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WATER SOFTENING DETERGENT AND
METHOD OF MAKING SAMEEdward A. Robinson, Pittsburgh, Pa., assignor to
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corporation of DelawareNo Drawing. Application November 13, 1946,
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This invention relates to water softening detergent compositions and to methods for making the same. It more particularly relates to a combination of alkali metal metasilicate and alkali metal polyphosphate having enhanced properties of stability and improved action in either soft or hard water.

The use of combinations of alkali metal metasilicates and alkali metal polyphosphates has heretofore been proposed for various detergent purposes. Insofar as such compositions containing polyphosphates having a higher ratio of Na_2O to P_2O_5 than 3:2 are concerned, such products have been found to be satisfactory both from the standpoint of effectiveness for the purpose intended and stability for periods of normal storage. However, combinations of alkali metal metasilicates and polyphosphates having a $\text{Na}_2\text{O} \cdot \text{P}_2\text{O}_5$ ratio of 3:2 or below have not been as satisfactory principally because even after relatively short periods of storage, a reaction occurs between the somewhat acid polyphosphate and the alkaline silicate, which reaction results in the release of free silica. As a consequence of this reaction, such products cake badly in storage. In addition, when such a composition is used as a detergent, the thus formed silica deposits films on objects that are washed and scales upon washing equipment. Since the purpose of using polyphosphates is to prevent the formation of films and scales of magnesium and calcium silicates in hard water, the usefulness of the phosphates is substantially defeated by this undesired reaction between the phosphate and silicate prior to its use.

While I have not observed that the art has attempted to solve this specific problem before, it has been proposed to spot-coat thin flakes of various alkali metal phosphates with alkaline materials in order to render the said phosphates compatible to soap in solution. Whatever may be the usefulness of this expedient, flakes so spot-coated have exposed uncoated surfaces and when such spot-coated materials are mixed with alkali metal metasilicates, the undesired reaction referred to above occurs with consequent caking on storage and cloudy solutions on use.

The present invention provides a simple and inexpensive solution to the problem described above whereby detergent compositions comprising alkali metal phosphates of sufficiently low alkali metal oxide content to act as an acid when combined with metasilicate, such as those polyphosphates having $\text{Na}_2\text{O} \cdot \text{P}_2\text{O}_5$ ratios of 3:2 or below, may suitably be compounded with alkali

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metal metasilicates to form a perfectly stable product which may be stored indefinitely without caking and without the production of free silica.

In general, the invention contemplates a product comprising alkali metal polyphosphate having a $\text{Na}_2\text{O} \cdot \text{P}_2\text{O}_5$ ratio of 3:2 or below, the particles of the phosphate having adhered thereto a layer of alkaline material, and the coated phosphate being admixed with a suitable proportion of alkali metal metasilicate. The method of this invention, in general, includes wetting such a phosphate with a small percentage of water admixing the moistened phosphate with a quantity of alkaline material, which is preferably anhydrous or nearly so and of which anhydrous sodium carbonate is an example, whereby the particles are substantially covered and the free water is taken up to give a dry mix, and then combining the so coated phosphate with a desired quantity of alkali metal metasilicate.

In the practice of this invention, flakes or granules of a chosen alkali metal polyphosphate having an alkali metal oxide ratio lower than 3:2, as stated above, are sprayed or otherwise suitably moistened with a small quantity of water. For this purpose I have found quantities of water up to 5% to be useful but amounts of the order of 1 to 2% are usually most satisfactory and are preferred. The spraying may take place in any suitable mixing device, it being preferred to employ mixers which will give rapid mixing and prevent caking of the sprayed material. The water may suitably be added in any convenient manner but I have found that a combination of a fine spray with substantial agitation produces the optimum effect. After about a minute or so of mixing with the water, the particles are suitably coated with a film of moisture. A quantity of powdered alkaline material, for example light soda ash, sufficient to coat the phosphate particles substantially completely, is then added while continuing the agitation. The particles quickly become completely coated with a substantial layer of alkaline material. The stirring is preferably not continued in this part of the process for more than a few minutes to avoid a tendency on the part of the alkaline material layer to rub off under continued agitation. Accordingly, after preferably not more than 5 minutes of agitation of the alkaline material coated phosphate, a suitable amount of alkali metal metasilicate is added while continuing agitation. After about 5 minutes more of mixing, the product is dry, free-flowing and granular and ready to be packed into shipping containers.

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Products treated in this manner have been found to have a storage life of more than 6 months and even when exposed in open packages in the relatively humid atmosphere of domestic kitchens or of dairy bottle washing establishments and the like, remain perfectly dry and stable and give clear limpid solutions when ultimately dissolved in water for use. Combinations of the phosphate here disclosed with metasilicate, lacking the treatment set forth herein, after having been stored for periods as short as 1 or 2 weeks cake badly and give definite silica flock precipitates when dissolved in water. Phosphate flakes which have been treated by the prior art spot-coating technique for a purpose not contemplated by this invention and then combined with a metasilicate are found to store reasonably well in substantially air and moisture tight containers. However, immediately upon the package being broken the exposure to the relatively humid air of the normal place of use of such materials results in substantial immediate reaction between the unprotected phosphate and the metasilicate whereby caking occurs particularly in materials not immediately used and subsequent use yields the characteristic silica precipitate in water solutions.

For a further understanding of the invention and further to teach those skilled in the art of the manner in which this invention may be practiced, the following specific examples of its practice are offered:

1. 60 lbs. of sodium tetraphosphate ($\text{Na}_6\text{P}_4\text{O}_{13}$), granules are placed in a ribbon mixer and sprayed with .9 lb. of water while mixing. After 2 minutes 12 lbs. of light soda ash (Na_2CO_3), are added and the mixing is continued for 1 to 2 minutes. Then 227 lbs. of sodium metasilicate (Na_2SiO_3), are added and the mixing is continued for 5 minutes. The resulting product, which is dry, free-flowing, granular, and substantially free from dusty particles, is then ready for packaging and is found to be stable to storage for indefinite periods.

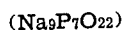
2. 90 lbs. of sodium decaphosphate



is placed in a ribbon mixer and sprayed with 1.35 lbs. of water while mixing. After 2 minutes 22.5 lbs. of light soda ash are added and the mixing is continued for 1 to 2 minutes. Then 186 lbs. of sodium metasilicate are added and the mixing is concluded at the end of 5 minutes. The product is comparable to that obtained in the process of that of Example 1 and is found to have indefinite storage life without chemical modification.

3. 24 lbs. of sodium hexametaphosphate ($\text{Na}_6\text{P}_6\text{O}_{22}$), are placed in a ribbon mixer and sprayed with .35 lb. of water while mixing. After 2 minutes, 6 lbs. of light soda ash are added and the mixing is continued for 1-2 minutes. Then 270 lbs. of sodium metasilicate are added and the mixing continued for 5 minutes. The product is then ready for packaging.

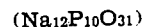
4. 120 lbs. of sodium septaphosphate



are placed in a ribbon mixer and sprayed with 1.8 lbs. of water while mixing. After 2 minutes 30 lbs. of powdered anhydrous tetrasodium pyrophosphate are added and the mixing continued for 1-2 minutes. Then 150 lbs. of sodium metasilicate are added and the mixing is continued for 5 minutes. The product is then ready for packaging.

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The present process is applicable to alkali metal polyphosphates generally where the ratio of alkali metal oxide to P_2O_5 is 3:2 or below. Included in this classification are alkali metal tetraphosphates, such as sodium tetraphosphate ($\text{Na}_6\text{P}_4\text{O}_{13}$), alkali metal septaphosphates, such as sodium septaphosphate ($\text{Na}_9\text{P}_7\text{O}_{22}$), alkali metal hexametaphosphates, such as sodium hexametaphosphate ($\text{Na}_6\text{P}_6\text{O}_{22}$), alkali metal hexapolyphosphates, such as sodium hexapolyphosphate ($\text{Na}_6\text{P}_6\text{O}_{19}$), and alkali metal decaphosphates, such as sodium decaphosphate



Where reference is made herein to $\text{Na}_2\text{O} \cdot \text{P}_2\text{O}_5$ ratios, polyphosphates of other alkali metals are intended to be disclosed. While it has been found preferable to employ anhydrous sodium carbonate suitably in the form of light soda ash as the alkaline material for coating the phosphate particles, any substantially anhydrous alkaline material compatible with the other two ingredients of the mixture and imparting no deleterious qualities to a detergent composition may be employed. By substantially anhydrous alkaline materials is meant generally those materials within the general disclosure herein whose affinity for water of crystallization is sufficiently unsatisfied so that they possess a substantial affinity for further water. Examples of materials in addition to that mentioned above which have been found useful in this connection are anhydrous alkali metal pyrophosphates, such as sodium pyrophosphate, anhydrous trialkali phosphates, such as trisodium phosphate, alkali carbonate monohydrates, such as sodium carbonate monohydrate, trialkali phosphate monohydrates, and the like. These materials may be used in quantities varying between about 10 and 25% of the weight of the dry phosphate. In addition, I may use any alkali metal metasilicate for admixture with the thus coated phosphate. The metasilicate may be suitably employed in amounts varying between 50 and 90% of the final detergent product, depending upon the ultimate use to which the detergent mixture is to be applied and the desired alkalinity of such mixture for such use.

While I have described in detail certain forms of my invention and certain specific examples of its practice, I do not wish to be understood as limiting myself to the use of such examples as I realize that changes within the scope of the invention are possible, and I further intend each step in the following claims to refer to all equivalent steps for accomplishing the same result in substantially the same or equivalent manner, it being my purpose to cover my invention broadly in whatever form its principle may be utilized.

What is claimed is:

1. The method of producing stable water softening detergent compositions of alkali metal metasilicate and alkali metal polyphosphate, wherein such polyphosphate has an alkali metal oxide content with relation to P_2O_5 of 3:2 or less, which includes the steps of moistening said polyphosphate, substantially completely coating said polyphosphate with a substantially anhydrous alkaline material chosen from the group of alkali metal carbonates and alkali metal non-polyphosphate phosphates, and combining said coated polyphosphate with alkali metal metasilicate.

2. The method of claim 1 wherein the sub-

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stantially anhydrous alkaline material is sodium carbonate.

3. The method of claim 1 wherein the substantially anhydrous alkaline material is tetrasodium pyrophosphate.

4. The method of producing stable water softening detergent compositions of alkali metal metasilicates and alkali metal polyphosphates having an alkali content sufficiently low to render them acid to alkali metal metasilicates, which includes the steps of substantially completely coating said alkali metal polyphosphate with a substantially anhydrous alkaline material chosen from the group of alkali metal carbonates and alkali metal non-polyphosphate phosphates, and combining said coated polyphosphate with alkali metal metasilicate.

5. The method of claim 4 wherein the substantially anhydrous alkaline material is sodium carbonate.

6. The method of claim 4 wherein the substantially anhydrous alkaline material is tetrasodium pyrophosphate.

7. The method of producing a stable water softening detergent composition of alkali metal metasilicate and alkali metal polyphosphate, wherein said phosphate has a ratio of alkali metal oxide to phosphorus pentoxide of 3:2 or less, which includes the steps of moistening said phosphate with less than 5% of water while stirring, substantially completely coating said phosphate with between 10% and 25% of powdered sodium carbonate based on the weight of the dry phosphate, and combining said coated phosphate with alkali metal metasilicate.

8. The method of producing a stable water softening detergent composition of alkali metal metasilicate and alkali metal polyphosphate, wherein said polyphosphate has a ratio of alkali metal oxide to phosphorus pentoxide of 3:2 or less, which includes the steps of moistening said polyphosphate with less than 5% of water while stirring, substantially completely coating said polyphosphate with between 10% and 25% of powdered tetrasodium pyrophosphate based on the weight of the dry polyphosphate, and combining said coated polyphosphate with alkali metal metasilicate.

9. The method of producing a stable water softening detergent composition, which includes the steps of moistening sodium tetraphosphate with not more than 5% of water, substantially completely coating said tetraphosphate with between 10% and 25% of sodium carbonate, and combining said coated tetraphosphate with sodium metasilicate.

10. The method of producing a stable water softening detergent composition, which includes the steps of moistening sodium tetraphosphate with not more than 5% of water, substantially completely coating said moistened tetraphosphate with between 10% and 25% of tetrasodium pyrophosphate, and combining said coated tetraphosphate with sodium metasilicate.

11. The method of producing a stable water softening detergent material, which includes the steps of moistening sodium decaphosphate with not more than 5% of water, substantially completely coating said sodium decaphosphate with between 10% and 25% of sodium carbonate, and combining said coated decaphosphate with sodium metasilicate.

12. The method of producing a stable water softening detergent material, which includes the steps of moistening sodium decaphosphate with

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not more than 5% of water, substantially completely coating said sodium decaphosphate with between 10% and 25% of tetrasodium pyrophosphate, and combining said coated decaphosphate with sodium metasilicate.

13. A stable water softening detergent material consisting essentially of a mixture of an alkali metal polyphosphate in which the ratio of alkali metal oxide to phosphorus pentoxide is 3:2 or less, said polyphosphate being coated substantially entirely with a material chosen from the group of alkali metal carbonates and alkali metal non-polyphosphate phosphates, and alkali metal metasilicate, said polyphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said material.

14. A stable water softening detergent material consisting essentially of a mixture of an alkali metal polyphosphate in which the ratio of alkali metal oxide to phosphorus pentoxide is 3:2 or less, said polyphosphate being coated substantially entirely with sodium carbonate, and alkali metal metasilicate, said polyphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said sodium carbonate.

15. A stable water softening detergent material consisting essentially of a mixture of an alkali metal polyphosphate in which the ratio of alkali metal oxide to phosphorus pentoxide is 3:2 or less, said polyphosphate being coated substantially entirely with tetrasodium pyrophosphate, and alkali metasilicate, said polyphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said tetrasodium pyrophosphate.

16. A stable water softening detergent composition consisting essentially of sodium tetraphosphate substantially completely coated with an alkaline material chosen from the group of alkali metal carbonates and alkali metal non-polyphosphate phosphates, and sodium metasilicate, said tetraphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said material.

17. A stable water softening detergent composition consisting essentially of sodium tetraphosphate substantially completely coated with sodium carbonate, and sodium metasilicate, said tetraphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said sodium carbonate.

18. A stable water softening detergent composition consisting essentially of sodium tetraphosphate substantially completely coated with tetrasodium pyrophosphate, and sodium metasilicate, said tetraphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said tetrasodium pyrophosphate.

19. A stable water softening detergent composition consisting essentially of sodium decaphosphate substantially completely coated with an alkaline material chosen from the group of alkali metal carbonates and alkali metal non-polyphosphate phosphates, and sodium metasilicate, said decaphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said material.

20. A stable water softening detergent composition consisting essentially of sodium decaphosphate substantially completely coated with sodium carbonate, and sodium metasilicate,

said decaphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said sodium carbonate.

21. A stable water softening detergent composition consisting essentially of sodium decaphosphate substantially completely coated with tetra-sodium pyrophosphate, and sodium metasilicate, said decaphosphate and said metasilicate being maintained substantially out of contact with each other in said mixture by said pyrophosphate.

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