated temperatures for substantial periods of time in the presence of strong alkalies.

Besides the improved solubility exhibited by the compositions of the present invention, they are advantageous because of their improved water softening and detergent value. They are therefore especially adapted to a large number of industrial cleaning operations.

Having described my invention, I claim:

1. A method of obtaining a dry, granular composition of an alkali metal silicate and an alkali metal phosphate, which method comprises admixing an anhydrous alkali metal pyrophosphate with an alkali metal metasilicate containing from 2 to 9 molecules water of hydration while the said silicate is in a melted condition until a composition is obtained of uniform consistency and in such proportion that the alkali pyrophosphate constitutes about 5 to 40% by weight of said composition, then hardening said composition by permitting it to cool and then grinding said composition, said composition being characterized in that it is more readily soluble in water than a physical mixture of said components.

2. A method of making a dry, granular composition of sodium metasilicate and anhydrous tetrasodium pyrophosphate, which method comprises adding anhydrous tetrasodium pyrophos-

phate to sodium metasilicate pentahydrate while the sodium metasilicate is in a molten condition, admixing these components until a composition of uniform consistency is obtained, then permitting said composition to cool and harden, and then grinding the hardened material, the tetrasodium pyrophosphate component of said composition constituting from about 5 to 40% by weight of said composition, and said composition being characterized in that it is more readily soluble in water than a physical mixture of said components.

3. A method of obtaining a water-soluble composition of an alkali metal silicate and an alkali metal phosphate, which method comprises adding an anhydrous alkali metal pyrophosphate to a hydrated alkali metal metasilicate while said metasilicate is in a molten condition, admixing said components to obtain a composition of uniform consistency, permitting said composition to cool and harden, and then comminuting said composition, the anhydrous phosphate component of said composition constituting from about 5 to 40% by weight of the whole and said composition being characterized in that it is more readily soluble in water than a physical mixture of the said components.

4. A method of obtaining a water-soluble composition of an alkali metal silicate and an alkali

metal phosphate, which method comprises adding an anhydrous alkali metal pyrophosphate to a hydrated alkali metal metasilicate while said metasilicate is in a molten condition, admixing said components to obtain a composition of uniform consistency and permitting said composition to cool and harden, then grinding said composition, the anhydrous phosphate component of said composition constituting from about 5 to 15% by weight of the whole and said composition being characterized in that it is more readily soluble in water than a physical mixture of the said components.

5. A method of obtaining a water-soluble composition of an alkali metal silicate and an alkali metal phosphate, which method comprises admixing an alkali metal pyrophosphate with an alkali metal metasilicate pentahydrate while said metasilicate component is in a molten condition until a composition of uniform consistency is obtained and in such proportion that the anhydrous phosphate component constitutes about 5 to 15% by weight of said admixture, then permitting said composition to cool and harden and subsequently comminuting said composition, the said composition being characterized in that it is more readily soluble in water than a physical mixture of said components.

EDWARD A. ROBINSON.