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1

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PROCESS FOR PRODUCING
CLEANSING TABLETS

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9 Claims. (Cl. 252-99)

ABSTRACT OF THE DISCLOSURE

Process for producing strong, fast-dissolving cleansing tablets containing about 35 to 65% by weight of sodium tripolyphosphate wherein the dissolving rate of the tablets are increased by utilizing sodium tripolyphosphate which is all in the Form II crystalline configuration and has a bulk density of from about 0.7 to 1.0 g./cc. The tablets also may include bleaching agents such as sodium dichloroisocyanurate or potassium dichloroisocyanurate.

This invention relates to a process for making built, heavy-duty cleansing tablets, and, more specifically, to the production of detergent tablets or bleaching tablets which are sufficiently strong in the dry state to withstand breakage, but which have desirably rapid disintegration rates when placed in water.

This application is a continuation-in-part of U.S. patent application No. 385,043 filed on July 24, 1964, now now abandoned.

The use of both detergent tablets and bleach tablets has become increasingly popular compared with their liquid or powder counterparts because of the more desirable handling characteristics which these tablets have. These tablets obviate the need for measuring cups used to dispense the required powder or liquid used in domestic washing machines. They also eliminate the problem of spillage and storage of bulky detergent or bleach containers. In normal manufacture, the heavy-duty, built, detergent formulations, or bleaching compositions, are blended and pressed into tablets so that they have sufficient strength to resist breakage in handling and use, but are capable of disintegration at a rapid rate when placed in wash water.

In the make-up of detergent tablets one or more working ingredients are employed in combination with a phosphate builder such as sodium tripolyphosphate. These include anti-redeposition agents such as sodium carboxymethylcellulose, anionic or nonionic surfactants, and anti-corrosion agents such as sodium silicate. Some formulations also contain inert ingredients which act as extenders and are used to obtain the desired bulk density in the pelletizing mix and to yield a smoother and more solid appearing tablet. In general, these ingredients are mixed together to form a homogeneous dry mixture and are compressed in a mold to form a tablet.

In the case of making built tablets intended for bleaching, it is customary to include a dry bleaching agent such as a chlorocyanuric acid or salts thereof in the make-up of the tableting mix. The built, bleaching tablets are

2

made up in the same manner as the detergent tablets above in that they contain sodium tripolyphosphate and one or more working ingredients admixed with a dry bleaching agent.

In the make-up of cleansing tablets it has been customary to use -16 +100 mesh particles of low bulk density sodium tripolyphosphate, i.e. having a bulk density of from about 0.35 to about 0.6 g./cc. This lighter sodium tripolyphosphate is desirable because it can be compressed into detergent tablets leaving a sufficient number of voids in the tablet to accelerate disintegration and dissolution of the resultant tablet in the wash water. The bulk density refers to the amount of sodium tripolyphosphate which will flow, without applied pressure, into a package of given volume.

A problem that has arisen in the manufacture of cleansing tablets is the inability to produce faster dissolving tablets without also diminishing the strength of the dry tablets to withstand the shocks of packaging, handling, dispensing, and the like.

One approach to obtaining faster dissolution of the highly compressed tablets is the addition of additives such as CO₂ generators, starch, or other inert materials which increase disintegration. However, many of these materials are either insoluble, expensive, or are otherwise undesirable as ingredients in the cleansing tablet and therefore have not met with wide success. Other techniques such as using low ram pressures during the pressing of the tablets have been proposed but have not been successful because the resultant tablet is not sufficiently strong to withstand normal dry handling.

In our copending application Serial No. 381,216, filed on July 8, 1964, it is disclosed that detergent tablets having a very high dissolving rate, with acceptable dry strengths sufficient to withstand normal handling and packaging, can be prepared by using a "compacted" sodium tripolyphosphate having a density of from about 1.0 to about 1.25 g./cc. and having an extremely fast hydration time. While this process yields tablets of high quality, it suffers the drawback that a specially processed high density sodium tripolyphosphate must be used in the production of the detergent tablets.

As a result, there is a need for a process for producing a cleansing tablet whose strength in the dry state is comparable with other tablets presently used in the art, but whose disintegration rate in the wash water of conventional washing machines is substantially increased over conventional tablets. Moreover, the ingredients used in the make-up of such a cleansing tablet should be those which are easily produced in the trade.

It is an object of the present invention to produce a cleansing tablet having an acceptable dry strength sufficient to withstand normal handling and packaging, but whose dissolving rate is extremely high compared with conventional tablets, and which requires ingredients which can be readily produced in commercial quantities.

These and other objects will be apparent from the following description.

We have made the surprising discovery that built, cleansing tablets which are made up with a dense sodium tripolyphosphate having a density of from about 0.7 to about 1.0 g./cc. and which is substantially all in the Form

II crystalline structure, have an extremely fast dissolving rate and good dry strengths sufficient to withstand normal handling and packaging.

We have also found that the excellent dissolving rates and dry strengths of the tablets are obtained even when large amounts of Form II sodium tripolyphosphate fines (—100 mesh particles) are present in the formulation used to make up the tablets.

As a corollary to the above discovery, we have also found that built, cleansing tablets which are made up with a dense sodium tripolyphosphate having a density of about 0.7 to about 1.0 g./cc., a size of —100 mesh, and which contain at least about 16% of Form I crystalline structure, have extremely slow dissolving rates.

It is most surprising that a dense sodium tripolyphosphate yields a tablet having fast dissolving rates since only low bulk density sodium tripolyphosphate (which is used to make up conventional detergent tablets) produces the large amounts of air voids within the tablet which are considered mandatory to fast dissolving rates. The general belief prevalent in the detergent industry has been that the more dense sodium tripolyphosphate is not suitable for incorporation in detergent tablets because its denseness would preclude a fast dissolving tablet. In contrast to this belief, conventional high density (0.7–1.0 g./cc.) sodium tripolyphosphate which is substantially all in the Form II crystalline structure has been found eminently suitable for the production of fast dissolving detergent tablets.

Sodium tripolyphosphate, which is a crystalline anhydrous product, is capable of having two forms, Form I and Form II. Form I is produced at temperatures of from about 500–620° C., while Form II is produced at temperatures below about 500° C. In general, most sodium tripolyphosphate is a mixture of Form I and Form II. In certain cases, the proportion of Form I to Form II is regulated by the manufacturer by careful control of the temperature and duration of heating to meet a specific ratio. However, in the normal high temperature rotary kiln process, high density sodium tripolyphosphate normally always contains sizable quantities of Form I, the exact amount depending on local temperatures within the rotary kiln.

One method for measuring the amount of Form II sodium tripolyphosphate in a sample of sodium tripolyphosphate is to slurry 50 g. of sodium tripolyphosphate with 50 g. of glycerine until the mixing is complete. The glycerine preferentially inhibits the hydration of Form II sodium tripolyphosphate. The temperature of the solution is then carefully read and recorded in degrees centigrade. Thereafter, 25 ml. of water is added and the mixing is continued until the temperature rise levels off. The highest temperature obtained at the leveling off point is recorded and the percent of Form I is determined in accordance with the following formula:

$$\% \text{ Form I} = 4(\Delta T - 6)$$

where ΔT is the difference between the initial and the final temperature in degrees centigrade.

In the preferred mode of practicing the present invention, a detergent formulation for producing rapidly dissolving detergent tablets is made up as follows:

The principal ingredient, the Form II sodium tripolyphosphate, is measured into the detergent formulation in amounts sufficient to constitute from about 35 to about 65% by weight of the detergent formulation. The preferred range of sodium tripolyphosphate is from about 50 to about 65% by weight with about 58% being the optimum amount.

The next added ingredient is the surface active agent (surfactant). The surfactant, which can be used in the make-up of the formulation, may be either nonionic or anionic; certain cationic surfactants cannot be employed because they are incompatible with the Form II sodium tripolyphosphate. The surfactant is added to the mixture

in amounts of from about 5% to about 15% by weight of the formulation. Ten percent by weight is generally considered optimum.

To the above mixture is added a sodium silicate having a $\text{Na}_2\text{O}/\text{SiO}_2$ mole ratio of from about 1:1 to about 1:3.2 in amounts of from about 3% to about 15% by weight of the formulation. Generally, about 7% of 81.5% active sodium silicate having a $\text{Na}_2\text{O}/\text{SiO}_2$ mole ratio of 1:2 has been found to give good results. In general the optimum amounts of sodium silicate normally supply from about 3 to about 3.5 weight percent of SiO_2 to the formulation.

The next added ingredient is a filler, e.g. sodium sulfate, which constitutes the remaining major portion of the formulation. The sodium sulfate is an inert filler which is added to control the bulk density of the tableting mixture and to improve the surface appearance of the tablets by giving them a smoother and more compact appearance. In addition, there is also added small amounts of auxiliary compounds such as sodium carboxymethylcellulose, generally in amounts of from about 0.2% to about 1.5%, foam stabilizers such as lauroyl diethanolamide, tarnish inhibitors, fluorescent brighteners, perfumes, bacteriostats, coloring matter, etc. The resultant mixture thus formulated is uniformly mixed and pressed into tablets. The pressing is normally accomplished using pressures of from about 175 to about 200 p.s.i. and the finished tablet has a specific gravity of from about 0.8 to about 1.3 with about 1.02 being preferred. The newly formed tablets normally have strengths (when pressed on edge) of from about 10 to about 15 pounds. If the tablets are suitably aged for at least 24 hours, they have a strength (when pressed on edge) of up to about 25 pounds. Desirably, these tablets have dissolving times of less than 120 seconds when placed in a conventional washing machine containing water at 120° F.

In the make-up of these detergent tablets, the Form II sodium tripolyphosphate preferably has a bulk density of from about 0.7 to about 1.0 g./cc. The Form II sodium tripolyphosphate desirably is made up of substantially all —16 +100 mesh fraction. However, it can be made up of all —100 mesh Form II sodium tripolyphosphate fines without any material effect on the dissolving rate, although fines are more difficult to handle on a large scale. By contrast, the use of such large quantities of fines when utilizing conventional sodium tripolyphosphate results in a material and undesirable decrease in the dissolving rate of the resulting tablet.

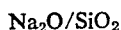
The anionic surface active agents are useful in the present formulations in amounts of from about 5 to about 15% by weight of the formulation. These anionic surface active agents are non-soap synthetic detergents made up of water-soluble salts of organic sulfuric reaction products having from about 8 to about 18 carbon atoms in the form of an alkyl or acyl radical within the molecular structure and containing sulfuric or sulfonic acid ester radicals. Typical examples of these anionic surface active agents are sodium or potassium alkyl benzene sulfonates in which the alkyl group contains from about 8 to about 18 carbon atoms, preferably in a straight chain to achieve biodegradability, e.g., sodium dodecylbenzene sulfonate, sodium tridecylbenzene sulfonate; the sodium and potassium alkyl glycerol ether sulfonates, including ethers of higher fatty alcohols derived from the reduction of coconut oils; the reaction products of higher fatty acids, e.g. coconut oil with sodium or potassium isethionate; sodium or potassium alkyl sulfonates and sulfates obtained by sulfonation of coconut or tallow fatty alcohols and mixtures of such alkyl sulfates; dialkyl esters of sodium or potassium salts of sulfosuccinic acid; sodium and potassium salts of sulfated or sulfonated monoglycerides, e.g., those derived from coconut oil; sodium or potassium salts of higher fatty alcohol esters of sulfoacetic acids, e.g., sodium salt of lauryl alcohol ester of sulfoacetic acid; and other anionic agents set forth in U.S. Patent

2,486,921, issued to Byerly on November 1, 1949. If desired, the anionic surfactant can be added in the form of a dense, dry bead or as a flake admixed with sodium sulfate. In this latter case, the sodium sulfate constitutes a portion of the total sodium sulfate used in making up the entire mixture.

The nonionic surface active agents useful in the present invention are non-soap synthetic detergents made up of a water solubilizing polyoxyethylene group in chemical combination with an organic hydrophobic compound. Among the hydrophobic compounds which can be used are polyoxypropylene, the reaction product of propylene oxide and ethylene diamine, aliphatic alcohols, etc. Examples of nonionic synthetic detergents useful in the present invention are condensation products of 6-30 moles of ethylene oxide, and preferably 7-11 moles, with 1 mole of an alkyl phenol containing 6-12 carbon atoms in the alkyl group; condensation products of 6-30 moles of ethylene oxide with 1 mole of an aliphatic straight or branched-chained alcohol containing 8-18 carbon atoms; condensation products of ethylene oxide and the reaction product of propylene oxide and ethylene diamine; nonylphenol polyethoxy ethanol (commercially known as "Triton N" series); isooctyl phenol polyethoxy ethanol (commercially known as "Triton X" series). Another well-known group of nonionic detergents is known under the trade name of the "Pluronic" series. These compounds are the reaction products obtained by condensing ethylene oxide with a hydrophobic base produced by the condensation of propylene oxide with propylene glycol, and have molecular weights on the order of about 1800. The addition of polyoxyethylene radicals to the hydrophobic base increases the water solubility of the nonionic detergent and concurrently increases the foaming properties of the detergent in aqueous solution in proportion to the mole ratio of polyoxyethylene radicals to the hydrophobic base. In general, a surfactant which has a mole ratio of 7.5 moles of ethylene oxide per mole of an alkyl phenol, e.g. nonylphenol, is low-foaming while one with a mole ratio of 10:1 foams moderately. The molecular weight of these nonionic synthetic detergents will range from as low as 800 up to about 11,000.

Nonionic surfactants should be added to the present formulation in amounts of about 6% by weight of the total formulation or above in order for the surfactant to be completely effective. Amounts below about 6% by weight reduce the cleaning action of the detergent and should be avoided. Amounts over 14% similarly should be avoided because the nonionic surfactant tends to exude or "oil out" of the detergent formulation when it is pressed into tablets. Within the range of 6% to 14%, the nonionic surfactant gives effective washing action, and has been found to be effective as a binder for the remainder of the detergent formulation without "oiling out" of the pressed tablet. The preferred amount is about 10%.

The sodium silicate that is added to the mixture normally constitutes from about 3% to about 15% by weight of the detergent formulation. The mole ratio of



in the sodium silicate determines the degree of alkalinity of this compound; as the ratio approaches 1:1, the sodium silicate becomes more alkaline. Ratios above 1:2, e.g. 1:1, although normally quite alkaline, are commonly used. However, ratios of not above 1:2 are preferred to prevent the wash water reaching pH levels which are unsafe for certain fibers. Ratios below 1:3.2, e.g. 1:4, dissolve too slowly and are not effective.

After the ingredients have been uniformly mixed together, the resulting dry mixture should have a bulk density in the range of from about 0.4 to about 0.85 g./cc., with the preferred bulk density being about 0.7 g./cc. Predetermined quantities of this mixture are then fed to a die and pressed at from about 100 to about 350 p.s.i. to

yield a tablet having a specific gravity of about 1.02. A tablet having a specific gravity of about 1.0 g./cc. is commonly produced by the above process. A typical tablet has the following formulation:

	Percent by weight
5 Sodium tripolyphosphate	35 to 65
Sodium silicate	3 to 15
Sodium carboxymethylcellulose	0.2 to 1.5
Anionic or nonionic surface active agent	5 to 15
10 Sodium sulfate	Balance

In the procedure of tableting this detergent formulation, it has been found that small portions of the pressed mixture do not adhere to the dies, nor is there any capping during the pressing of this formulation. The term "capping" refers to the internal horizontal separation of the tablet into two or more pieces because of the adherence of these pieces to each of the dies. In general, the use of standard dies is eminently satisfactory without special provision for rotation of the dies during the pressing operation.

In the make-up of bleach tablets the same procedure is used as in the make-up of the detergent tablets, except that from about 1-90% by weight of a dry bleaching agent and from about 8-65% by weight of sodium tripolyphosphate can be incorporated in the pelletizing mix. In the case of tablets intended for textile bleaching, a preferred formulation contains from about 20-65% by weight of sodium tripolyphosphate and from about 10-50% by weight of a dry bleaching agent. When the tablets are intended for a hard surface cleaning, bleaching, and sanitizing, the preferred formulation contains from about 20-65% by weight of sodium tripolyphosphate and from about 2-10% by weight of a dry bleaching agent. However, the dry bleaching agent can range from 2 to 90% by weight of the tablet. The dry bleaching agent preferably is a stable active chlorine yielding compound such as an alkali metal or alkaline earth metal salt of a chlorocyanuric acid, e.g. sodium dichlorocyanurate, or potassium dichlorocyanurate. A typical formulation of such a textile bleaching tablet is listed below.

	Percent by weight
Sodium tripolyphosphate	30
Sodium dichloroisocyanurate	40
45 Sodium alkylaryl sulfonate	4
Inert filler	26

The resulting pressed tablets may be subjected, if desired, to steaming, spraying, or fogging with water, with or without additives, in order to hydrate a surface layer of hydratable components, e.g. sodium tripolyphosphate and sodium sulfate. The steaming or spraying helps form a film or layer of increased strength to help prevent fracturing of the product during normal handling. This steaming is helpful because the hydratable ingredients crystallize with such action that they create a binding force between adjacent particles. In addition, the application of moisture makes the surface more homogeneous, thereby increasing the surface density and the cohesive force between the particles.

The term "tablet" as used in the present disclosure and claims refers not only to that product which is obtained by compressing the homogeneous dry mix in a mold to form a unitary pressed shape, but also refers to any product in which a dry mixture is compressed into a dense shape, whether or not this dense shape is subsequently left intact or whether it is broken into individual discrete pieces of smaller size.

The following examples are given to illustrate the invention and are not deemed to be limiting thereof.

EXAMPLE 1

Run A.—The following anionic detergent formulations were mixed in a Kitchen-Aid planetary mixer (Model 4-C, Hobart Manufacturing Co., Troy, Ohio) for about 75 3½ minutes.

7
TABLE I

Ingredients	Formulations			
	1	2	3	4
Sodium Tripolyphosphate:				
(a) ΔT	5.5	5.5	7.2	7.2
(b) Bulk Density (g./cc.).....	0.95	0.88	0.98	0.89
(c) Fines Content (percent).....	1.4	30	1.4	30
(d) Weight (g.).....	346	346	346	346
Spray Dried Bead ¹	546	546	546	546

¹ A spray dried powdered product containing the following products:

	Percent by wt.
Sodium Tripolyphosphate.....	25
Anionic Surfactant (Sodium Dodecylbenzene Sulfonate, 100%).....	26
Sodium Silicate ($\text{Na}_2\text{O}/\text{SiO}_2$ mole ratio 1:2.4).....	5
Moisture.....	8-11
Sodium Carboxymethylcellulose.....	0.67
Optical Brightener.....	0.2
Sodium Sulfate, Balance of Bead Formulation.....	

The formulations were pressed in a Carver hand press to form tablets weighing 53 g., having a thickness of 25.2 mm. and a specific gravity of 1.01. The fines level ($-100 +270$ mesh fraction) of the sodium tripolyphosphate used in formulations 1, 2, 3, and 4 are expressed as percent by weight of the sodium tripolyphosphate. The tablets were then conditioned for approximately 16 hours in sealed jars at ambient room temperature before testing.

The tablets were tested for disintegration time by observing the time (in seconds) required for the tablets to be sufficiently reduced in size to allow escape from a $\frac{1}{2}$ " wire mesh cage suspended in a fixed position in a transparent-sided Kenmore washer containing 120° F. agitating tap water. In addition, the crush strength of the tablets was obtained by measuring the force in pounds needed to crush the tablets when placed on edge. The tablets were not steamed after manufacture to increase their crush strength in order to obtain the best possible reproducibility in test results. The results of the test are set forth in Table III.

Run B.—By way of comparison, several detergent mixtures (Formulas A, B, C, and D) were made up using the same proportion of ingredients as set forth in Run A with samples of sodium tripolyphosphate having about the same density as the Form II sodium tripolyphosphate except that they contained substantial portions of Form I sodium tripolyphosphate as set forth below:

TABLE II

Ingredients	Formulations			
	A	B	C	D
Sodium Tripolyphosphate:				
(a) ΔT	12.1	12.1	17.5	17.5
(b) Bulk Density (g./cc.).....	0.99	0.90	0.95	0.88
(c) Fines Content (percent).....	1.4	30	1.4	30

When such tablets were pressed into identical tablets by the same procedure as set forth in Run A, the crush strengths and disintegration times set forth in Table III were obtained.

TABLE III

	Formulations							
	1	2	3	4	A	B	C	D
Bulk Density of Formulation (g./cc.).....	0.495	0.510	0.518	0.515	0.506	0.498	0.517	0.519
Properties of Sodium Tripolyphosphate Utilized:								
(a) ΔT	5.5	5.5	7.2	7.2	12.1	12.1	17.5	17.5
(b) Bulk Density (g./cc.).....	0.95	0.88	0.98	0.89	0.99	0.90	0.95	0.88
(c) Fines Content (percent).....	1.4	30	1.4	30	1.4	30	1.4	30
Crush Strength of Tablets (lbs.).....	23	24	26	20	26	19	19	30
Disintegration Time (seconds).....	102	104	90	98	118	134	134	166

EXAMPLE 2

Run A.—The following anionic detergent formulations, Formulations 5 and 6, were mixed and tableted in the

8

same manner as set forth in Example 1, Run A using the same proportions of ingredients. The fines level ($-100 +270$) mesh fraction) of the sodium tripolyphosphate used in these formulations was 15% in Formulation 5 and 30% in Formulation 6, expressed as percent by weight of sodium tripolyphosphate. In this run, only Form II fines were introduced into the conventional $-16 +100$ mesh fraction of Form II sodium tripolyphosphate. The tablets were then tested as set forth in Example 1 and these test results are reported in Table IV.

Run B.—By way of comparison, detergent formulations E and F were prepared in the same manner as set forth above except that the 15% and 30% fines level in Formulations E and F, respectively, were made up of sodium tripolyphosphate containing substantial amounts of Form I (ΔT 12:1). These tablets were tested as set forth in Example 1 and the results are reported in Table IV.

TABLE IV

	Formulations			
	5	6	E	F
Sodium Tripolyphosphate:				
(1) Conventional Size ($-16 +100$ mesh):				
(a) ΔT	5.5	5.5	5.5	5.5
(2) Fines ($-100 +270$ mesh):				
(a) ΔT	5.5	5.5	12.1	12.1
(b) Fines Content (percent).....	15	30	15	30
Crush Strength of Tablets (lbs.).....	23	24	27	25
Disintegration Time (seconds).....	106	104	142	160

The above example is designed to shown the effect of utilizing a small amount of fines in the sodium tripolyphosphate which contain an appreciable amount of Form I sodium tripolyphosphate. Note that large quantities of Form II sodium tripolyphosphate fines do not deleteriously affect the disintegration time of the resultant tablet. However, if the Form II sodium tripolyphosphate contains even small amounts of Form I fines, the advantageous decrease in disintegration time is lost.

EXAMPLE 3

Run A.—The following series of formulations were mixed in a Kitchen-Aid planetary mixer such as employed in Example 1. In this series of formulations, various amounts of -100 mesh sodium tripolyphosphate fines were used ranging from 0-100%. The sodium tripolyphosphate employed was all Form II. The formulations were pressed in a Carver hand press at 12,000 lbs. on the ram to form tablets weighing 50 g. and having a $1\frac{1}{4}$ " diameter. The formulations were listed below in Table V.

TABLE V

Ingredients	Formulations			
	7	8	9	10
Sodium Tripolyphosphate:				
(a) ΔT	7.0	7.0	7.0	7.0
(b) Particle Size:				
-100 mesh (percent).....	100	30	10	0
$+100$ mesh (percent).....	0	70	90	100
(c) Weight Percent.....	30	30	30	30
(d) Bulk Density (g./cc.).....	0.70	0.87	0.92	0.95
Sodium Dichlorocyanurate (wt. percent).....	40	40	40	40
Sodium Alkylaryl Sulfonate (wt. percent).....	4	4	4	4
Sodium Chloride (wt. percent).....	25.8	25.8	25.8	25.8

The tablets were steamed for 10 seconds and tested for strength and disintegration time in the same manner as specified in Example 1. The crush strength and disintegration time are set forth in Table VII.

Run B.—By way of comparison, several detergent mixtures were made up by using the same proportions of ingredients as set forth in Run A, except that they contain substantial portions of Form I sodium tripolyphosphate. The formulations are set forth below in Table VI.

TABLE VI

Ingredients	Formulations	
	G	H
Sodium Tripolyphosphate:		
(a) ΔT	10.5	10.5
(b) Particle Size:		
-100 mesh (percent).....	100	30
+100 mesh (percent).....	0	70
(c) Weight Percent.....	30	30
(d) Bulk Density (g./cc.).....	0.71	0.90
Sodium Dichloroisocyanurate (wt. percent).....	4	40
Sodium Alkylaryl Sulfonate (wt. percent).....	4	4
Sodium Chloride (wt. percent).....	25.8	25.8

When these formulations were pressed into identical tablets by the same procedure set forth in Run A, the crush strength and disintegration time of the resultant tablets were obtained. The results are listed in Table VII.

TABLE VII

	Formulations					
	7	8	9	10	G	H
Sodium Tripolyphosphate:						
(a) ΔT	7.0	7.0	7.0	7.0	10.5	10.5
(b) Particle Size:						
-100 mesh (percent).....	100	30	10	0	100	30
+100 mesh (percent).....	0	70	90	100	0	70
Crushed Strength of Tablets (lbs.).....	41	49	38	38	37	37
Disintegration Time (sec.).....	77	81	78	64	127	91

EXAMPLE 4

Run 11.—The following chlorinated hard surface cleaning formulation was mixed in a Kitchen-Aid planetary mixer under the same conditions specified in Example 1. The composition contained 40% by weight of granular sodium tripolyphosphate Form II, 30% by weight of sodium metasilicate, 2.5% by weight of potassium dichloroisocyanurate, and 27.5% by weight of sodium chloride. This dry mix was pressed into 2 oz. $2\frac{1}{4}$ " diameter tablets at 4,000 lbs. ram pressure. The tablets were tested for disintegration time as specified in Example 1, except that the agitated tap water was maintained at 110° F. The sample tablets were found to completely disintegrate in from 30–35 seconds.

Run J.—The above procedure was repeated using granular sodium tripolyphosphate containing 20% of Form I sodium tripolyphosphate. The disintegration time was 320 seconds.

EXAMPLE 5

Run 12.—The following dry bleach formulation was mixed in a Kitchen-Aid planetary mixer under the same conditions specified in Example 1. The composition contained 47.6% powdered sodium tripolyphosphate Form II, 14% water, and 38.4% sodium dichloroisocyanurate. This mix was pressed into 2 oz. $2\frac{1}{4}$ " tablets at 4,000 lbs. ram pressure. The tablets were broken into small discrete particles and were tested for the dissolving rate. The dissolving test consisted of adding one gram of the discrete particles to 200 ml. of agitated distilled water and measuring the time for all particles to disappear when observed by reflected light. The discrete particles required 80 seconds to dissolve.

Run K.—An identical mixture was made up in exactly the same manner as Run A, except that sodium tripolyphosphate containing 26% of Form I was employed instead of the Form II sodium tripolyphosphate used in Run 12. The dissolving rates of the discrete particles were tested in like manner and this formulation containing Form I sodium tripolyphosphate had a dissolving time of 175 seconds.

The above example illustrates that the present favorable dissolving rates are contained not only in the compressed tablets, but also in discrete particles obtained from a compressed, denser shape such as may be produced commercially, for example, by use of a compactor. Thus, in place of tablets, the present process can be utilized in making large preformed sheets or briquettes of formula which may then be sectionalized into smaller discrete shapes and still obtain the advantageous fast dissolving rates.

While the present invention is concerned primarily with the production of fast dissolving tablets, our research has shown that extremely slow dissolving cleansing tablets can be obtained by using sodium tripolyphosphate fines containing at least 16% Form I (ΔT of 12.5). This is important, only in a limited class of tablets which are intended for use in commercial plants in which periodic changes of the wash water are employed and in which it is desired to have the cleansing tablet remain active throughout a series of these changes in wash water. Heretofore, one principal method for decreasing dissolving rates was to increase the pressure used in pressing the tablets. However, this technique has limitations because the pressure used in pressing the tablets is limited by the fact that capping (separation of the tablets in the die) results above a given ram pressure. By means of the present ancillary discovery, ordinary pressing techniques can be utilized to make a slow dissolving tablet by mere use of Form I sodium tripolyphosphate fines in the detergent mixture.

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.

What is claimed is:

1. In a process for producing strong, heavy-duty cleaning tablets having a fast dissolving rate wherein a substantially dry formulation containing about 35% to about 65% by weight of sodium tripolyphosphate is pressed into tablets, the improvement which consists essentially of formulating said substantially dry formulation with sodium tripolyphosphate having a bulk density from about 0.7 to about 1.0 g./cc. and which is substantially all in the Form II crystalline configuration, whereby the dissolving rate of said tablets is increased without diminishing the dry strength of said tablets.

2. In a process for producing strong, heavy-duty cleaning and bleaching tablets having a fast dissolving rate wherein a substantially dry formulation comprising from about 8% to about 65% by weight of sodium tripolyphosphate and from about 1% to about 90% by weight of a dry bleaching agent, selected from the group consisting of sodium dichloroisocyanurate and potassium dichloroisocyanurate, is pressed into tablets, the improvement consisting essentially of formulating said substantially dry formulation with said sodium tripolyphosphate having a bulk density from about 0.7 to about 1.0 g./cc. and which is substantially all in the Form II crystalline configuration whereby the dissolving rate of said tablets is increased without diminishing the dry strength of said tablets.

3. Process of claim 2 in which the sodium tripolyphosphate is present in amounts of from about 20% to about 65% by weight and wherein the dry bleaching agent is present in amounts from about 10% to about 65% by weight.

4. In a process for producing a strong, heavy-duty, hard surface cleaning, bleaching, and sanitizing tablet having a fast dissolving rate wherein a substantially dry formulation comprising about 20% to about 65% by

weight of sodium tripolyphosphate and from about 2% to about 90% by weight of a dry bleaching agent, selected from the group consisting of sodium dichloroisocyanurate and potassium dichloroisocyanurate, is pressed into tablets, the improvement consisting essentially of formulating said substantially dry formulation with said sodium tripolyphosphate having a bulk density from about 0.7 to about 1.0 g./cc. and which is substantially all in the Form II crystalline configuration whereby the dissolving rate of said tablets is increased without diminishing the dry strength of said tablets.

5. In a process for producing strong, heavy-duty detergent tablets having a fast dissolving rate wherein a substantially dry formulation comprising from about 35% to about 65% by weight of sodium tripolyphosphate, from about 3% to about 15% by weight of sodium silicate having an $\text{Na}_2\text{O}/\text{SiO}_2$ mole ratio of 1:1 to 1:3.2, from about 5% to about 15% by weight of a water soluble non-soap organic synthetic detergent selected from the group consisting of anionic detergents selected from the group consisting of the sodium and potassium salts of alkyl sulfate, alkyl sulfonates and alkyl aryl sulfonates having from about 8 to about 18 carbon atoms in the alkyl group, and nonionic detergents selected from the group consisting of the condensation products of about 6 to about 30 moles of ethylene oxide with one mole of an alkyl alcohol wherein the alkyl group contains from about 8 to about 18 carbon atoms, and the condensation products of about 7 to about 11 moles of ethylene oxide with one mole of an alkyl phenol wherein the alkyl group contains from about 6 to about 12 carbon atoms, is pressed into tablets having a specific gravity of about 1.0, the improvement consisting essentially of formulating said substantially dry formulation with sodium tripolyphosphate having a bulk density of from about 0.7 to about 1.0 g./cc. and which is substantially all in the Form II crystalline configuration whereby dissolving rate of said tables is increased without diminishing the dry strength of said tablets.

6. Process of claim 5 in which the formulation contains from about 50% to about 65% by weight of sodium tripolyphosphate.

7. Process of claim 5 in which the water soluble non-soap organic synthetic detergent is sodium dodecylbenzene sulfonate.

8. In a process for producing strong, heavy-duty detergent tablets having a fast dissolving rate wherein a substantially dry formulation comprising about 58% by weight of sodium tripolyphosphate, about 5% by weight of sodium silicate having an $\text{Na}_2\text{O}/\text{SiO}_2$ mole ratio of 1:1 to 1:3.2, from about 5% to about 15% by weight of a water soluble non-soap organic synthetic detergent selected from the group consisting of anionic detergents selected from the group consisting of the sodium and potassium salts of alkyl sulfate, alkyl sulfonates and alkyl aryl sulfonates having from about 8 to about 18 carbon

atoms in the alkyl group, and nonionic detergents selected from the group consisting of the condensation products of about 6 to about 30 moles of ethylene oxide with one mole of an alkyl alcohol wherein the alkyl group contains from about 8 to about 18 carbon atoms, and the condensation products of about 7 to about 11 moles of ethylene oxide with one mole of an alkyl phenol wherein the alkyl group contains from about 6 to about 12 carbon atoms, from about 0.2% to about 1.5% by weight of sodium carboxymethylcellulose and sodium sulfate is pressed into tablets having a specific gravity of about 1.0, the improvement consisting essentially of formulating said substantially dry said formulation with sodium tripolyphosphate which is substantially all in the Form II crystalline configuration and which has a density of from about 0.7 to about 1.0 g./cc., whereby the dissolving rate of said tablets is increased without diminishing the dry strength of the tablets.

9. A novel heavy-duty detergent tablet having a fast dissolving rate consisting essentially of from about 35% to about 65% by weight of sodium tripolyphosphate having a density of from about 0.7 to about 1.0 g./cc. and which is substantially all in the Form II crystalline configuration, from about 3% to about 15% by weight of sodium silicate having an $\text{Na}_2\text{O}/\text{SiO}_2$ mole ratio of 1:1 to 1:3.2, from about 5% to about 15% by weight of a water soluble non-soap synthetic organic detergent selected from the group consisting of anionic detergents selected from the group consisting of the sodium and potassium salts of alkyl sulfate, alkyl sulfonates and alkyl aryl sulfonates having from about 8 to about 18 carbon atoms in the alkyl group, and nonionic detergents selected from the group consisting of the condensation products of about 6 to about 30 moles of ethylene oxide with one mole of an alkyl alcohol wherein the alkyl group contains from about 8 to about 18 carbon atoms, and the condensation products of about 7 to about 11 moles of ethylene oxide with one mole of an alkyl phenol wherein the alkyl group contains from about 6 to about 12 carbon atoms, from about 0.2% to about 1.5% by weight of sodium carboxymethylcellulose, the balance of the formulation being made up of sodium sulfate, said detergent tablets having a specific gravity of about 1.0.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,367,876

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Russell R. Keast et al.

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 1, line 17, "rate" should read -- rates --; line 32, cancel "now", second occurrence. Column 6, line 19, "provision" should read -- provisions --. Columns 7 and 8, TABLE III, ninth column, line 3 thereof, ".088" should read -- 0.88 --. Column 8, line 3, cancel the parenthesis after "+270"; same column 8, TABLE IV, first column, line 5 thereof, "(a) JT" should read -- (a) Δ T --. Column 12, line 13, after "dry" cancel "said"; line 28, cancel "group", second occurrence.

Signed and sealed this 7th day of October 1969.

(SEAL)
Attest:

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