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(54) Detergent composition

(57) Tablets of detergent composition incorporate particles which contain sodium tripolyphosphate with a high proportion of the phase I form, and desirably from 2 to 4% prehydration. This promotes rapid disintegration and dissolution at the time of use, even when a large proportion of further, detergent-containing particles is present.

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Description

This invention relates to cleaning compositions in the form of tablets, e.g. for use in fabric washing.

Detergent compositions in tablet form are described, for example, in GB 911204 (Unilever), US 3953350 (Kao), JP 60-015500A (Lion), JP 60-135497A (Lion) and JP 60-135498A (Lion); and are sold commercially in Spain. Tablets have several advantages over powdered products: they do not require measuring and are thus easier to handle and dispense into the washload, and they are more compact, hence facilitating more economical storage.

Detergent tablets are generally made by compressing or compacting a detergent powder, which includes detergent active and detergency builder. It is desirable that tablets have adequate strength when dry, yet disperse and dissolve quickly when added to wash water. Detergent included in such tablets functions as a binder for other solids.

It is known to include materials whose function is to enhance disintegration of tablets when placed in wash water. Some tablets which are sold commercially incorporate urea for this purpose. Urea has a very high solubility in water exceeding 100g per 100ml water at 20°C.

Sodium tripolyphosphate is very well known as a sequestering builder in detergent compositions.

It exists in a hydrated form and two crystalline anhydrous forms. These are the normal crystalline anhydrous form, known as phase II which is the low temperature form, and phase I which is stable at high temperature. The conversion of phase II to phase I proceeds fairly rapidly on heating above the transition temperature, which is about 420°C, but the reverse reaction is slow. Consequently phase I sodium tripolyphosphate is metastable at ambient temperature.

Phase I material is known to hydrate to the hexahydrate more rapidly than phase II material. It is also known to dissolve somewhat more rapidly when there is no obstacle to dispersion in the solution. However, during dissolution, this phase I material can form a viscous or solid mass which, in a confined space can hinder dissolution. For instance when making a slurry for spray drying, phase I tripolyphosphate can form so-called "grit", which is a mass of interlocked crystals.

GB-A-1375131 includes a comparison of the dissolution of sodium tripolyphosphate samples containing 38% and 75% phase I material. Each also contained 5% water of hydration. The sample with 38% of phase I sodium tripolyphosphate dissolved very slightly faster, but both formed a viscous mass during dissolution. A sample with 4% phase I and 12% water of hydration dissolved even more slowly. This document teaches that a powdered dishwasher composition should contain partially hydrated sodium tripolyphosphate with a phase I content which is between 4 and 14% of the anhydrous tripolyphosphate.

EP-A-178986 teaches that sodium tripolyphosphate intended for incorporation into a spray-dried detergent powder should have over 50% phase I material, with some water of hydration present.

US-A-4362641 teaches that granular sodium tripolyphosphate for a dishwasher composition should contain some phase I material to enhance absorption of free water during manufacture and packing.

EP-A-219314 and EP-A-220024 teach that phase I anhydrous tripolyphosphate should be avoided in a spray-dried component of a particulate detergent composition which is formulated to dissolve rapidly.

For tablets, where speed of dissolution of the entire tablet is an important objective, it is important that water should penetrate into the tablet and dissolve material from the tablet interior, as well as at the tablet exterior. It would therefore seem desirable that any material incorporated as an aid to dissolution should have high solubility and should not impede contact with water by the formation of a poorly mobile mass which could block pores in the tablet.

Surprisingly, we have now found that the speed of disintegration of tablets can be enhanced by including sodium tripolyphosphate with a substantial content of phase I. Moreover, this is effective even when this sodium tripolyphosphate is not directly mixed with detergent active but is mixed with more than its own weight of other particulates.

A tablet of the invention may be either homogeneous or heterogeneous. In the present specification, the term "homogeneous" is used to mean a tablet produced by compaction of a single particulate composition, but does not imply that all the particles of that composition will necessarily be of identical composition. The term "heterogeneous' is used to mean a tablet consisting of a plurality of discrete regions, for example layers, inserts or coatings, each derived by compaction from a particulate composition.

Broadly, this invention provides a tablet of compacted particulate detergent composition, containing one or more detergent-active compounds together with sodium tripolyphosphate and other ingredients, characterised in that the tablet or a region thereof comprises particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in said particles.

It is strongly preferred that the sodium tripolyphosphate in the particles is partially hydrated. These particles may contain sodium tripolyphosphate as their only ingredient, or at least as a high proportion of the particles, such as at least 80% or 90% by weight. However, it is conceivable that they could contain sodium tripolyphosphate mixed with some other constituent of the overall tablet composition.

Generally, the composition within the tablet, or region thereof, will contain one or more detergent active compounds together with other detergent ingredients. For this, the tablet or region thereof may contain further particles which include detergent, such as a spray dried or granulated detergent composition, plus the said particles containing phase

l sodium tripolyphosphate, possibly accompanied by additional particulate ingredients of the overall composition. It is a feature of this invention that the quantity of the particles containing phase I sodium tripolyphosphate can be less than the quantity of the particles which include detergent.

So, in a first aspect this invention provides a tablet of compacted particulate detergent composition, containing one or more detergent-active compounds together with sodium tripolyphosphate and other ingredients, characterised in that the tablet or a region thereof comprises particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration distributed throughout the tripolyphosphate in an amount between 1% and 5% by weight of the sodium tripolyphosphate in the particles.

In a second aspect, the invention provides a process for making such a tablet, which comprises mixing (a) particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the particles, wherein the sodium tripolyphosphate in the particles contains water of hydration in an amount between 1% and 5% by weight of the sodium tripolyphosphate in the particles, with (b) other material to form a particulate detergent composition and compacting the composition to form a tablet or region thereof.

In a further aspect the invention provides a tablet of compacted particulate detergent composition, containing one or more detergent-active compounds together with sodium tripolyphosphate and other ingredients, characterised in that the tablet or a region thereof comprises:

i) particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration in an amount between 0.5% and 5% by weight of the sodium tripolyphosphate in the particles; and

ii) further particles which contain detergent active mixed with other material, preferably including additional sodium tripolyphosphate, these further particles being present in a greater quantity than the tripolyphosphate-containing first said particles.

Features of this invention, suitable materials and preferences will now be described in more detail.

Sodium Tripolyphosphate with High Phase I Content

As mentioned above, sodium tripolyphosphate can be converted to the phase I form by heating to above the transition temperature at which phase II anhydrous sodium polyphosphate is transformed into the phase I form. A process for the manufacture of particles containing a high proportion of the phase I form of sodium tripolyphosphate by spray drying below 420°C is given in US-A-4536377.

Suitable material is commercially available. Suppliers include Rhone-Poulenc, Courbevoie, France and Albright & Wilson, Warley, West Midlands, UK. The sodium tripolyphosphate should be partially hydrated, but the phase I anhydrous form should also be present. Thus, the sodium tripolyphosphate in the particles may incorporate from 0.5% up to, at least, 5% (by weight of the sodium tripolyphosphate in these particles) of water of hydration. The extent of hydration is desirably from 1% to 4% or 5% by weight.

The sodium tripolyphosphate in these particles is preferably hydrated by a process which leads to a homogeneous distribution of the water of hydration within the tripolyphosphate.

This can be accomplished by exposing anhydrous sodium tripolyphosphate to steam or moist air. The particles preferably consist solely of sodium tripolyphosphate with a high content of the phase I form.

The particles preferably contain sodium tripolyphosphate in a porous form so as to have high surface area. This can be achieved by spray drying the tripolyphosphate as a mixture with a blowing agent, that is a compound such as ammonium carbonate which decomposes to yield a gas during the course of the spray drying. This gives the dried material a porous structure, with higher surface area than hollow beads of tripolyphosphate obtained without blowing agent.

The particles which contain or consist of sodium tripolyphosphate preferably have a small mean particle size, such as not over 300μm, better not over 250μm. Small particle size can if necessary be achieved by grinding.

Uniform prehydration, high phase I content, porosity and small particle size all promote rapid hydration when the tripolyphosphate comes into contact with water. A standard test for the rapidity of hydration is the Olten test. It is desirable that in such a test the tripolyphosphate reaches 90% of the final value (ie 90% of complete hydration when exposed to water at 80°C) within 60 seconds.

"Rhodiaphos HPA 3.5" is a grade of sodium tripolyphosphate from Rhone-Poulenc which has been found to be particularly suitable. It consists of porous particles of small particle size (mean size below 250 μm) with 70% phase I and prehydrated with 3.5% water of hydration.

Preferably the said particles containing sodium tripolyphosphate with more than 40% of phase I material provide

this phase I tripolyphosphate as at least 3%, better at least 8% by weight of the tablet or region thereof. More preferably they provide sodium tripolyphosphate, including the phase I tripolyphosphate, in a quantity which is from 12% up to 30% or 35% by weight of the tablet or region thereof. A quantity of at least 25% has been found useful in some tablets.

The remainder of the tablet composition may include additional sodium tripolyphosphate. This may be in any form, including anhydrous sodium tripolyphosphate with a high content of the phase II form, hydrated sodium tripolyphosphate or some combination of the two.

The total quantity of sodium tripolyphosphate, in all forms, present in the tablet composition will generally lie in a range from 15% to 60% by weight of the tablet. Therefore it will be appreciated that the overall quantity of sodium tripolyphosphate may be provided at least partially by other material in addition to the said particles.

Detergent-Active Compounds

Tablet compositions of this invention generally contain one or more organic detergent-active compounds. Preferably these provide from 5% to 50% by weight of the composition of the overall tablet or of a region thereof, more preferably from 8% or 9% by weight of the composition up to 40% or 50% by weight. Organic detergent-active compounds may be anionic (soap or non-soap), cationic, zwitterionic, amphoteric, nonionic or a combination of these, especially combinations of non-soap anionic, nonionic and possibly soap.

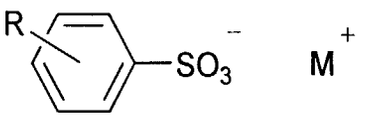
Anionic detergent-active compounds may be present in an amount from 0.5% to 50% by weight, preferably from 2% or 4% up to 30% or 40% by weight of the composition of the tablet or of a tablet region.

Synthetic (i.e. non-soap) anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

Primary alkyl sulphate having the formula:

$$ROSO_3^- M^+$$

in which R is an alkyl or alkenyl chain of 8 to 18 carbon atoms especially 10 to 14 carbon atoms and M^+ is a solubilising cation, is commercially significant as an anionic detergent active. Linear alkyl benzene sulphonate of the formula



where R is linear alkyl of 8 to 15 carbon atoms and M^+ is a solubilising cation, especially sodium, is also a commercially significant anionic detergent active.

Frequently, such linear alkyl benzene sulphonate or primary alkyl sulphate of the formula above, or a mixture thereof will be the desired anionic detergent and may provide 75 to 100 wt. % of any anionic non-soap detergent in the composition.

In some forms of this invention the amount of non-soap anionic detergent lies in a range from 5 wt % to 15 wt % of the composition of the tablet or a tablet region.

It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rapeseed oil.

Suitable nonionic detergent compounds which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide ether alone or with propylene oxide.

Specific nonionic detergent compounds are alkyl (C_{8-22}) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C_{8-20} primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-diamine. Other nonionic detergent compounds include alkyl polyglycosides, long-chain amine oxides, tertiary phosphine oxides, and dialkyl sulphoxides.

Especially preferred are the primary and secondary alcohol ethoxylates, especially the C_{9-11} and C_{12-15} primary and secondary alcohols ethoxylated with an average of from 3 or 5 moles to 20 moles of ethylene oxide per mole of

alcohol.

In certain forms of this invention the amount of nonionic detergent lies in a range from 4% to 40%, better 4% or 5% to 30% by weight of the composition of the tablet or a tablet region.

Many nonionic detergent-active compounds are liquids. These may be absorbed onto particles of the composition.

Detergency Builder

The composition of a tablet or of a region of a tablet will generally contain from 15% to 80%, more usually 15% to 60% by weight of detergency builder. This may be provided solely by sodium tripolyphosphate, or part of it may be provided by other material with water-softening properties.

Conceivably this could be a water-insoluble aluminosilicate or a layered sodium silicate as described in GB-A-1429143 and US-A-4664839 respectively.

More probably it will be a water-soluble builder. Water-soluble builders may be organic or inorganic. Inorganic builders that may be present include alkali metal (generally sodium) carbonate; while organic builders include polycarboxylate polymers, such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphonates, monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono- di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates and hydroxyethyliminodiacetates.

Tablets may include polycarboxylate polymers, more especially polyacrylates and acrylic/maleic copolymers which can function as builders and also inhibit unwanted deposition onto fabric from the wash liquor.

In preferred forms of this invention the composition which is compacted into tablets or regions thereof contains

i) the above mentioned particles which contain phase I sodium tripolyphosphate
ii) further particles which contain detergent active and other ingredients, and optionally
iii) yet further particulate ingredients.

The particles (ii) may be present in a quantity which is greater than the quantity of particles (i). It is a feature of this invention that particles (i) give effective disintegration even under these circumstances.

If a composition is required to include nonionic detergent, this is preferably incorporated in particles (ii) along with anionic detergent. It is preferred that particles (i) are substantially free of organic detergent compounds (ie contain less than 5% of their own weight of detergent, better none at all).

Incorporating the detergent active into further particles (ii) together with other ingredients has the advantage that these and other particles in the composition can form a reasonably free flowing particulate composition, which is useful for handling prior to compaction into tablet form. The particles (ii) may be made by conventional methods for the manufacture of particulate detergent compositions such as spray drying or granulation.

Binder

Tablets of this invention may include an organic water-soluble polymer, applied as a coating to some of the constituent particles, and serving as a binder when the particles are compacted into tablets. This polymer may be a polycarboxylate such as those mentioned above.

It is preferred that such a binder material, if present, should melt at a temperature of at least 35°C, better 40°C or above, which is above ambient temperatures in many temperate countries. For use in hotter countries it will be preferable that the melting temperature is somewhat above 40°C, so as to be above the ambient temperature.

For convenience the melting temperature of the binder material should be below 80°C.

Preferred binder materials are synthetic organic polymers of appropriate melting temperature, especially polyethylene glycol. Polyethylene glycol of average molecular weight 1500 (PEG 1500) melts at 45°C and has proved suitable. Polyethylene glycol of higher molecular weight, notably 4000 or 6000, can also be used.

Other possibilities are polyvinylpyrrolidone, and polyacrylates and water-soluble acrylate copolymers.

The binder may suitably be applied to the particles by spraying, e.g. as a solution or dispersion. If used, the binder is preferably used in an amount within the range from 0.1% to 10% by weight of the tablet composition, more preferably the amount is at least 1% or even at least 3% by weight of the tablets. Preferably the amount is not over 8% or even 6% by weight unless the binder serves some other additional function.

Tablets may include other ingredients which aid tableting. Tablet lubricants include calcium, magnesium and zinc soaps (especially stearates), talc, glyceryl behapate, sugar Myvatex (Trade Mark) TL ex-Eastman Kodak, polyethylene glycols, and colloidal silicas (for example, Alusil (Trade Mark) ex-Crosfield Chemicals Ltd).

Bleach System

Tableted detergent compositions according to the invention may contain a bleach system. This preferably comprises one or more peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. If any peroxygen compound is present, the amount is likely to lie in a range from 10% to 25% by weight of the composition.

Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate, advantageously employed together with an activator. Bleach activators, also referred to as bleach precursors, have been widely disclosed in the art. Preferred examples include peracetic acid precursors, for example, tetraacetylethylene diamine (TAED), now in widespread commercial use in conjunction with sodium perborate; and perbenzoic acid precursors. The quaternary ammonium and phosphonium bleach activators disclosed in US 4751015 and US 4818426 (Lever Brothers Company) are also of interest. Another type of bleach activator which may be used, but which is not a bleach precursor, is a transition metal catalyst as disclosed in EP-A-458397, EP-A-458398 and EP-A549272. A bleach system may also include a bleach stabiliser (heavy metal sequestrant) such as ethylenediamine tetramethylene phosphonate and diethylenetriamine pentamethylene phosphonate.

As indicated above, if a bleach is present and is a water-soluble inorganic peroxygen bleach, the amount may well be from 10% to 25% by weight of the composition.

Other Detergent Ingredients

The detergent tablets of the invention may also contain one of the detergency enzymes well known in the art for their ability to degrade and aid in the removal of various soils and stains. Suitable enzymes include the various proteases, cellulases, lipases, amylases, and mixtures thereof, which are designed to remove a variety of soils and stains from fabrics. Examples of suitable proteases are Maxatase (Trade Mark), as supplied by Gist-Brocades N.V., Delft, Holland, and Alcalase (Trade Mark), and Savinase (Trade Mark), as supplied by Novo Industri A/S, Copenhagen, Denmark. Detergency enzymes are commonly employed in the form of granules or marumes, optionally with a protective coating, in amount of from about 0.1% to about 3.0% by weight of the composition; and these granules or marumes present no problems with respect to compaction to form a tablet.

The detergent tablets of the invention may also contain a fluorescer (optical brightener), for example, Tinopal (Trade Mark) DMS or Tinopal CBS available from Ciba-Geigy AG, Basel, Switzerland. Tinopal DMS is disodium 4,4'bis-(2morpholino-4-anilino-s-triazin-6-ylamino) stilbene disulphonate; and Tinopal CBS is disodium 2,2'-bis-(phenylstyryl) disulphonate.

An antifoam material is advantageously included, especially if the detergent tablet is primarily intended for use in front-loading drum-type automatic washing machines. Suitable antifoam materials are usually in granular form, such as those described in EP 266863A (Unilever). Such antifoam granules typically comprise a mixture of silicone oil, petroleum jelly, hydrophobic silica and alkyl phosphate as antifoam active material, sorbed onto a porous absorbed water-soluble carbonate-based inorganic carrier material. Antifoam granules may be present in an amount up to 5% by weight of the composition.

It may also be desirable that a detergent tablet of the invention includes an amount of an alkali metal silicate, particularly sodium ortho-, meta- or disilicate. The presence of such alkali metal silicates at levels, for example, of 0.1 wt % to 10 wt %, may be advantageous in providing protection against the corrosion of metal parts in washing machines, besides providing some measure of building and giving processing benefits in manufacture of the particulate material which is compacted into tablets.

Further ingredients which can optionally be employed in the detergent tablet of the invention include anti-redeposition agents such as sodium carboxymethylcellulose, straightchain polyvinyl pyrrolidone and the cellulose ethers such as methyl cellulose and ethyl hydroxyethyl cellulose, fabric-softening agents; heavy metal sequestrants such as EDTA; perfumes; and colorants or coloured speckles.

Particle Size and Distribution

A detergent tablet of this invention, or a discrete region of such a tablet, is a matrix of compacted particles.

Preferably the particulate composition has an average particle size in the range from 200μm to 2000μm, more preferably from 250μm to 1400μm. Fine particles, smaller than 180μm or 200μm may be eliminated by sieving before tableting, if desired, although we have observed that this is not always essential.

While the starting particulate composition may in principle have any bulk density, the present invention is especially relevant to tablets made by compacting powders of relatively high bulk density, because of their greater tendency to exhibit disintegration and dispersion problems. Such tablets have the advantage that, as compared with a tablet derived from a low bulk density powder, a given dose of composition can be presented as a smaller tablet.

Thus the starting particulate composition may suitably have a bulk density of at least 400g/litre, preferably at least 500g/litre, and advantageously at least 700g/litre.

Granular detergent compositions of high bulk density prepared by granulation and densification in a high-speed mixer/granulator, as described and claimed in EP 340013A (Unilever), EP 352135A (Unilever), and EP 425277A (Unilever), or by the continuous granulation/densification processes described and claimed in EP 367339A (Unilever) and EP 390251A (Unilever), are inherently suitable for use in the present invention.

As mentioned earlier, a tablet may be either homogeneous or heterogeneous.

It is envisaged that the particles with a high content of phase I sodium tripolyphosphate will preferably be distributed throughout the whole of a homogeneous tablet. However, it is possible that they will be contained within one or more discrete regions of a heterogeneous tablet, such as a layer or an insert large enough to constitute from 10% to 90% of the weight of the whole tablet. The presence of such a layer or insert could assist break up of the entire tablet when placed in water. A heterogenous tablet may well contain overall the abovementioned proportions of from 5 to 50 wt% detergent active and from 10 or 15 to 80 wt% detergency builder.

Tableting

Tableting entails compaction of a particulate composition. A variety of tableting machinery is known, and can be used. Generally it will function by stamping a quantity of the particulate composition which is confined in a die.

Tableting may be carried out at ambient temperature or at a temperature above ambient which may allow adequate strength to be achieved with less applied pressure during compaction. In order to carry out the tableting at a temperature which is above ambient, the particulate composition is preferably supplied to the tableting machinery at an elevated temperature. This will of course supply heat to the tableting machinery, but the machinery may be heated in some other way also.

If any heat is supplied, it is envisaged that this will be supplied conventionally, such as by passing the particulate composition through an oven, rather than by any application of microwave energy.

The size of a tablet will suitably range from 10 to 160 grams, preferably from 15 to 60 g, depending on conditions of intended use, and whether it represents a dose for an average load in a fabric washing or dishwashing machine or a fractional part of such a dose. Tablets may be of any shape. However, for ease of packaging they are preferably blocks of substantially uniform cross-section, such as cylinders or cuboids. Overall density of a tablet preferably lies in a range from 1040 or 1050 grams per litre up to 1300 gm/litre. The tablet density may well lie in a range up to no more than 1250 or even 1200 gm/litre.

Example 1

Tablets for use in fabric washing were made, starting with a spray-dried base powder of the following composition:

Sodium linear alkylbenzene sulphonate	11.83%
Sodium tripolyphosphate, type 1A[1]	44.83%
Nonionic detergent[2]	7.88%
Sodium silicate	11.83%
Soap	1.13%
Sodium carboxymethyl cellulose	0.90%
Acrylate/maleate copolymer	3.20%
Sodium sulphate and minor ingredients	3.00%
Water balance to	100%

1. This contained less than 30% of the phase I form of anhydrous sodium tripolyphosphate.

2. C_{13-15} fatty alcohol 7EO.

This powder was mixed with particles of sodium tripolyphosphate specified to contain 70% phase I form and contain 3.5% water of hydration (Rhodia-Phos HPA 3.5 from Rhone-Poulenc) and other detergent ingred-ients as tabulated below. As a comparative composition the base powder was mixed with urea and other detergent ingredients.

The two compositions thus contained the following percentages by weight:

	Example 1	Comparative A
Base powder	63.25	63.25

(continued)

	Example 1	Comparative A
Sodium perborate tetrahydrate	10.40	10.40
Tetraacetylethylenediamine (TAED) granules	4.00	4.00
Anti-foam granule	2.00	2.00
Enzymes	0.85	0.85
Phosphonate	0.50	0.50
Sodium carbonate	3.60	3.60
HPA sodium tripolyphosphate	15.00	None
Urea	None	15.00

35g portions of each composition were made into cylindrical tablets of 44mm diameter, using a Carver hand press.

The strength of these tablets was measured using an Instron universal testing machine to compress a tablet until fracture. The value of diametral fracture stress (DFS) was then calculated using the equation:

$$\sigma = \frac{2P}{\pi Dt}$$

where σ is the diametral fracture stress in Pascals, P is the applied load in Newtons to cause fracture, D is the tablet diameter in metres and t is the tablet thickness in metres.

The break-up, dispersion and dissolution of tablets was measured by a test procedure in which a tablet is placed on a plastic sieve with 2mm mesh size which was immersed in 9 litres of demineralised water at ambient temperature of 22°C and rotated at 200rpm. The water conductivity was monitored until it reached a constant value.

The time for break up and dispersion of the tablets was taken as the time (T_{90}) for change in the water conductivity to reach 90% of its final magnitude. This was also confirmed by visual observation of the material remaining on the rotating sieve. The results were:

	Example 1	Comparative A
Tablet strength (kPa)	19.50	21.9
Tablet dissolution T_{90} (min)	3.35	13.4

This shows that tablets of this Example dissolved much faster than the comparative tablets of similar strength made with urea.

Example 2

Further tablets were prepared using the same spray dried base powder as in Example 1. Tablets embodying the invention and comparative tablets were prepared with a formulation as follows:-

Ingredient	% by weight
Base powder	62.00
Sodium perborate tetrahydrate	12.60
TAED granules	3.00
Antifoam granule	2.00
Enzymes	0.85
Phosphonate	0.50
Perfume	0.45
Variable, soluble ingredient	18.60

Various soluble materials were used as the variable, soluble ingredient, as set out in the table below. Tablets were

made as in Example 1, using varying levels of compaction force. The resulting DFS and T_{90} values are also given in the table below:

Variable Soluble Ingredient	Compaction force (kN)	DFS (kPa)	$T_{90\%}$ dissolution (minutes)
HPA (high phase I)	2.0 3.0 4.0	11.0 16.0 24.7	4.55 5.00 9.00
Empiphos D (low phase I)	2.0 3.0	6.0 11.4	7.00 16.00
Empiphos E (high phase I)	4.0	20.0	7.00
Urea	2.0 3.0	14.8 25.2	4.55 8.00
Na-sulphate fine granular	2.0	10.4	18.90

HPA grade sodium tripolyphosphate (as used also in Example 1) is specified to contain 70% of phase I, and to contain 3.5% water of hydration.

Empiphos D, available from Albright & Wilson, has a very high content of phase II tripolyphosphate. The content of phase I is 10% and the water content is 0.2%.

Empiphos E, available from Albright & Wilson is specified as having 50% of phase I, and to contain 1% water of hydration.

These results show that sodium tripolyphosphate of high phase I content enhance dissolution, compared to similar tablets containing sodium sulphate and sodium tripolyphosphate of low phase I content. Unlike urea, it functions as a detergency builder in the wash liquor.

Example 3

Further tablets for use in fabric washing were made starting with a spray dried base powder of the following composition:

Ingredient	Parts by weight	Percent by weight
Sodium linear alkyl benzene sulphonate	11	24.4
Nonionic surfactant	5	11.1
Soap	0.2	0.4
Sodium tripolyphosphate type 1A*	16.8	37.3
Acrylate/maleate copolymer	1.5	3.3
Sodium carboxymethylcellulose	0.3	0.7
Sodium silicate	4	8.9
Fluorescer	0.2	0.4
Water	6	13.3
TOTAL	45	100

*Less than 30% phase I form

Tablets were prepared with the following three formulations in which different types of sodium tripolyphosphate were used:

INGREDIENT	% BY WEIGHT		
EXAMPLE NO.	3A	3B	3C
Base powder as above	45	45	45
Sodium percarbonate	15	15	15
Tetraacetyethylenediamine (TAED) granules	4	4	4
Antifoam granules	2	2	2
Perfume, enzymes and other ingredients	4	4	4
HPA sodium tripolyphosphate 70% phase I; 3.5% water of hydration	30	0	0
Thermphos NW tripolyphosphate 84% phase I; no water of hydration	0	30	0
Thermphos NW tripolyphosphate 84% phase I; 3.5% water of hydration	0	0	30

The different grades of sodium tripolyphosphate which were used were as follows:
HPA tripolyphosphate was the same as in Examples 1 and 2.

Thermphos NW, available from Hoechst, was used in grades containing 84% phase I and either 3.5% water of hydration or no water of hydration.

These various compositions were made into detergent tablets as in example 1 so that all tablets had a weight of 35g and a diameter of 44mm. The tablets were made with sufficient compaction force to produce tablets of very similar strength. All tablets had diametral fracture stress of about 12kPa.

The time for break up and dispersion of these tablets was measured as in example 1 and the results obtained are set out in the following table:

	Tripolyphosphate	Phase I %	Water of hydration %	$T_{90\%}$ dissolution (minutes)
3A	HPA	70.0	3.5	2.5
3B	Thermphos NW	84.0	0	7.0
3C	Thermphos NW	84.0	3.5	4.5

These results provide further demonstration that by incorporating sodium tripolyphosphate with a high content of phase I, and some prehydration, fast rates of dissolution and disintegration are achieved.

Claims

1. A tablet of compacted particulate detergent composition, containing one or more detergent-active compounds together with sodium tripolyphosphate and other ingredients, characterised in that the tablet or a region thereof comprises particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration distributed throughout the tripolyphosphate in an amount between 1% and 5% by weight of the sodium tripolyphosphate in the particles.

2. A tablet according to claim 1, wherein the sodium tripolyphosphate in the said particles has a content of the phase I form which is at least 50% by weight of the sodium tripolyphosphate in the said particles.

3. A tablet according to any one of the preceding claims wherein the amount of said water of hydration lies in a range from 2% to 4% by weight of the sodium tripolyphosphate in the particles.

4. A tablet according to any one of the preceding claims wherein the tripolyphosphate in said particles is porous.

5. A tablet according to any one of the preceding claims wherein the tripolyphosphate in said particles hydrates to at least 90% of its fully hydrated state within one minute when said particles come into contact with water at 80°C.

6. A tablet according to any one of the preceding claims wherein said particles have a mean particle size not greater

than 300μm.

7. A tablet according to any one of the preceding claims, wherein the sodium tripolyphosphate in said particles provides from 12% to 35% by weight of the tablet or region thereof.

8. A tablet according to any one of the preceding claims wherein the tablet or region thereof also contains further particles which contain detergent active mixed with other material, these further particles being present in a greater quantity than the tripolyphosphate-containing first said particles.

9. A tablet of compacted particulate detergent composition, containing one or more detergent-active compounds together with sodium tripolyphosphate and other ingredients, characterised in that the tablet or a region thereof comprises:

i) particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration in an amount between 0.5% and 5% by weight of the sodium tripolyphosphate in the particles; and
ii) further particles which contain detergent active mixed with other material, these further particles being present in a greater quantity than the tripolyphosphate-containing first said particles.

10. A tablet according to claim 8 or claim 9 wherein the tripolyphosphate-containing first said particles are substantially free of organic detergent-active, so as to contain not more than 5% of their own weight of organic detergent-active, preferably none.

11. A tablet according to any one of claims 8 to 12 wherein said detergent-containing further particles contain both anionic and nonionic organic detergent active together with sodium tripolyphosphate.

12. A tablet according to any one of the preceding claims, wherein the tablet or region thereof contains from 5% to 50% by weight of organic detergent-active and 10% to 80% by weight of detergency builder including sodium tripolyphosphate.

13. A tablet according to any one of the preceding claims, wherein the tablet or region thereof contains from 2% to 30% by weight of water-soluble polycarboxylate.

14. A process for making a tablet according to any one of the preceding claims, which comprises mixing

a) particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration in an amount between 0.5% and 5% by weight of the sodium tripolyphosphate in the particles, with
b) other material to form a particulate detergent composition and compacting the composition to form a tablet or region thereof.

15. A process for making a tablet according to any one of claims 8 to 11, which comprises mixing

a) particles which contain sodium tripolyphosphate with a content of the phase I form which is more than 40% by weight of the sodium tripolyphosphate in the said particles, wherein the sodium tripolyphosphate in said particles contains water of hydration in an amount between 0.5% and 5% by weight of the sodium tripolyphosphate in the particles, and said particles are substantially free of organic detergent so as to contain less than 5% of their own weight of organic detergent and preferably none,
with a greater quantity of
b) further particles which contain detergent active mixed with other material,

to form a particulate detergent composition and compacting that composition to form a tablet or region thereof.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 97 30 8608

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
Y	DE 195 02 774 A (HENKEL KGAA) * the whole document, especially claims 1-3 *	1-15	C11D17/00 C11D3/06
Y	DE 14 67 624 A (KNAPSACK AG) * page 2, line 10 - line 27; claims 1-8; examples 1-4 *	1-15	
A	DE 11 91 509 B (HENKEL & CIE GMBH) * column 8, line 11 - line 37; claims 1-14; examples 1-6 *	1-15	
A	US 3 417 024 A (GOLDWASSER SEYMORE) * column 2, line 50 - line 64; table A *	1-15	
A	US 3 461 074 A (SCHWALLEY LAWRENCE L) * column 3, line 18 - line 59; claims 1-12 *	1-15	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6) C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	11 February 1998	Ainscow, J

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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