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DETERGENT TABLET AND PROCESS FOR MAKING SAME

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This invention relates to a process for making a detergent tablet and to the tablet itself. More particularly it relates to a process for making a detergent tablet at commercially feasible production rates which will have sufficient strength to permit packaging and handling without breakage yet be capable of rapid disintegration under ordinary washing machine use.

Tablets of heavy duty synthetic detergent compositions have a number of advantages over their granular and liquid counterparts and are therefore quite desirable. The use of tablets of a premeasured quantity of material obviates the need for measuring cups used with granules and liquids and eliminates the usual problems of spilling and over or under usage encountered in employment of granules or liquids. Moreover, tablets, because of their size and compactness, are quite useful in dispensing devices such as those which can store tablets and inject them into a washing machine automatically or which can be placed in public laundries as coin operated machines. Because they are compressed, tablets take up less space than the same amount of a detergent composition in liquid or granule form.

A number of methods have been proposed to produce detergent tablets, but apparently have not been commercially successful in producing a strong, rapidly disintegrating tablet at high production rates. In general, the best method for making detergent tablets, as in most tablet making art, is to compress the ingredients in granular form into a tablet. Methods using high pressures have been proposed. These methods result in a strong tablet, but one which will not disintegrate rapidly in the ordinary washing machine. Additives to effect rapid disintegration of highly compressed tablets, such as CO_2 generators, starch or other inert materials are expensive and/or undesirable as ingredients in heavy duty detergent compositions. Methods using low pressures have been proposed and result in tablets which are rapidly disintegrating but which are not strong enough to withstand the shocks of packaging, handling, dispensing and the like.

Whether high pressures or low pressures are used, the most effective heavy duty synthetic detergent compositions have a strong tendency to stick to the dies which are used to compress the compositions into tablet form. Sticking of the detergent composition to the dies causes breakage of the tablet when the dies are moved apart and when the tablets are ejected. Additives which are used in the pharmaceutical tablet making art to prevent ingredients from adhering to the dies are undesirable as additives to the detergent compositions, are difficult to apply to die surfaces and are not effective in continuous production.

It is an object of this invention to provide a process for making detergent tablets which are strong, which disintegrate rapidly in water and which are effective detergents.

It is another object of this invention to provide a process for making detergent tablets which overcomes the problem of the adhering of the detergent composition to the dies used in compression thereby permitting rates of tablet production which are commercially feasible.

It was found that these and other objects are obtained by compressing a detergent composition so formulated that when it is compressed at moderate pressures and with rotating compressive forces, an effective detergent

tablet is produced which is strong, which quickly disintegrates on addition to the wash water and which will not adhere to the dies. After the tablet is compressed, its surface is sprayed with water and then dried to make the tablet less chalky and more abrasion resistant.

It was found that the use of moderate pressures, i.e. in the range of about 150 p.s.i. to about 350 p.s.i., results in a tablet which is strong and quickly disintegrating if a properly formulated detergent composition is used. The use of such moderate pressures overcomes the weakness problems of detergent tablets which are lightly compressed and overcomes the slow disintegration characteristics of the tablets compressed at high pressures.

In order to obtain strong, quickly disintegrating tablets, it is not sufficient to merely use moderate pressures. It was found that the granular detergent composition being compressed should have the particular characteristics hereinafter described.

The composition should comprise sodium tripolyphosphate and a non-soap, water soluble, organic synthetic detergent in the ratio range of sodium tripolyphosphate to detergent by weight of about 9:1 to about 3:1. A greater proportion of sodium tripolyphosphate reduces the detergent efficiency of the tablet and weakens the tablet since synthetic detergent in an amount corresponding to a 9:1 ratio is the minimum permissible for good detergency and the binding effect it exerts on the sodium tripolyphosphate in the tablet. If the proportion of synthetic detergent is greater than that corresponding to a 3:1 ratio the resuling tablet disintegrates too slowly in water. Speed of tablet disintegration increases with increasing amounts of sodium tripolyphosphate. The preferred ratio range is 7:1 to 4:1. The preferred composition comprises about 40% to about 80% spray dried detergent composition and about 60% to about 20% unhydrated granular sodium tripolyphosphate.

Not more than about 20% of the granular composition should have a particle size (mean particle diameter) smaller than about .2 mm. The average density of the individual particles of the composition should be in the range of about 0.5 to about 1.5, preferably 0.7 to about 1.5. The use of a minimum amount of fines and an average particle density in this range is necessary to obtain, upon compression of from about 2.5 to about 1.5 unit volumes of the aforementioned detergent compositions to one unit volume, a detergent tablet having numerous void spaces within the tablet with a total volume in the range of about 40% to about 60% of the tablet volume. This void space should be predominantly interparticulate, i.e. between the particles. It was found that a void space within this range and which is predominantly interparticulate is necessary to obtain a strong tablet which will disintegrate rapidly on addition to the wash water. A network of voids within the tablet permits the water, in which the tablet is placed for use, to enter into the tablet, displace the air in the voids and disintegrate it rapidly. The network of voids communicates with the external air. A void space of less than about 40% does not provide a network which permits sufficiently rapid disintegration (disintegration in not more than about 3 minutes, preferably not more than about 2 minutes, in an ordinary washing machine containing a load of clothes in water at not less than about 130° F.). A void space greater than about 60% provides a network which makes the tablet too weak.

The void space should be predominantly interparticulate in order to provide a network of voids which is sufficiently continuous to provide rapid disintegration according to the above minimum requirement. If the void space is predominantly intraparticulate, i.e. within the particles, such as that found in the usual low density, hollow, spray-dried granules (having a density of about .2 to about .4), such a network is not provided. Moreover, such spray dried granules are so fragile that when they are compressed, they break and an undue amount of fines is formed. As mentioned above, the particles of detergent to be compressed should not contain more than about 20% fines, i.e. those which are finer than about .2 mm. To avoid breakage and the formation of fines, and to avoid intraparticulate void space, the particles should have an average density in the above mentioned range. The composition to be compressed can be a mixture of particles of varying densities if the average density falls within this range. Fine particles are to be avoided because they fill up the interparticulate network of void space within the tablet which is necessary to provide rapid disintegration in the wash water.

The upper size limit of the particles of detergent composition to be compressed is not particularly critical. Granular sodium tripolyphosphate and granular and flake synthetic detergents of the sizes commercially available are useful as are the sizes of the usual spray dried and roll dried compositions comprising these ingredients. Flakes of synthetic detergent as large as ¼" or more in diameter, for example, can be used.

When from about 2.5 to 1.5 unit volumes of a detergent composition having the above described characteristics (ingredients, minimum amount of fines, and density) are compressed to one unit volume using pressures in the range of about 150 p.s.i. to about 350 p.s.i., a tablet having the proper network of interparticulate void space is obtained. The necessary void space in the tablet is not obtained if the unit volume of detergent composition (being compressed to one unit volume) is greater than about 2.5 or less than about 1.5. Moreover, the use of a unit volume of material greater than about 2.5 makes difficult the construction of the dies and compressing apparatus. The use of a unit volume of material less than about 1.5 does not provide enough margin to effect satisfactory compression.

During the compression of the tablet ingredients in accordance with the aforementioned conditions, the compressive forces being exerted through the dies should be rotated in order to prevent adherence of the tablet ingredients to the dies. The compressive forces exerted against both faces of the tablet should be rotated around the axis of compression with respect to the tablet, and preferably with respect to each other. That is, the complementary die sections are preferably rotated in opposite directions around a tablet. However, the dies can be rotated in the same direction, if the tablet remains stationary or the tablet could rotate with the dies at a rate of rotation different from the rate of either of the dies. The principal requirements are that both dies rotate, that the rotation be sufficient to effect a definite shearing effect between the die surface and the face of the tablet and that there be die rotation during the period of maximum compression. Rotation of the dies during this period prevents adherence of the tablet material to the dies thereby preventing tablet breakage or build up of material which leads to imperfect tablets and eventual clogging of the dies.

Speed of die rotation is not particularly critical but it is necessary that the dies be rotated through at least about 5° of arc (around the axis of compression) preferably at least about 10° to 25°, during the period of maximum compression. This much rotation is necessary to effect a shearing force between the die and the tablet face. If the rotation is insufficient to effect a shearing force, particles of detergent composition will adhere to the dies and score the tablet face or break the tablet. Die rotation through an arc greater than about 180° can be used, but is not necessary and would be difficult to effect at high production rates and might tend to cause friction heat which would adversely affect the tablet ingredients, especially those at the surface.

After the tablet has been compressed, as thus far described, it is removed from the die cavity and its surface is moistened with water in an amount of about ½% to about 5% by weight of the tablet. The water is applied preferably by a fine spray but other means such as brushing the water on or subjecting the tablet to steam can be used. The water applied to the surface of the tablet hydrates sodium tripolyphosphate on the surface of the tablet and cements tablet ingredients on the surface together to form a strong hard surface on the tablet. This surface makes the tablet non-chalky and more abrasion resistant. The strong hard surface, however, is porous and the pores communicate with the network of voids within the tablet thereby permitting rapid entry, into the tablet, of the water in which it is placed for use which, in turn results in rapid disintegration of the tablet.

The tablet is then dried if necessary to remove any excess water remaining on the surface of the tablet so that it is dry or at most slightly damp to the touch. Much of the water applied to the surface forms a hydrate with sodium tripolyphosphate. Drying can be effected in air or preferably by passing the moistened tablets through an oven on a continuous conveyor. Three pronged supports such as those used in the ceramic art are useful to support the tablets during the surface moistening and drying operations.

As mentioned above, the detergent compositions used to make the tablets of this invention should comprise sodium tripolyphosphate and a non-soap, water soluble, organic synthetic detergent in the ratio range of about 9:1 to about 3.0:1, preferably 7:1 to 4:1. These detergents may be broadly designated as anionic and nonionic, non-soap, organic synthetic detergents. The cationic non-soap synthetic detergents are not suitable because of their relative incompatibility with sodium tripolyphosphate.

The anionic non-soap synthetic detergents may be designated as water soluble salts of organic sulfuric reaction products having in their molecular structure an alkyl or acyl radical of carbon atom content within the range of about 8 to about 18 and a sulfonic acid or a sulfuric acid ester radical. Important examples of these anionic detergents are: sodium or potassium alkyl benzene sulfonate in which the alkyl group contains from about 9 to about 15 carbon atoms in either a straight chain or a branched chain which is derived from polymers of propylene; sodium and potassium alkyl glyceryl ether sulfonates, especially those ethers of higher fatty alcohols derived from the reduction of coconut oil; the reaction product of higher fatty acids with sodium or potassium isethionate, where, for example, the fatty acids are derived from coconut oil; sodium or potassium alkyl sulfonates and sulfates, especially those alkyl sulfates derived by the sulfation of coconut or tallow fatty alcohols and mixtures of such alkyl sulfates; dialkyl esters of sodium or potassium salts of sulfosuccinic acid, for example, the dihexyl ester; sodium and potassium salts of sulfated or sulfonated monoglycerides derived, for example, from coconut oil; sodium or potassium salts of the higher fatty alcohol esters of sulfocarboxylic acids, for example, the sodium salt of the lauryl alcohol ester of sulfoacetic acid; sodium or potassium salts of a higher fatty acid amide of methyl taurine in which the higher acyl radicals for example, are derived from coconut oil; and others known in the art a number being specifically set forth in U.S. Patent 2,486,921, issued to Byerly on November 1, 1949. Examples of other useful anionic non-soap synthetic detergents are acyl sarcosinates, e.g. sodium N-lauroyl sarcosinate. The sodium alkyl benzene sulfonates in which the alkyl group contains about 9 to about 15 carbon atoms are preferred in the practice of this invention.

Non-ionic non-soap synthetic detergents may be broadly classed as being constituted of a water solubilizing polyoxethylene group in chemical combination with an organic hydrophobic compound such as polyoxypropylene, alkyl phenol, the reaction product of an excess of propylene oxide and ethylene diamine, and aliphatic alcohols. The nonionic synthetic detergents have a molecular weight in the range of from about 800 to about 11,000.

For example, a well known class of nonionic detergents is made available on the market under the trade name of "Pluronic." These compounds are formed by condensing ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The molecular weight of the hydrophobic base is of the order of 1500 to 1800. The addition of polyoxyethylene radicals to this hydrophobic base increases the water solubility of the entire molecule. Liquid products are obtained up to the point where the polyoxyethylene content is about 50% of the total weight of the condensation product; higher proportions of polyoxyethylene renders the products solid in consistency. The molecular weights of Pluronic L61, L64, and F68, for example, are approximately 2000, 3000 and 8000 respectively.

Examples of other nonionic synthetic detergents useful in the present invention are: condensation products of 10 to 30 moles of ethylene oxide with one mole of an alkyl phenol containing 6 to 12 carbon atoms, either in a straight or branched chain, in the alkyl group (e.g. nonyl or octylphenol); condensation products of 10 to 30 moles of ethylene oxide with one mole of an aliphatic straight or branched chain alcohol containing 8 to 18 carbon atoms (e.g. lauryl alcohol or tallow fatty alcohol); condensation products of ethylene oxide and the reaction product of propylene oxide and ethylene diamine wherein the reaction product has a molecular weight of 2500–3000, for example and the condensation product has a polyoxyethylene content of 40% to 80%.

The detergent compositions used in this invention should comprise at least 55% of the mixture of sodium tripolyphosphate and non-soap synthetic detergent in the ratios mentioned above. Any of the other auxiliary materials generally employed with synthetic detergent compositions can also be included. These auxiliary materials can be inorganic or organic in character and can be mixed with the non-soap synthetic detergent and sodium tripolyphosphate in any suitable manner. Examples of such conventional inorganic auxiliary materials are alkali metal silicates, carbonates, sulfates and phosphates (e.g. metaphosphate, pyrophosphate and orthophosphate). Examples of organic auxiliary materials are carboxymethyl cellulose, suds builders (e.g. coconut fatty acid mono and di-ethanolamides and lauryl dimethyl amine oxide), N-chloro bleaches, tarnish inhibitors, fluorescent brightening agents, bacteriostatic agents, perfumes, coloring matter, suds depressors and the like.

The granular detergent composition to be compressed preferably contains from about ¾% to about 4½% moisture (by weight of the total amount of granules) which acts as a binder for the tablet ingredients, i.e. moisture assists the non-soap synthetic detergent in causing the compressed particles to stick together. The moisture content of the composition to be compressed can be adjusted by using ingredients which already contain moisture (e.g. spray dried granules) or if the ingredients are too dry, moisture can be added. But overall its should be less than about 4½%. A greater amount of moisture results in an undue reduction in the amount of void space when moderate compression pressures are used. Moisture can be uniformly added to the granular detergent composition, if necessary, by spraying water onto the granules in a rotary drum or a moving belt for example. The preferred moisture content is 1% to 3%.

Although water in the amounts indicated adequately achieves the desired binding, greater adherence between the compressed granules can be produced if desired by uniformly adding, to ingredients containing adequate moisture, up to about 3% (by weight of the total amount of granules) of a liquid non ionic synthetic detergent as an auxiliary binding agent. However, when the moisture content is too low to give adequate binding, the non ionic can be relied on as the primary binding agent. Thus, amounts of liquid non ionic auxiliary binding agent from 0% to about 3% can be used, and when it is used, amounts from ¼% to 1½% based on the weight of the granules are preferred. The liquid Pluronics are preferred as the auxiliary non ionic binding agent.

The uniform addition of moisture and/or liquid nonionic detergent to the granules to be compressed is intended to include those situations where the moisture and/or nonionic detergent is uniformly added to a portion of the granular material to be compressed which portion is then uniformly mixed with the remainder of the material to be compressed which can be dry and can contain no nonionic detergent. The portion containing the moisture or nonionic detergent is preferably at least 40% of the total amount of granules.

The liquid non-ionic synthetic detergent can be added to the granules by spraying it on the granules or a portion of the granules. The nonionic detergent can be sprayed on the granules in a rotary drum or a moving belt for example. The nonionic detergent being sprayed on can be diluted with water if desired.

The granular detergent composition to be compressed in accordance with the process of this invention can comprise a uniform mechanical mixture of the individual components, a homogeneous mixture such as that obtained by spray drying or roll drying an aqueous slurry of the ingredients, or a mechanical mixture of the ingredients a portion of which constitutes spray dried granules. The preferred composition to be compressed in accordance with this invention comprises about 60% to about 20% unhydrated granular sodium tripolyphosphate and about 40% to about 80% spray dried granules.

The spray dried granules contain part of the sodium tripolyphosphate in the tablet formulation, all of the non-soap synthetic detergent and any of the minor ingredients, such as sodium sulfate, moisture, non-ionic binder, brightening agents or anti-redeposition agents, which are included. Spray-dried granules in the above range are preferred because the process of spray drying is a convenient way to ensure that the minor components, if any, of the usual detergent compositions are uniformly distributed throughout the composition.

Unhydrated granular tripolyphosphate in the above range is preferred because it reduces the quantity of product to be spray dried, and more important, it permits the formation of a stronger porous surface on the tablet than is obtainable using 100% spray-dried granules. Since the process of spray drying hydrates much of the sodium tripolyphosphate in the composition being dried, tablet surface hardening by the application of water to compressed spray dried granules is effected mainly through the cementing together of the tablet ingredients and only partially through further hydration of sodium tripolyphosphate. By including unhydrated sodium tripolyphosphate in the compressed tablet ingredients a greater degree of hydration is effected during the water treatment of the tablet surface thereby making the porous tablet surface harder and stronger and more like a hard porous shell. Such a shell is nevertheless rapidly disintegrable in the wash water.

The tablet forming apparatus used to perform the process of this invention can be any device which provides a cavity for holding the granular composition to be compressed, opposing dies, both of which can be rotated with respect to the tablet faces, and a means for compression of the granular composition by exerting pressure on at least one of the dies. Tablet making machines known in the art can be adapted to these requirements. Preferably, the tablet making apparatus described in copending application of Robert V. Burt, Serial No. 2,088, filed concurrently herewith (now U.S. Patent 3,014,240), is used in the practice of this invention.

Because of the requirement of rotary compressive forces, the tablets of this invention will be round of nearly so in at least one dimension.

The following examples are given to illustrate the manner in which this invention can be practiced.

Example I

A spray-dried granular detergent composition having the following composition was formed:

	Parts
Sodium tripolyphosphate	28
Sodium alkyl benzene sulfonate (the alkyl radical being derived from polypropylene and averaging 12 carbon atoms)	20
Sodium sulfate	27
Sodium silicate having an SiO_2 to Na_2O ratio of 2:1	14
Sodium carboxymethyl cellulose	0.7
Optical brightener	0.14
A 1:1 mixture of Pluronic F68 and L64 (condensation products of ethylene oxide and the reaction product of propylene oxide and propylene glycol. They have molecular weights of about 8000 and 3000 respectively)[1]	2.8
A mixture of hydrogenated fish oil fatty acids (a suds depresor described in British Patent 808,945)	2.8
Moisture	4

[1] The Pluronic mixture was added to chemically deaerate the crutcher mix which was spray dried in order to obtain high density granules. See British Patent 812,249.

The spray dried composition had a density of 0.5. 0.92% (by weight of the spray dried granular composition) of Pluronic L64 was sprayed uniformly on the granules in a rotary drum. This nonionic detergent spray-on was used to act as a binding agent during tablet compression. The granules were then subjected to an air lift, a gravity separator and a screen whereby the moisture content was reduced to 3% and particles larger in size than 1.4 mm. and most of the particles smaller in size than .2 mm. (mean diameter) were removed. The resulting spray dried composition contained 18% particles having a size smaller than .2 mm.

990 parts of this spray dried composition were uniformly mixed with 840 parts of anhydrous granular sodium tripolyphosphate which contained 2% particles smaller in size than .2 mm. The density of the sodium tripolyphosphate was 1.3. The resulting mixture contained 1.7% moisture substantially all of which was in the spray dried granules. The mixture contained 10.6% particles smaller in size than .2 mm. The average particle density of the mixture was 0.85. 2 parts by weight of perfume was sprayed on the mixture.

6.2 cubic inches of the mixture was charged into a tablet cavity having a diameter of 2¼ inches. One tablet die was forced onto the mixture with a pressure of 200 p.s.i. to form the mixture into a tablet having a volume of 3.2 cubic inches. Both the compression die and the non-yielding die opposing this die were rotated with respect to the tablet. The non-yielding die was rotated through an arc of 10° during the period of maximum compression. The compression die was rotated in a direction opposite to that of the non-yielding die through an arc of 10° during the first part of the period of maximum compression and in the same direction as that of the compression die through an arc of 10° during the last part of the period of maximum compression. The rotating dies exerted a definite shearing force on the faces of the tablet being formed.

There was no tendency for the tablet or the tablet ingredients to adhere to the dies which, when drawn apart, permitted the tablet to be easily removed. The tablet was then placed on a three pronged support and passed through a chamber where the entire surface of the tablet was subjected to a fine spray of water. During this treatment 2½% water (by weight of the tablet) was added to the tablet surface. The tablet was then placed in a drying oven at 250° F. for 1.5 minutes. Sodium tripolyphosphate on the surface of the tablet hydrated and detergent ingredients on the surface of the tablet cemented together forming a non-chalky, abrasion-resistant, porous surface on the tablet. When the tablet was removed from the oven it was only slightly damp to the touch and was ready for packaging. It weighed two ounces and had a total moisture content of 3.3%. It had a network of voids amounting to 45% of the volume of the tablet. This network was predominantly interparticulate.

The finished tablets were economically, conveniently and successfully handled and packaged with the aid of high speed machinery. The finished tablets, when placed in an ordinary washing machine containing a load of soiled clothing in water at 130° F., disintegrated in 80 seconds. The detergency effectiveness of the tablet was excellent.

The condensation product of lauryl alcohol and 15 moles of ethylene oxide or the condensation product of 15 moles of ethylene oxide and nonyl phenol can be substituted in equivalent amounts for the Pluronic L64 sprayed on the spray-dried granules in Example I with substantially equal results.

Compression pressures of 250 p.s.i. and 300 p.s.i. can be used in the process described in Example I with substantially equal results except that the volume of the tablet will be slightly reduced and the time of tablet disintegration will be slightly greater (about 2 and 2½ minutes respectively under the above conditions).

The tablets made in accordance with Example I were made of a low sudsing detergent composition particularly useful in a tumbler-type washing machine where a large amount of foam is not desirable. A tablet can be made from a higher sudsing detergent composition by substituting, for the spray dried granular detergent composition used in Example I, a spray dried detergent composition having the following composition.

	Parts
Sodium tripolyphosphate	33
Sodium alkyl benzene sulfonate (the alkyl radical being derived from polypropylene and averaging 12 carbon atoms)	22
Sodium sulfate	19.1
Sodium silicate having an SiO_2 to Na_2O ratio of 1.6:1	11
Sodium carboxymethyl cellulose	0.65
Optical brightener	0.2
Monoethanolamide of coconut oil fatty acids	3
Sodium chloride (added to chemically dearate the crutcher mix which was spray dried in order to obtain high density granules)	7.25
Moisture	4

Example II

The following ingredients were mechanically uniformly mixed:

	Parts
Anhydrous granular sodium tripolyphosphate containing 2% particles smaller in size than .2 mm. and having a density of 1.3	55
Sodium alkyl benzene sulfonate flakes containing 14% particles smaller in size than .2 mm., containing 15% sodium sulfate, containing 1% moisture and having a density of 1.1	20
Sodium silicate having an SiO_2:Na_2O ratio of 2:1	6
Sodium sulfate	17
Monoethanolamide of coconut oil fatty acids	2.7

The average particle density of the mixture was 1.3.

6 cubic inches of this mixture was placed in a tablet cavity having a diameter of 2¼ inches. One tablet die was forced onto the mixture with a pressure of 180 p.s.i. to form the mixture into a tablet having a volume of 3.2 cubic inches. Both the compression die and the non-yielding die opposing this die were rotated with respect to the tablet during the period of maximum compression. Both dies were rotated through an arc of 10° around the axis of compression and in opposite directions. The rotating dies exerted a definite shearing force on the faces of the tablet being formed.

There was no tendency for the tablet or the tablet ingredients to adhere to the dies which, when drawn apart, permitted the tablet to be easily removed. The entire surface of the tablet was then sprayed with water in an amount equivalent to 2½% by weight of the tablet. The tablet was permitted to air dry at room temperature. Sodium tripolyphosphate on the surface of the tablet hydrated and detergent ingredients on the surface of the tablet cemented together forming a non-chalky, abrasion-resistant, porous surface on the tablet.

The tablet weighed 2 ounces and had a total moisture content of 3%. It had a network of voids amounting to 45% of the volume of the tablet. This network was predominantly interparticulate.

The tablet was strong and showed no signs of breakage when subjected to the stresses and handling typical of packaging and transporting operations. When the tablet was placed in an ordinary washing machine containing a load of soiled clothing in water at 130° F., it disintegrated in 90 seconds. The detergency effectiveness of the tablet was excellent.

Sodium lauryl glyceryl ether sulfonate, sodium N oleoyl N methyl taurate, sodium lauryl sulfate, potassium oleoyl isethionate or Pluronic F68(described above) can be substituted in equivalent amounts for the alkyl benzene sulfonate in Example I with substantially equal results.

What is claimed is:

1. In a process for making a detergent tablet the steps of (1) forming a particulate mixture comprising sodium tripolyphosphate and a water soluble, non-soap organic synthetic detergent, selected from the group consisting of anionic and non-ionic detergents in which the weight ratio of sodium tripolyphosphate to said detergent is in the range of about 9:1 to about 3:1, in which the average density of the particles is in the range of about 0.5 to about 1.5 and in which no more than about 20% of the particles are smaller than about 0.2 mm., (2) pressing from about 2.5 to about 1.5 unit volumes of said mixture into a tablet of one unit volume using a compression pressure in the range of about 150 p.s.i. to about 350 p.s.i. thereby obtaining a tablet having a network of void space in the range of about 40% to about 60% of the tablet volume, said void space being predominantly interparticulate, and using compressive forces which rotate around the axis of compression with respect to the tablet being formed and with sufficient rotation to effect a shearing force on the faces of said tablet thereby preventing adherence of said tablet to the pressing means, (3) treating the surface of the resulting tablet with water in an amount in the range of about ½% to about 5% by weight of the tablet thereby hydrating sodium tripolyphosphate on the surface of the tablet to form a non-chalky, abrasion resistant surface.

2. The process of claim 1, wherein the said particulate mixture comprises about 20% to about 60% unhydrated granular sodium tripolyphosphate and about 80% to about 40% of a mixture of sodium tripolyphosphate and the said non-soap synthetic detergent in form of spray-dried granules, the weight ratio of sodium tripolyphospate to said non-soap synthetic detergent in the total particulate mixture ranging from 7:1 to 4:1, the compressive forces are rotated around the axis of compression through an arc of at least about 5°, but not more than about 180°, and the tablet treated in step (3) is dried.

3. The process of claim 2 wherein the said synthetic detergent is an alkyl benzene sulfonate, the alkyl radical containing about 9 to about 15 carbon atoms.

4. In a process for making a detergent tablet the steps of (1) forming a particulate mixture comprising sodium tripolyphosphate and a normally solid, water soluble, non-soap, organic synthetic detergent, selected from the group consisting of anionic and non-ionic detergents, in which the weight ratio of sodium tripolyphosphate to said detergent is in the range of about 9:1 to about 3.0:1, in which the density of particles is in the range of about 0.5 to about 1.5, in which no more than about 20% of the particles are smaller than about 0.2 mm., and in which at least a portion of the particles have a coating of a liquid nonionic synthetic detergent in an amount equal to about 3% of the mixture, said liquid nonionic detergent being constituted of a water solubilizing polyoxyethylene group in chemical combination with an organic hydrophobic compound, the mixture containing from about ¾% to about 4½% moisture, (2) pressing from about 2.5 to about 1.5 unit volumes of said mixture into a tablet of one unit volume using a compression pressure in the range of about 150 p.s.i. to about 350 p.s.i. thereby obtaining a tablet having a network of void space in the range of about 40% to about 60% of the tablet volume, said void space being predominantly interparticulate, and using compressive forces which rotate around the axis of compression with respect to the tablet being formed and with sufficient rotation to effect a shearing force on the faces of said tablet thereby preventing adherence of said tablet to the pressing means, (3) treating the surface of the resulting tablet with water in an amount in the range of about ½% to about 5% by weight of the tablet thereby hydrating sodium tripolyphosphate on the surface of the tablet to form a non-chalky, abrasion resistant surface.

5. The process of claim 4 wherein the said particulate mixture comprises about 20% to about 60% unhydrated granular sodium tripolyphosphate and about 80% to about 40% of a mixture of sodium tripolyphosphate and the said non-soap synthetic detergent in the form of spray-dried granules, the weight ratio of sodium tripolyphosphate to said non-soap synthetic detergent in the total particulate mixture ranging from 7:1 to 4:1, the compressive forces are rotated around the axis of compression through an arc of at least about 5°, but not more than about 180°, and the tablet treated in step (3) is dried.

6. The process of claim 5 wherein the said synthetic detergent is an alkyl benzene sulfonate, the alkyl radicals containing about 9 to about 15 carbon atoms, and the said liquid nonionic detergent is the condensation product of ethylene oxide and the reaction product of propylene oxide and propylene glycol wherein the polyoxyethylene content is up to about 50% of the total weight of the condensation product.

7. In a process for making a detergent tablet the steps of (1) forming a particulate mixture comprising sodium tripolyphosphate and a water-soluble, non-soap, organic synthetic detergent selected from the group consisting of anionic and non-ionic detergents, in which the weight ratio of sodium tripolyphosphate to said detergent is in the range of about 9:1 to about 3:1, in which the average density of the particles is in the range of about 0.5 to about 1.5 and in which no more than about 20% of the particles are smaller than about 0.2 mm., (2) pressing from about 2.5 to about 1.5 unit volumes of said mixture into a tablet of one unit volume using a compression pressure in the range of about 150 p.s.i. to about 350 p.s.i. thereby obtaining a tablet having a network of void space in the range of about 40% to about 60% of the tablet volume, said void space being predominantly interparticulate, and using compressive forces which rotate around the axis of the compression with respect to the tablet being formed and with sufficient rotation to effect a shearing force on the faces of said tablet thereby preventing adherence of said tablet to the pressing means.

8. A round heavy-duty laundry detergent tablet consisting essentially of a compressed water-soluble particulate mixture consisting essentially of about 20% to about 60% granular sodium tripolyphosphate, prior to compression all of which was unhydrated, and about 80% to about 40% of a mixture, in the form of spray-dried granules, of sodium tripolyphosphate and a normally solid water-soluble non-soap organic synthetic detergent selected from the group consisting of anionic

and nonionic detergents, the weight ratio of sodium tripolyphosphate to said non-soap synthetic detergent in the total particulate mixture ranging from about 9:1 to about 3:1, said tablet having a network of void space, which is predominantly interparticulate, in the range of about 40% to about 60% of the tablet volume, said tablet having a strong, hard, non-chalky porous surface of hydrated sodium tripolyphosphate and cemented tablet ingredients, the pores in said surface communicating with said network, said particulate mixture having been compressed at a pressure in the range of about 150 p.s.i. to about 350 p.s.i.

9. The detergent tablet of claim 8 wherein said spray-dried granules in said particulate mixture have a coating of a liquid nonionic synthetic detergent binding agent, said agent being present in an amount sufficient to exert a binding effect, but not more than about 3% of the weight of the compressed particles, said liquid nonionic agent being constituted of a water solubilizing polyoxyethylene group in chemical combination with an organic hydrophobic compound.

10. The detergent tablet of claim 8 wherein the said detergent is an alkyl benzene sulfonate in which the alkyl radical contains about 9 to about 15 carbon atoms.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,081,267 March 12, 1963

Richard P. Laskey

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 10, line 4, after "equal" insert -- to from 0% --; column 12, line 6, after "said" insert -- synthetic --.

Signed and sealed this 1st day of October 1963.

;EAL)
;ttest:

INEST W. SWIDER
ttesting Officer

DAVID L. LADD
Commissioner of Patents