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J. B. MARTIN

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PROCESS FOR PREPARING DETERGENT COMPOSITIONS

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Hydration of STPI in Aqueous Slurries

> 1.20 % H_2O

< 1.20 % H_2O

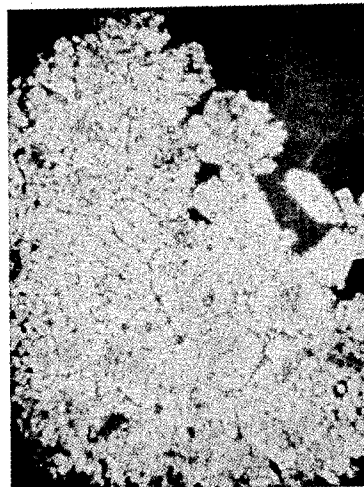


FIG. 1 A. 30 sec.

FIG. 1 B. 1-2 min.

Both fully crystallized as $STP \cdot 6H_2O$

Inventor

John Bruce Martin

By

Watson, Cole, Grindle & Watson
Attorneys

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2,961,410

PROCESS FOR PREPARING DETERGENT COMPOSITIONS

John Bruce Martin, Wyoming, Ohio, assignor to The Procter & Gamble Company, Cincinnati, Ohio, a corporation of Ohio

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This invention relates to a method for producing heat dried detergent compositions which contain sodium tripolyphosphate. More particularly, this invention relates to a process for producing heat dried detergent compositions which comprises incorporating anhydrous sodium tripolyphosphate in an aqueous detergent mixture and then heat drying the resultant slurry.

This application is a continuation-in-part of my co-pending application Serial No. 437,178, filed June 16, 1954, and now abandoned.

Heat dried sodium tripolyphosphate-containing detergent compositions are well known. Such compositions are commonly produced in the industry by first preparing a fluid detergent mixture, adding to this fluid mixture sodium tripolyphosphate and then heat drying the resultant slurry either by spray drying or by drying on a heated steel roll.

The sodium tripolyphosphate which is normally used in the production of such detergent compositions is known to exist in two forms—Form I, produced if the phosphate is calcined at high temperature, and Form II if the phosphate during its manufacture is calcined at lower temperature. Both of these forms of tripolyphosphate have been recognized from time to time as giving rise to certain undesirable characteristics in detergent compositions of which they comprise a substantial part. Thus, Form I sodium tripolyphosphate, in its commercially available form, when added to a fluid detergent mixture tends to hydrate very rapidly and form hard small lumps or agglomerates which persist through the entire processing and appear as hard sand-like particles in the final heat dried product. Form II sodium tripolyphosphate, on the other hand, hydrates rather slowly when added to a fluid detergent mixture, and unless the crutcher mix is aged as set forth in Hizer's U.S. Patent 2,622,068, has the objectionable tendency of producing final heat dried compositions which are sticky in nature and which lump and cake badly under even very slight pressure.

In a conventional method for producing heat dried tripolyphosphate-containing detergent compositions a fluid detergent mixture, as hereinafter defined, and commonly referred to in the industry as a crutcher mix, is first prepared. The sodium tripolyphosphate is then added to this aqueous detergent crutcher mix with thorough agitation and the resultant slurry is spray dried. It is in connection with this tripolyphosphate-containing crutcher mix that additional difficulties are experienced in that after the addition of the sodium tripolyphosphate the resultant slurry is rather viscous in nature and may be referred to as having a high consistency. This high consistency usually gives rise to difficulties in mixing, pumping and spraying. If an effort is made to lower the consistency, as by the addition of more water to the slurry, the amount of moisture which must be removed during the spray drying process is increased and this increased

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evaporative load will be reflected in costly decreased production with a given amount of heat input to the spray drying tower.

Moreover, depending upon the particular mixture of anhydrous tripolyphosphates being used, variable amounts of hydration to the hexahydrate occurs in the crutcher mix and this tends to cause variations in consistency, in the sprayed particle sizes, and in fluctuating conditions generally in the spray-drying tower as regards temperature, evaporative load etc., thus causing undesirable non-uniform operation of the process and non-uniformity in the physical characteristics of the spray-dried product.

It is an object of this invention to provide a method for the manufacture of detergent compositions free from sand-like particles and stickiness which tend to occur using Form I sodium tripolyphosphates.

It is a further object of this invention to provide a method for the manufacture of detergent compositions wherein complete hydration of Form I sodium tripolyphosphate in aqueous fluid detergent crutcher mixtures is obtained without the fluid detergent mass becoming highly viscous.

A still further object of this invention is to utilize processing advantages stemming from the lower and more uniform consistency of the completely hydrated Form I sodium tripolyphosphate containing fluid detergent crutcher mix.

I have found that these objects may be achieved by taking steps to make sure that Form I sodium tripolyphosphate added to the aqueous detergent crutcher mix during manufacture of the detergent compositions contains less than 0.1% moisture, and preferably less than 0.05% moisture other than water of constitution, as contrasted with the much higher and variable moisture contents normally found in supposedly anhydrous tripolyphosphates of commerce.

Wherever herein moisture content values are given for specific samples of tripolyphosphate, the moisture was determined by the loss on ignition method. Although this method of determining moisture would include any water of constitution in the phosphate, i.e. water that is chemically combined, such as in the sodium hydrogen phosphates, it is to be understood that the water of constitution has a negligible effect on the consistency of phosphate-containing crutcher mixes.

In the accompanying illustrations, Figures 1A and 1B are photomicrographs of the hexahydrate crystal structure derived from slightly hydrous Form I sodium tripolyphosphate and substantially anhydrous Form I sodium tripolyphosphate respectively.

It is to be understood that the sodium tripolyphosphate referred to herein and in the claims is the normal salt, commonly identified in the art by the empirical formula $\text{Na}_5\text{P}_3\text{O}_{10}$.

Also, wherever the term "detergent" appears herein it is meant to designate organic detergents including:

(1) Ordinary alkali metal soaps such as the sodium and potassium salts of the higher fatty acids of naturally occurring plant or animal esters (e.g. palm oil, coconut oil, babassu oil, soybean oil, castor oil, tallow, whale and fish oils, grease and lard, and mixtures thereof) or of synthetically produced fatty acids (e.g. by the oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process), of resin acid (e.g. rosin and those resin acids in tall oil) and/or of naphthenic acids. Sodium and potassium soaps can be made by direct saponification of the fats and oils or by the neutralization of the free fatty acids which are prepared in a separate manufacturing process.

(2) Synthetic organic detergents characterized by their high solubility in water, their resistance to precipitation by the constituents of hard water and their surface active and effective detergent properties, including:

(a) Anionic synthetic detergents (excluding true soaps): This class of synthetic detergents can be broadly described as the water-soluble salts, particularly the alkali metal salts, of organic sulfuric reaction products having in the molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals. Important examples of the synthetic detergents which form a part of the preferred compositions of the present invention are the sodium or potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols produced by reducing the glycerides of tallow or coconut oil; sodium or potassium alkyl benzene sulfonates, in which the alkyl group contains from about 9 to about 15 carbon atoms, especially those of the types described in United States Letters Patent Numbers 2,220,099 and 2,477,383; sodium alkyl glyceryl ether sulfonates, especially those ethers of the higher alcohols derived from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfates and sulfonates; sodium or potassium salts of sulfuric acid esters of the reaction product of one mole of a higher fatty alcohol (e.g. tallow or coconut oil alcohols) and about three moles of ethylene oxide; sodium or potassium salts of alkyl phenol ethylene oxide ether sulfate with about four units of ethylene oxide per molecule and in which the alkyl radicals contain about 9 carbon atoms; the reaction product of fatty acids esterified with isethionic acid and neutralized with sodium hydroxide where, for example, the fatty acids are derived from coconut oil; sodium or potassium salts of fatty acid amide of a methyl taurine in which the fatty acids, for example, are derived from coconut oil; and others known in the art, a number being specifically set forth in United States Letters Patent Numbers 2,486,921, 2,486,922 and 2,396,278.

(b) Nonionic synthetic detergents: This class of synthetic detergents may be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. The length of the hydrophilic or polyoxyalkylene radical which is condensed with any particular hydrophobic group can be readily adjusted to yield a water-soluble compound having the desired degree of balance between hydrophilic and hydrophobic elements.

For example, a well known class of nonionic synthetic detergents is made available on the market under the trade name of "Pluronic." These compounds are formed by condensing ethylene oxide with an hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The hydrophobic portion of the molecule which, of course, exhibits water insolubility has a molecular weight of from about 1500 to 1800. The addition of polyoxyethylene radicals to this hydrophobic portion tends to increase the water solubility of the molecule as a whole and the liquid character of the products is retained up to the point where polyoxyethylene content is about 50% of the total weight of the condensation product.

Other suitable nonionic synthetic detergents include:

(i) The polyethylene oxide condensates of alkyl phenols, e.g. the condensation products of alkyl phenols having an alkyl group containing from about 6 to 12 carbon atoms in either a straight chain or branched chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to 10 to 25 moles of ethylene oxide per mole of alkyl phenol. The alkyl substituent in such compounds may be derived from polymerized propylene, diisobutylene, octane, or nonane, for example.

(ii) Those derived from the condensation of ethylene oxide with the product resulting from the reaction of propylene oxide and ethylene diamine—products which may be varied in composition depending upon the balance between hydrophobic and hydrophilic elements which is desired. For example, compounds containing from about 40% to about 80% polyoxyethylene by weight and having a molecular weight of from about 5000 to about 11,000, resulting from the reaction of ethylene oxide groups with a hydrophobic base constituted of the reaction product of ethylene diamine and excess propylene oxide, said base having a molecular weight of the order of 2500 to 3000, are satisfactory.

(iii) The condensation product of aliphatic alcohols having from 8 to 18 carbon atoms, in either straight chain or branched chain configuration, with ethylene oxide, e.g. a coconut alcohol ethylene oxide condensate having from 10 to 30 moles of ethylene oxide per mole of coconut alcohol, the coconut alcohol fraction having from 10 to 14 carbon atoms.

It is to be understood that the term detergent mixture and/or detergent mix as used herein includes, in addition to one or more of the above detergents, alkaline "builders" or detergent improving agents such as the sodium and other alkaline carbonates, silicates, phosphates and borates and/or such compounds as the perborates, persulfates and percarbonates; also, organic builders and perfumes, coloring matter, preservatives and other substances commonly found in detergent compositions.

Commercially available Form I and Form II sodium tripolyphosphates, or mixtures thereof, as a rule average above 0.2% in moisture content as received, and furthermore tend to be variable in moisture from outside to inside the mass. It is to be understood of course that this moisture content is not inherent in the sodium tripolyphosphate as manufactured and that as obtained from the calcining kiln the tripolyphosphate is completely anhydrous. The moisture content is picked up, usually from the atmosphere, during subsequent handling of the anhydrous product. It is to be appreciated that, depending upon the manner by which the moisture in the tripolyphosphate product is picked up, the sodium tripolyphosphates available on the market vary widely as to the amount of moisture which they contain and moisture contents as high as 1% or greater are not unusual.

It is the presence of this water in excess of 0.1% which imparts to the sodium tripolyphosphate hydration properties which give rise to the aforementioned undesirable characteristics in detergent compositions prepared with them. I have made the totally unexpected discovery that if this water is removed, or if its pickup by the sodium tripolyphosphate is prevented, the hydration characteristics of the tripolyphosphate are radically changed.

A difference in hydration characteristics between a slightly hydrous sample of Form I sodium tripolyphosphate (about 1.2% water) and a substantially completely anhydrous Form I sample (0.02% water) is evident from a comparison of Figure 1A, a photomicrograph of slightly hydrous Form I sodium tripolyphosphate fully crystallized as the hexahydrate, and Figure 1B, a photomicrograph of substantially completely anhydrous Form I sodium tripolyphosphate also fully crystallized as the hexahydrate. (It is to be appreciated with regard to the slightly hydrous Form I sample that the hydration characteristics shown are apparent in a sample having only a fraction of the water content of the sample identified here.)

The technique used in obtaining these photomicrographs was as follows:

A small sample of the dry sodium tripolyphosphate powder was placed between a microscope slide and cover glass. The layer was thin enough so that some of the individual particles could be seen in the dry form, but not so thin that all of the sample dissolved when a drop

of water was allowed to flow under the cover glass by capillary action. Agitation of the slurry was carefully avoided. Microscopic examination was made at a magnification of 85 diameters with crossed nicols.

As can be seen in Figure 1A, the slightly hydrous Form I sodium tripolyphosphate converted within a few seconds after the addition of the water to sub-microscopic crystals which appear as large opaque masses. (Because of the rapid hydration here the observer, in order to see the changes which occur on hydration, must be observing the sample as the water is admitted.) In contrast, when water was added to the substantially anhydrous Form I sodium tripolyphosphate, it dissolved appreciably in the first few seconds, then a few hexahydrate crystallization centers developed and rapidly grew into a relatively few large spherulites as shown in Figure 1B.

The consistency of the sodium tripolyphosphate-containing crutcher mix slurry is believed to be dependent upon the size of the tripolyphosphate hexahydrate crystals present in the slurry and the larger the size of these crystals the lower the consistency of the slurry. Thus, the use of the substantially anhydrous sodium tripolyphosphate which results in the formation of larger hexahydrate crystals as hereinbefore described, gives a lower tripolyphosphate-containing crutcher mix consistency, and the mixing, pumping, and spraying advantages which are inherent in such a more fluid slurry.

Heretofore, because of the high consistency imparted to the crutcher mix by the addition of slightly hydrous Form I sodium tripolyphosphate, care had to be taken to control the amount of Form I present in the mixture of Form I and Form II sodium tripolyphosphate which was added to the crutcher mix. It was found that in a tripolyphosphate mixture, the presence of as little as 5% Form I is detectable by the behavior characteristics of the tripolyphosphate when it is added to a detergent crutcher mix; that with 10% or more of Form I tripolyphosphate the ascendancy of the Form I behavior characteristics is quite evident; and that when about 25% or more Form I is present the behavior characteristics associated with Form I tripolyphosphate dominate. It was also found that increasingly thicker crutcher mix slurries resulted from the addition of sodium tripolyphosphate in which increasingly greater amounts of Form I tripolyphosphate were present. With the process of my invention, however, the amount of Form I tripolyphosphate present in the sodium tripolyphosphate added to the fluid detergent crutcher mix is not a consideration and the sodium tripolyphosphate may, if desired, be 100% Form I. I desire to include within the scope of my invention only those mixtures of sodium tripolyphosphate containing from about 10% to 100% Form I tripolyphosphate and conversely from 90% to 0% Form II tripolyphosphate, and more preferably those mixtures of sodium tripolyphosphate containing at least 50% Form I tripolyphosphate.

It is to be noted that the percentages of Form I and Form II tripolyphosphates wherever they appear herein or in the claims are expressed on the basis of the sodium tripolyphosphates present in the mixture of phosphates employed, commercial sodium tripolyphosphates containing small percentages of other phosphates, such as ortho- or pyro-phosphate, which are incident to the sodium tripolyphosphate manufacturing process.

I have further found that with the process of my invention the anhydrous Form I sodium tripolyphosphate becomes completely hydrated, yet the crutcher mix slurry retains its thin consistency. This has a particular advantage in commercial operation in that it allows the preparation of crutcher mix slurries with less water than is necessary with slightly hydrous Form I sodium tripolyphosphate, thus reducing the evaporative load in the heat drying operation. Moreover, since with my invention the Form I sodium tripolyphosphate becomes completely

hydrated in the crutcher, it makes possible the production of a product having a higher moisture content without the caking and lumping disadvantages normally attendant upon such high moisture products. Also, the normally thin consistency of the crutcher mix slurries prepared according to my invention allows for more uniform operation in the process as a whole and thus affords the additional economic advantages identified with such uniform operation.

In the following examples all consistency measurements given are expressed in arbitrary units as determined with a commercially available instrument called the Brabender Plastograph which records continuously the torsion moment required to rotate a pair of agitators in the viscous mixture being examined.

It is to be appreciated in connection with the examples, and with the described detergent manufacturing process in general, that when the sodium tripolyphosphate is rapidly added to an aqueous detergent crutcher mix there is a rapid and large increase in the consistency of the mix because of the then poor distribution of the phosphate in the mix. The high consistency incident to the tripolyphosphate addition is only temporary however and the consistency drops off as the tripolyphosphate is evenly distributed throughout the crutcher mix. Furthermore, it is common practice when making a tripolyphosphate-containing detergent to add sodium silicate to the crutcher mix for several beneficial purposes including inhibition of aluminum corrosion by the finished detergent product. This silicate is normally added, because of pH considerations and for convenience, shortly after the tripolyphosphate has been added to the detergent crutcher mix and the addition therefore frequently occurs when the consistency of the crutcher mix slurry is at a maximum level as a result of the tripolyphosphate addition. For these reasons I have chosen to use the silicate addition as a time base and in the following examples the consistency for any given crutcher mix slurry is expressed for minutes after the addition of the silicate.

It is to be understood, however, that the above described mode of expression is merely for convenience in presenting the examples and that some other time basis could just as well have been chosen, the addition of silicate with the process of my invention being optional.

The following specific examples are to be considered as illustrative only and changes and modifications therein as would normally occur to those skilled in the art are contemplated.

Example 1.—The effect of moisture pickup by sodium tripolyphosphate on the consistency of a detergent crutcher mix was determined as follows:

A sample of sodium tripolyphosphate, containing about 75% Form I tripolyphosphate, was shipped in two containers, portion A sealed in glass, and portion B in a fiber drum. Upon arrival at their destination, several days later, the moisture content of the samples was determined by the loss of ignition. These were respectively 0.01% for portion A and 0.23% for portion B. A third sample, C, of the tripolyphosphate was prepared by igniting a part of portion B to 0.00% water.

371 parts by weight of each of these samples of tripolyphosphate was then added, with constant stirring, to 535 parts of a fluid detergent mixture containing 9.1% sodium alkyl benzene sulfonate detergent (the alkyl radical averaging about 12 carbon atoms and being derived from polypropylene), 15.5% sodium alkyl sulfate detergent (the alkyl groups being derived from higher alcohols produced by the reduction of tallow and being about 65% C₁₈, 33% C₁₆ and 2% C₁₄), 59.1% water and 15.5% sodium sulfate, the remainder of the mixture comprising unsulfated material. 93 parts of sodium silicate was then added to this mixture and the consistency of the resultant slurries was determined with the Brabender Plastograph with the following results.

Minutes after Silicate Addition	Consistency of Slurry		
	STP "A"	STP "B"	STP "C"
5.....	70	450	90
10.....	75	435	90
20.....	75	400	95
60.....	85	400	110

STP=sodium tripolyphosphate.

It may be seen from the above that the moisture content of the sodium tripolyphosphate used in preparing a crutcher mix slurry has a very marked effect on the consistency of the crutcher mix slurry. The ability of anhydrous or substantially anhydrous sodium tripolyphosphate to form and maintain crutcher mix slurries of thin consistency is evident.

Example 2.—Four crutcher mix slurries each utilizing a different sample of sodium tripolyphosphate containing about 50% Form I tripolyphosphate were prepared by adding the tripolyphosphate to an aqueous detergent mix comprising 9.1% sodium alkyl benzene sulfonate detergent (the alkyl radical averaging about 12 carbon atoms and being derived from polypropylene), 15.1% sodium alkyl sulfate detergent (the alkyl groups being derived from higher alcohols produced by the reduction of tallow and being about 65% C₁₈, 33% C₁₆ and 2% C₁₄), 59.1% water and 15.5% sodium sulfate, the remainder of the mixture comprising unsulfated material. The tripolyphosphates, which were added in the amount of 371 parts to 535 parts of the aqueous detergent mix, were identified as follows.

Tripolyphosphate	Percent H ₂ O
A.....	0.04.
B.....	0.67 (STP "A" partially hydrated by water vapor at 90° F. and 80% R.H.).
C.....	0.01 (STP "A" ignited at 400° C. for 2 hours).
D.....	0.00 (STP "A" ignited at 550° C. for 2 hours).

¹ The ignition very probably converted the sample wholly to Form I.

93 parts of sodium silicate was added to this mixture and the consistency of the resultant slurries was determined on the Brabender Plastograph with the following results. All consistency values given in the table below are at 100% hydration of the tripolyphosphate in the slurry.

Minutes after Silicate Addition	Consistency of Slurry			
	STP "A"	STP "B"	STP "C"	STP "D"
10.....	190	460	85	75
20.....	230	435	85	85
30.....	250	440	95	95

STP=sodium tripolyphosphate.

Again it may be seen from the above that the anhydrous or substantially anhydrous tripolyphosphates promote the formation and maintenance of consistently thin crutcher mix slurries.

Example 3.—Two samples of 100% Form I sodium tripolyphosphate, Sample A containing 1.28% moisture (by loss on ignition), and Sample B containing 0.00% moisture (prepared by igniting a portion of Sample A at 550° C. for 2 hours), were used to prepare crutcher mix slurries with the aqueous detergent mixes of Example 2. The tripolyphosphates were added in the amount of 371 parts, by weight, to 535 parts, by weight, of the detergent mix.

93 parts of sodium silicate was added to the above mixture and the consistency of the resultant slurries was determined with the Brabender Plastograph. The consistency data is tabulated below.

Minutes After Silicate Addition	Consistency of Slurry	
	STP "A"	STP "B"
5.....	600	170
10.....	450	180
20.....	435	180
30.....	430	185

STP=sodium tripolyphosphate.

Here it may be seen that even with 100% Form I sodium tripolyphosphate, thin crutcher mix slurries may be obtained if the tripolyphosphate is anhydrous.

In the preceding examples detergents of each of the various types enumerated hereinbefore can be substituted for the detergents specifically mentioned in the examples with comparable results, it being understood that the operation can be readily adjusted to accommodate any minor changes in crutcher mix consistency resulting from the use of different detergents. Also it is to be appreciated that tripolyphosphates of potassium and other alkali metals may be used in conjunction with the sodium tripolyphosphates specifically mentioned, and that the sodium tripolyphosphate-containing detergent compositions produced in accordance with my process may be in flake, thread, powder or spray dried granular or powdery form.

Following are some additional examples of the practice of my invention in which parts shown are by weight.

Example 4.—50 parts of completely anhydrous 100% Form I sodium tripolyphosphate is added to 50 parts of an aqueous detergent mix containing about 40% sodium alkyl sulfate detergent (the alkyl groups being derived from the higher alcohols obtained by the reduction of coconut oil), 50% water and 10% sodium sulfate. The resultant mixture, after spray drying, is an excellent all-purpose detergent.

If desired, the alkyl sulfate detergents in the foregoing example are replaceable by like amounts of other detergent agents with comparable results. Thus, sodium alkyl glyceryl ether sulfonate (the alkyl groups of which are derived from a middle cut coconut fatty alcohol mixture having the composition: 10 carbon atoms—2%; 12 carbon atoms—65%; 14 carbon atoms—23%; and 16 carbon atoms—9%), the sodium salt of sulfated monoglyceride of hydrogenated coconut oil fatty acids, the sodium salt of the sulfuric acid esters of the reaction product of one mole of the higher fatty alcohols derived from the reduction of coconut oil and three moles of ethylene oxide, the reaction product of coconut oil fatty acids esterified with isethionic acid and neutralized with sodium hydroxide, and the sodium salt of oleic acid amide of N-methyl taurine, all find ready application in the foregoing formulation and provide excellent general-purpose detergents.

Example 5.—40 parts of completely anhydrous Form I sodium tripolyphosphate is added to 60 parts of an aqueous detergent mix containing as a detergent about 25% Pluronic F68 (the condensation product of ethylene oxide with a polyoxypropylene base, the said polyoxypropylene base having a molecular weight of about 1500 to about 1800, and the said condensation product having a molecular weight of about 8000), 15% sodium sulfate, 5% sodium silicate and 55% water. The resultant mixture, after spray drying, is an excellent detergent agent, particularly in uses where excessive sudsing is not desirable.

Other nonionic synthetic detergents, such as, Igepal CO-720 (a nonyl phenol ethylene oxide condensate having on the average 15 moles of ethylene oxide per mole of nonyl phenol), the alkyl ethylene oxide condensate wherein the alkyl radical is derived from alcohols produced by the reduction of coconut oil fatty acids having from 10 to 14 carbon atoms, the said condensate having

an average molecular weight of about 800, and Tetronic 707, (a commercially available nonionic condensation product identified by a molecular weight of about 11,000 and obtained from the condensation of ethylene oxide with a hydrophobic base derived from the condensation of propylene oxide with ethylene diamine, said base having a molecular weight of the order of 2500 to 3000), are readily substituted for the Pluronic F68 in the foregoing example in like amounts with comparable results.

Example 6.—33 parts of completely anhydrous Form I sodium tripolyphosphate is added to 67 parts of an aqueous detergent mixture containing about 15% sodium alkyl benzene sulfonate detergent (the alkyl radical averaging about 12 carbon atoms and being derived from polypropylene), about 37% of a sodium soap obtained by the saponification of tallow, about 5% sodium sulfate and about 43% water. The resultant mixture is spray dried and gives an excellent detergent agent having a relatively low sudsing level.

The substitution, in like amounts, of a sodium soap obtained by the saponification of coconut oil for the sodium soap obtained by the saponification of tallow in the above example results in a detergent composition having comparable performance characteristics. Also, with any of the foregoing compositions drying is readily accomplished on a heated steel roll with comparable results.

Example 7.—25 parts of completely anhydrous Form I sodium tripolyphosphate is added to 75 parts of an aqueous solution of 50% soap (normal sodium kettle soap obtained from the saponification of a mixture of 80% tallow and 20% coconut oil), 45% water and 5% sodium silicate. The mixture, after spray drying, is an excellent detergent having balanced sudsing and cleansing properties.

In the above example, the substitution, in like amounts, of the normal sodium kettle soap obtained from the saponification of a mixture of 60% palm oil, 20% babassu oil and 20% hydrogenated marine oils, for the normal sodium kettle soap specified gives comparable results. The substitution of normal potassium kettle soap for the sodium kettle soap in any of the foregoing compositions of this example is also readily accomplished and results in detergent compositions having comparable performance characteristics.

The utility of the invention is particularly attractive in the manufacture of detergent compositions wherein the ratio of sodium tripolyphosphate to organic detergent ranges from about 1:1 to 5:1 and the weight of water used in making the fluid mixture to be spray dried does not exceed the weight of the tripolyphosphate used.

It may be pointed out that in the manufacture of detergents in particulate form which contain substantial amounts of sodium tripolyphosphates, little of the tripolyphosphate is in solution during the processing of the crutcher mix, most of it being suspended as a slurry. Consequently, in order to insure the desired rapid solution of the final detergent product in water, the particle size of the sodium tripolyphosphate should be regulated and should, in general, be no larger than that desired in the final product.

Having thus described my invention, I claim:

1. In the process of producing a heat dried detergent composition containing substantial amounts of sodium tripolyphosphate and being free from a tendency to be sticky in nature and contain sand-like particles, the steps of incorporating a substantially completely anhydrous sodium tripolyphosphate containing from about 10% to 100% of Form I sodium tripolyphosphate and containing not more than 0.1% water, other than water of constitution, and insufficient, upon hydration and crystallization of the tripolyphosphate, to promote development of a preponderance of submicroscopic hexahydrate crystals, in a fluid detergent mixture, containing essentially (a) an organic detergent selected from the group consisting of sodium and potassium salts of higher molecular weight carboxylic

acids, water soluble salts of organic sulfuric reaction products having in their molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals, and water soluble nonionic synthetic detergent condensation products of alkylene oxide groups which are hydrophilic in nature with an organic hydrophobic compound selected from the group consisting of aliphatic compounds and alkyl aromatic compounds, the ratio of sodium tripolyphosphate to organic detergent ranging from about 1:1 to 5:1 and (b) more than sufficient water to hydrate the tripolyphosphate added and sufficient to provide and maintain a fluid detergent mixture, whereby 100% hydration of the Form I sodium tripolyphosphate is obtained and the fluid detergent mixture remains fluid.

2. The process of claim 1 wherein the sodium tripolyphosphate contains not more than 0.05% water, other than water of constitution.

3. The process of claim 1 wherein the sodium tripolyphosphate added contains at least 50% Form I sodium tripolyphosphate and less than 50% Form II sodium tripolyphosphate.

4. The process of claim 1 wherein the sodium tripolyphosphate is wholly anhydrous Form I tripolyphosphate.

5. The process of claim 1 wherein the weight of the water used in making the fluid mixture does not exceed the weight of said tripolyphosphate.

6. In the process of producing a heat dried detergent composition containing substantial amounts of sodium tripolyphosphate and being free from a tendency to be sticky in nature and contain sand-like particles, the steps which comprise, (1) drying a commercially available sodium tripolyphosphate containing from about 10% to 100% Form I sodium tripolyphosphate to a uniformly substantially completely anhydrous condition wherein not more than 0.1% water, other than water of constitution, is present, and (2) incorporating the thus dried tripolyphosphate in a fluid detergent mixture containing essentially (a) an organic detergent selected from the group consisting of sodium and potassium salts of higher molecular weight carboxylic acids, water soluble salts of organic sulfuric reaction products having in their molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals, and water soluble nonionic synthetic detergent condensation products of alkylene oxide groups which are hydrophilic in nature with an organic hydrophobic compound selected from the group consisting of aliphatic compounds and alkyl aromatic compounds, the ratio of sodium tripolyphosphate to organic detergent ranging from about 1:1 to 5:1 and (b) more than sufficient water to hydrate the tripolyphosphate added and sufficient to provide and maintain a fluid detergent mixture, whereby 100% hydration of the Form I sodium tripolyphosphate is obtained and the fluid detergent mixture remains fluid.

7. In the process of producing a heat dried detergent composition containing substantial amounts of sodium tripolyphosphate and being free from a tendency to be sticky in nature and contain sand-like particles, the steps which comprise, (1) maintaining sodium tripolyphosphate, containing from about 10% to 100% Form I sodium tripolyphosphate, in its initial completely anhydrous state wherein not more than 0.1% water, other than the water of constitution, is present, and (2) incorporating the said tripolyphosphate in a fluid detergent mixture containing essentially (a) an organic detergent selected from the group consisting of sodium and potassium salts of higher molecular weight carboxylic acids, water soluble salts of organic sulfuric reaction products having in their molecular structure an alkyl radical containing from about 8 to about 22 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals, and water soluble nonionic synthetic detergent

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condensation products of alkylene oxide groups which are hydrophilic in nature with an organic hydrophobic compound selected from the group consisting of aliphatic compounds and alkyl aromatic compounds, the ratio of sodium tripolyphosphate to organic detergent ranging from about 1:1 to 5:1 and (b) more than sufficient water to hydrate the tripolyphosphate added and sufficient to provide and maintain a fluid detergent mixture, whereby 100% hydration of the Form I sodium tripolyphosphate is obtained and the fluid detergent mixture remains fluid. 10

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UNITED STATES PATENT OFFICE
CERTIFICATION OF CORRECTION

Patent No. 2,961,410

November 22, 1960

John Bruce Martin

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 5, line 16, for "relatively fed" read -- relatively few --; column 6, line 67, for "15.5%" read -- 15.1% --; column 7, line 56, in the table, fifth column thereof, last line, for "95" read -- 85 --; column 8, line 45, for "65%" read -- 66% --.

Signed and sealed this 23rd day of May 1961.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents