

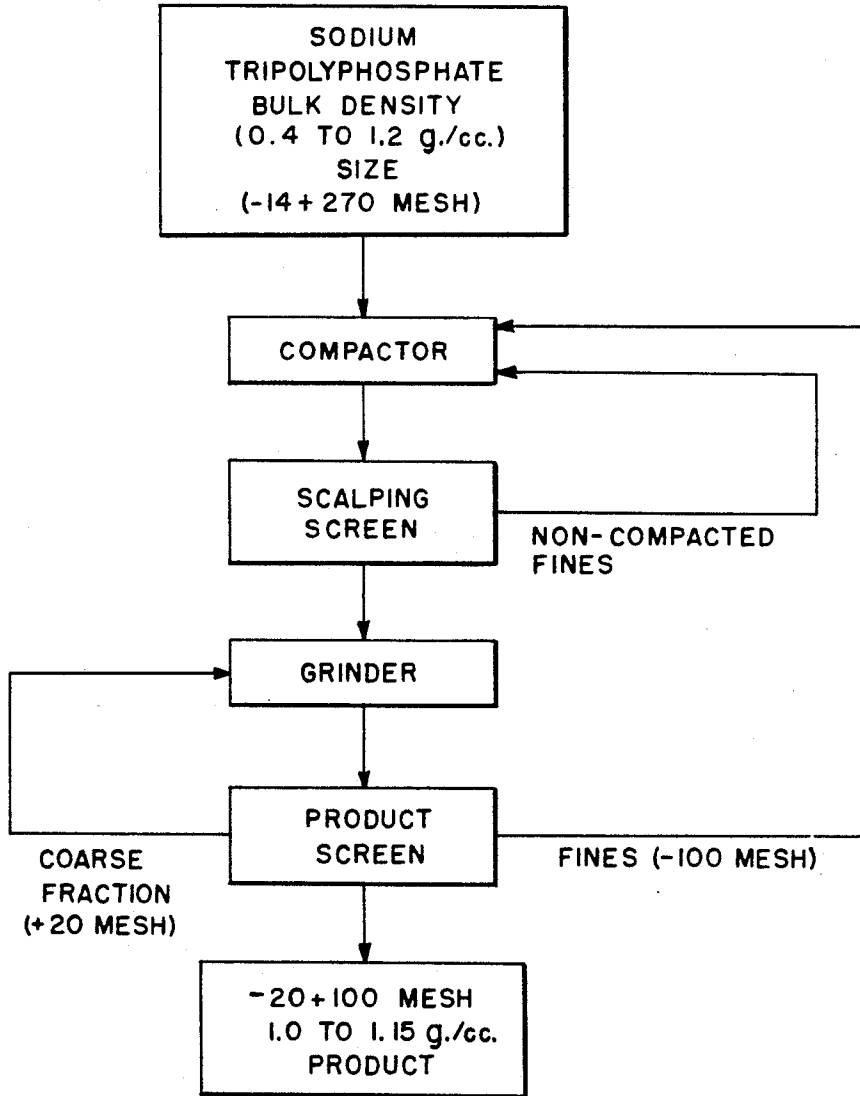
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SODIUM TRIPOLYPHOSPHATE GRANULAR PRODUCT

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**SODIUM TRIPOLYPHOSPHATE
GRANULAR PRODUCT**

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The present process is concerned with the formation of a granular sodium tripolyphosphate having a short hydration time.

In the formulation of modern detergents, sodium tripolyphosphate has become widespread for use as a phosphate "builder." Such formulation typically contains a surfactant such as one or more of the alkyl aryl sulfonates or alkyl sulfates, an anti-corrosion agent such as sodium silicate, an anti-redeposition agent such as sodium carboxymethylcellulose and other ingredients such as optical brighteners, dyes, perfumes, etc. While some formulators prefer a low bulk density sodium tripolyphosphate to offer the consumer the largest possible container per unit weight of detergent, many prefer to concentrate these detergent mixtures into smaller packages which are more convenient for customer use and which are less expensive to both package and ship; these latter formulators have found high-density sodium tripolyphosphate having a bulk density of 0.95 to 1.15 g./cc. most desirable. The bulk density refers to the amount of the sodium tripolyphosphate which will flow, without applied pressure, into a package of given volume.

When the sodium tripolyphosphate is produced and shipped to the detergent manufacturer it is normally in an anhydrous state to eliminate the excess shipping cost incurred in transporting hydrated sodium tripolyphosphate containing 25% by weight water. In addition, detergent formulators prefer working with anhydrous sodium tripolyphosphate so that it can take up some of the water from the slurry used to produce the final detergent formulation.

One difficulty with high bulk density sodium tripolyphosphate is the rather long hydration time which it exhibits compared with sodium tripolyphosphate of lower densities. The hydration rate is significant because the extent to which the sodium tripolyphosphate is hydrated affects the physical properties of the formulation in the final package. Desirably, the sodium tripolyphosphate should be fully hydrated when the formulation is packaged in the consumer package. If some of the sodium tripolyphosphate remains in an anhydrous form, it picks up water while in the consumer package and renders the detergent formulation cakey and gritty; it may then dissolve in a non-uniform manner, leaving hard, undissolved clumps floating on the wash water. In addition, anhydrous sodium tripolyphosphate, when placed on wet hands, liberates heat of hydration and exhibits a most undesirable "detergent burn" effect on the consumers' hands. The hydrated sodium tripolyphosphate, by contrast, does not pick up water of hydration and thus does not adversely affect the physical properties of the detergent mixture.

It is an object of the present invention to produce a sodium tripolyphosphate having a bulk density of about 1.0 to about 1.15 g./cc. and preferably from about 1.03 to about 1.10 g./cc. which has a materially short hydration time.

It has now been found that a sodium tripolyphosphate product can be produced having a bulk density of from about 1.0 to about 1.15 g./cc. and having a short hydration time by compressing sodium tripolyphosphate having a bulk density of about 0.4 to about 1.2 g./cc., and pref-

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erably having a size of from 0.0555 to 0.0021 in. (-14 +270 mesh U.S. sieve), into non-friable compacted particles, the compaction being carried out at temperatures below about 350° C., grinding the compacted particles, and removing a sodium tripolyphosphate product fraction (desirably -20 +100 mesh) having a hydration rate of less than 3 minutes and a bulk density of from about 1.0 to about 1.15 g./cc.

In carrying out the present process, the sodium tripolyphosphate can be produced in either a rotary kiln or a spray dryer. In the case of the rotary dryer, a slurry is made up by reacting phosphoric acid and an alkaline compound such as sodium hydroxide or sodium carbonate in a mole ratio of sodium to phosphorus of about 1.67. The resultant mixture of monosodium orthophosphate and disodium orthophosphate is passed into a heating zone to remove water from the phosphate slurry. Thereafter, the phosphate solution is progressively heated to a temperature of 350° C. or higher in a rotary kiln during which sodium tripolyphosphate is formed. A sodium tripolyphosphate granular mass is obtained having a bulk density of from about 0.85 to about 1.2 g./cc. If the granular mass is exceedingly fine, bulk densities up to 1.2 g./cc. are obtained. In the case of the spray dryer, an orthophosphate solution is made up in much the same manner as above and is fed into the spray dryer where it is dried to an orthophosphate which can subsequently be calcined in a rotary kiln, a fluid calciner or other suitable calcining equipment to produce a product having a bulk density of 0.4 to 0.7 g./cc. It is also possible to produce a low bulk density (about 0.5 g./cc.) sodium tripolyphosphate directly from the spray dryer, if it is suitably constructed.

The sodium tripolyphosphate product thus obtained is fed into a compactor where the sodium tripolyphosphate is compressed under pressure into compacted masses, e.g. briquettes having a size about 1 1/4 x 3/4 x 1/2 in. The temperature of the sodium tripolyphosphate during briquetting should remain below 350° C. and preferentially between 50° and 150° F. If the temperature of sodium tripolyphosphate feed is too high, the compaction pressure causes the phosphates to fuse. This is undesirable, since it changes the bulk density of the resultant product as well as the hydration rate and causes difficulty in the operation of the briquetting machine.

The specific compactor employed is not critical providing that it can apply the desired pressure to compact the sodium tripolyphosphate. Among some of the commercially available compactors are the Komarek-Greaves briquette press, which employs almond briquette rolls or rolls corrugated laterally parallel to the axis of the roll shaft. The compacting pressure required in a machine such as the Komarek-Greaves 10.3-4MS briquette press is from about 20,000 p.s.i. to 70,000 p.s.i. and preferably 45,000 to 65,000 p.s.i. In these compactors, the compacting pressure is applied by hydraulic pressures which are evenly distributed over the surface of the area of one briquette as it is formed. The following correlation between hydraulic pressure and compacting pressure has been calculated for the Komarek-Greaves 10.3-4MS press:

Hydraulic Pressure (p.s.i.)	Compaction Pressure (p.s.i.)
1,200	26,900
2,000	44,800
2,500	56,000
3,000	67,200

In these compactors, the hydraulic pressures during the test went from zero to the maximum hydraulic pressure

which is set in the apparatus. Hydraulic pressures of 1,200 p.s.i., giving compacting pressures of 26,900 p.s.i., were found to be satisfactory. Where the compaction takes place under compaction pressures of below 20,000 p.s.i., the resulting particle breaks up easily and is very friable.

Following the compaction step, the compacted particles are screened to separate any non-compacted feed. The non-compacted fines which pass through the screen are returned to the compactor along with the sodium tripolyphosphate feed. The compacted particles are then ground to yield a product fraction of sodium tripolyphosphate having the size range desired (normally -20 +100 mesh), a bulk density of about 1.0 to about 1.15 g./cc. and a hydration time of less than 3 min. compared with standard sodium tripolyphosphate of the same bulk density which has a hydration time of from 6 to 12 min.

The feed which enters the compactor is preferably less than 0.10 in. with a preferred range of 0.0555 to 0.0021 in. (-14 +270 mesh). Sodium tripolyphosphate feeds which have particles more than 20% by wt. finer than 0.0021 in. (-270 mesh) tend to give a product which is too "friable." The term "friable" refers to the extent to which the sodium tripolyphosphate particle breaks up and decrepitates under mechanical handling or treatment. On the other hand, if the feed contains large particles of sodium tripolyphosphate, the feed rate to the compactor will be irregular and the resultant compacted particles will have a variable bulk density.

The following illustrates the importance of employing a sodium tripolyphosphate feed having no more than 20% by wt. -270 mesh:

Two sodium tripolyphosphate compacted samples were made by compressing sodium tripolyphosphate under substantially the same conditions using the same compactor with the exception of the size of the feed to the compactor. The compacted granular samples were then tested for friability as follows: The samples were screened through a 65 mesh Tyler sieve, and 100±0.1 g. of the screened +65 mesh granular samples were transferred to a sieve pan together with 10 one-half inch diameter steel balls. The pans and their contents were rotated in a Ro-Tap sieve shaker (made by the Tyler Company) for 5 min. with the hammer disconnected. The contents of the pan were separated from the steel balls and transferred to a 65 mesh Tyler sieve. The material was screened by rotating for 5 min. on the Ro-Tap with a hammer engaged, the +65 and -65 mesh Tyler sieve fractions were then weighed to ±0.1 g. and the percent friability was calculated according to the following formula:

Percent friability=

$$\frac{\text{Weight passed through 65 mesh Tyler sieve}}{\text{Weight retained on 65 mesh Tyler sieve plus weight passed through 65 mesh Tyler sieve}} \times 100$$

The screen analysis of the feed and the percent friability are given in Table I.

TABLE I

U.S. series sieves (Weight percent)	STPP* Feed to Compactor (Weight percent)	
	Sample 1	Sample 2
-10.....	99	100
-50.....	96	88
-100.....	85	28
-270.....	6.1	4
Percent Friability, compacted granular product.		3.7

*Sodium tripolyphosphate.

In the case of a rotary kiln, where the sodium tripolyphosphate product is obtained in a wide range of particle sizes, the feed for the compactor may be chosen by selection of the desirable sieve fraction finer than 14 mesh or

by grinding the particles that are coarser than 14 mesh and then selecting the desired sieve fraction as feed to the compactor.

The method of determining the hydration time is an empirical one and is carried out as follows: One hundred mls. of water at 80° to 84° F. is placed in a clean, dry, 1-pint, wide-mouthed vacuum jar. A rotary-driven stirrer is inserted with a blade near the bottom of the jar and set at 400 r.p.m. Twenty-five grams of anhydrous sodium sulfate is then added to the vacuum jar. When the anhydrous sodium sulfate is dissolved, the stirrer speed is decreased to 200 r.p.m. and the solution temperature is adjusted to 80° to 84° F. A 75-gram sample of the sodium tripolyphosphate to be tested is then added evenly to the liquid over a period of 15 to 20 seconds. When all of the sodium tripolyphosphate has been added, a timer is begun. The surface of the mixture is observed and when all circular motion of the surface stops, the end point has been reached and the timer is stopped. The surface characteristics of sodium tripolyphosphate slurries vary. Usually though, the sodium tripolyphosphate water mixture thickens and a minute or so before the end point occurs, the mixture is so thick that the slurring action becomes very sluggish. When the circulatory motion of the surface stops, this indicates that the end point has been reached. This does not include the small area directly adjacent to the stirrer shaft.

In order to illustrate the present invention, the following schematic drawing of the process is presented for production of -20 +100 mesh product.

In the drawing, sodium tripolyphosphate having a bulk density of 0.4 to 1.0 g./cc. and a particle size of -14 +270 mesh U.S. sieve is sent to a compactor. Within the compactor, the sodium tripolyphosphate feed is compressed into relatively large compacted masses under high pressure. The compacted masses are then passed into a scalping screen for removal of non-compacted fines. These fines are produced by feed which has by-passed the compactor rolls or by the breakage of some of the compacted particles. The compacted particles from the scalping screens are then passed into a grinder where they are ground to provide the product size desired, in this case a -20 +100 mesh fraction. The product from the grinder is passed through a final screening step which separates a coarse fraction (+20 mesh), and a fine fraction (-100 mesh) from the desired -20 +100 mesh fraction. The coarse fraction (+20 mesh) is recycled to the grinder to reduce the size of the particles. The fines (-100 mesh fraction) which are separated in the final screening step are returned to the compactor for reprocessing. The -20 +100 mesh fraction is then recovered as product and has a bulk density of 1.0 to 1.15 and a hydration time of less than 3 min.

The following example is given to illustrate the invention and is not deemed to be limitative thereof.

Example I

Four runs were made in which both spray dried and rotary kiln dried sodium tripolyphosphate were employed as the feed. The sodium tripolyphosphate feed, having the properties set forth in Table II, was fed into a Komarek-Greaves 10.3-4MS briquette press at the rates given in Table II. The sodium tripolyphosphate was compacted at the pressures given in Table II, and the compacted particles were then sent to scalping screens to separate any fines. The fines were returned to the compactor at the rate given in Table II, while the compacted particles were sent to a grinding mill. The milled compacted particles were then sent to product screens where the -20 +100 mesh fraction was removed. The finer particles (-100 mesh) were returned to the compactor, and the coarser particles (+20 mesh) were returned to the grinder at the rate given in Table I. The resultant -20 +100 fraction was tested for its hydration rate according to the aforementioned method:

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One hundred milliliters of water at 80° to 84° F. was placed in a clean, dry, 1-pint, wide-mouthed vacuum jar. A rotary-driven stirrer was inserted with a blade at the bottom of the jar and set at 400 r.p.m. Twenty-five grams of anhydrous sodium sulfate was added to the vacuum jar. When the anhydrous sodium sulfate was dissolved, the stirrer speed was decreased to 200 r.p.m. and the solution temperature was adjusted to 80° to 84° F. A 75-gram sample of the sodium tripolyphosphate was added evenly to the solution in the jar over a period of 15 to 20 sec. When all of the sodium tripolyphosphate was added, a timer was begun. The surface of the mixture was observed and when all circular motion of the surface stopped, excluding the small area directly adjacent to the stirrer shaft, the timer was stopped and the time recorded in minutes. Both the bulk density and the hydration time of the -20 +100 particle fraction are reported in Table II.

TABLE II

	Run 1	Run 2	Run 3	Run 4
Sodium Tripolyphosphate Feed:				
Source.....	(¹)	(¹)	(²)	(²)
Bulk Density (g./cc.).....	.959	1.10	.655	.506
Size (U.S. sieves, wt. percent):				
-20.....	94	86	92	96
-50.....	82	51	79	53
-100.....	60	29	32	14
-270.....	10	4	5	4
Feed Rate (lb./hr.).....	1,610	3,200	2,480	2,380
Compactor Hydraulic Roll Pressure (p.s.i.).....	2,200	2,100	1,950	1,950
Recycle Rates (lb./hr.):				
From Scalping Screens.....	650	900	800	700
From Product Screens.....	400	1,200	750	900
Production Rate (lb./hr.).....	560	1,100	930	780
Product:				
Bulk Density (g./cc.).....	1.03	1.09	1.07	1.05
Hydration Time (min.).....	1.45	1.35	1.45	1.28
Size (U.S. sieves, wt. percent):				
-20.....	99.9	99.9	99.9	99.9
-30.....	90	83	86	87
-100.....	1.0	2.1	2.7	1.7

¹ Rotary dryer. ² Spray dryer.

Pursuant to the requirements of the patent statutes, the principle of this invention has been explained and exemplified in a manner so that it can be readily practiced by those skilled in the art, such exemplification including what is considered to represent the best embodiment of the invention. However, it should be clearly understood that, within the scope of the appended claims, the invention may be practiced by those skilled in the art, and having the benefit of this disclosure otherwise than as specifically described and exemplified herein.

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What is claimed is:

1. The process of producing granular sodium tripolyphosphate from an aqueous sodium orthophosphate mixture comprising the steps of passing said mixture through a heated zone to obtain an initial sodium tripolyphosphate in particulate form having a bulk density of about 0.4 to about 1.2 g./cc., compacting particles of said initial sodium tripolyphosphate at temperatures below about 350° C. to form non-friable compacted particles, grinding said compacted particles and removing a granular sodium tripolyphosphate product having a bulk density of from about 1.0 to about 1.15 g./cc., and having a hydration time of less than about 3 minutes.
2. Process of claim 1 wherein said product fraction is about -20 to about +100 mesh.
3. Process of claim 1 wherein said initial sodium tripolyphosphate is produced by spray drying said aqueous sodium orthophosphate mixture and has a bulk density of from about 0.4 to about 0.7.
4. Process of claim 1 wherein said initial sodium tripolyphosphate is produced by drying said aqueous sodium orthophosphate mixture in a rotary kiln and has a bulk density of from about 0.8 to about 1.2.
5. Process of claim 1 wherein the sodium tripolyphosphate product has a bulk density of about 1.03 to about 1.10.
6. Process of claim 1 wherein said fraction of said initial sodium tripolyphosphate to be compacted is essentially -50 mesh and contains not more than 20% by weight -270 mesh.
7. Process of claim 1 wherein said initial sodium tripolyphosphate has a mesh size of substantially about -14 to about +270.

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