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54 **Liquid cleaning products.**

57 Dispensing of liquid cleaning products containing dispersed particles of sodium tripolyphosphate in a liquid solvent phase is improved if the average Phase II content of the STP is at least 50% by weight.

**EP 0 339 999 A2**

## Description

## LIQUID CLEANING PRODUCTS

The present invention is concerned with liquid cleaning compositions of the kind comprising a dispersion of sodium tripolyphosphate particles in a liquid solvent phase.

Non-aqueous cleaning compositions which contain sodium tripolyphosphate are known from EP 28849 (Unilever), where the tripolyphosphate is included as a detergency builder. Such non-aqueous products can give poor dispensing performance, for example when poured from a bottle or dispensed into a wash liquor by way of a drawer dispenser of a washing machine. In particular, they can leave undesirable residues.

The applicants have now found that this problem can be mitigated if the average Phase II content of the sodium tripolyphosphate is at least 50% by weight.

Thus, according to the invention, there is provided a non-aqueous liquid cleaning composition comprising a dispersion of sodium tripolyphosphate particles in a liquid phase, characterised in that the average Phase II content of the sodium tripolyphosphate is at least 50% by weight.

There are two crystal forms of sodium tripolyphosphate (STP), referred to as Phase I and Phase II, for example as described in 'Seifen-Öle-Fette-Wasche', 93, No.10 (1967), pp 305-308. A given sample of STP will normally contain both Phase I and Phase II. The relative amounts, which may be determined by x-ray diffraction, depend on the method of manufacture and in particular, on the temperature used in the calcining stage of the process where a high temperature ( $> 500^{\circ}\text{C}$ ) favours Phase I formation.

Most preferably, the average Phase II content of the total sodium tripolyphosphate present is at least 60% by weight. It is especially preferred that substantially all of the sodium tripolyphosphate is of the Phase II form.

The total level of STP in the compositions of the invention is typically from 15% to 50% by weight. The total level of Phase II STP in the compositions of the invention is preferably at least 15%, most preferably at least 18.6% by weight of the compositions. The level of Phase I STP in the composition should ideally not be more than 15% by weight.

The use of Phase II STP in other cleaning compositions is not unknown. United States patent specification US 4057506 (Sung/Colgate Palmolive) describes aqueous detergent compositions which contain sodium tripolyphosphate. The specification refers to "Form I" and "Form II" tripolyphosphate, the latter of which is thought to be equivalent to the Phase II material referred to herein. According to Sung, Form I is known as the "lumping variety" because when added to water, it lumps or cements together because of its high solubility and rapid rate of nucleation of hexahydrate which causes a nearly instantaneous drop in the concentration of dissolved solids. Form II, on the other hand, is said to be less metastable in water than Form I and instead remains high in dissolved phosphate and only gradually increases in solubility over a period of time, which may be hours. For these reasons it is preferred to utilize a material containing an excess of Form II, so that the tripolyphosphate is not precipitated from the aqueous compositions in time, thereby ensuring the purpose of the Sung invention that the compositions should be substantially devoid of any builder salt particles.

The present invention on the other hand is concerned with non-aqueous compositions and the need to improve the dispensing performance thereof. In the product the tripolyphosphate is in an undissolved state. In fact, it is in the form of tripolyphosphate salt particles, the very form which the invention of Sung was intended to avoid. Sung therefore gives no clue as to how improved dispensing of non-aqueous liquids might be achieved.

It is also preferred that the STP contains at least some seed crystals. These comprise particles of substantially anhydrous crystals of STP coated with small crystals of  $\text{STP} \cdot 6\text{H}_2\text{O}$ . The water in the latter hydrated crystals may derive from atmospheric moisture or be added deliberately. Typically, the total amount of water incorporated in the STP in the liquid composition will be from 0.1% to 2% by weight of the STP.

As will be elaborated hereinbelow, many of the compositions of the present invention have a liquid phase which comprises nonionic surfactant. For these, it is preferred that to aid dispensing performance still further, the nonionic surfactant should comprise two such surfactants, one comprising a  $\text{C}_{12-18}$  fatty alcohol alkoxylated with from 5 to 20 moles of ethylene oxide and/or propylene oxide and the other comprising a  $\text{C}_{5-11}$  alcohol alkoxylated with from 1 to 8 moles of ethylene oxide and/or propylene oxide.

In the compositions of the present invention, the liquid phase can be a liquid surfactant, an organic non-aqueous non-surfactant liquid, or a mixture of such materials. Many of the compositions do contain a surfactant as a dispersed or dissolved solid, or more often, as all or part of said liquid phase. These surfactant compositions are liquid detergent products, e.g. for fabrics washing or hard surface cleaning. However, the wider term 'liquid cleaning product' also includes non-surfactant liquids which are still useful in cleaning, for example non-aqueous bleach products or those in which the liquid phase consists of one or more light, non-surfactant solvents for greasy stain pre-treatment of fabrics prior to washing. Such pre-treatment products can contain solid bleaches, dispersed enzymes and the like.

The STP, and any other particles, can be maintained in dispersion in the liquid phase (i.e. resist settling, even if not perfectly) by a number of means. For example, settling may be inhibited purely by virtue of the relative small size of the particles and the relatively high viscosity of the liquid phase. In other words, the particles settle very slowly at a rate predicted by Stokes' law or due to the formation of a loosely aggregated network of particle flocs. This effect is utilised in the compositions described in patent specifications EP-A-30 096 (ICI) and GB 2 158 838A (Colgate-Palmolive). However, there have also been several prior proposals to utilise

additional means to enhance solid-suspending properties in such non-aqueous liquids. These are somewhat analogous to so-called external structuring techniques used in aqueous systems; i.e., in addition to the particulate solids and the liquid solvent phase in which they are to be suspended, an additional dispersant is used which by one means or another, acts to aid stable dispersion or suspension of the solids for a finite period. Any of these means may be employed in the compositions according to the present invention. 5

One such suitable stabilisation involves use of nonionic surfactant as the liquid phase and to add an inorganic carrier material as the dispersant, in particular highly voluminous silica. This acts by forming a solid-suspending network. This silica is highly voluminous by virtue of having an extremely small particle size, hence high surface area. This is described in GB patent specifications 1,205,711 (Unilever) and 1,270,040 (Unilever). However, there can be a problem with these compositions of setting upon prolonged storage. 10

A similar suitable structuring can be effected using fine particulate chain structure-type clay, as described in specification EP-A-34,387 (Procter).

Another appropriate known use of a substance as a dispersant for particles in nonionic-based non-aqueous compositions entails incorporating a hydrolyzable co-polymer of maleic anhydride with ethylene or vinylmethylether, which co-polymer is at least 30% hydrolyzed. This is described in specification EP-A-28,849 (Unilever). However, a problem with these compositions can be the difficulty in controlling manufacture to obtain reproducible product stability. 15

Another appropriate means by which such dispersions have been stabilised is the use of a dispersant material which has been termed 'a deflocculant', according to the disclosure of the applicants' European Patent Specification EP-A-266 199, the text of which is incorporated herein by reference. 20

All compositions according to the present invention are liquid cleaning products. They may be formulated in a very wide range of specific forms, according to the intended use. They may be formulated as cleaners for hard surfaces (with or without abrasive) or as agents for warewashing (cleaning of dishes, cutlery etc) either by hand or mechanical means, as well as in the form of specialised cleaning products, such as for surgical apparatus or artificial dentures. They may also be formulated as agents for washing and/or conditioning of fabrics. 25

In the case of hard-surface cleaning, the compositions may be formulated as main cleaning agents, or pre-treatment products to be sprayed or wiped on prior to removal, e.g. by wiping off or as part of a main cleaning operation.

In the case of warewashing, the compositions may also be the main cleaning agent or a pre-treatment product, e.g. applied by spray or used for soaking utensils in an aqueous solution and/or suspension thereof. 30

Those products which are formulated for the cleaning and/or conditioning of fabrics constitute an especially preferred form of the present invention. These compositions may for example, be of the kind used for pre-treatment of fabrics (e.g. for spot stain removal) with the composition neat or diluted, before they are rinsed and/or subjected to a main wash. The compositions may also be formulated as main wash products, being dissolved and/or dispersed in the water with which the fabrics are contacted. In that case, the composition may be the sole cleaning agent or an adjunct to another wash product. Within the context of the present invention, the term 'cleaning product' also embraces compositions of the kind used as fabric conditioners (including fabric softeners) which are only added in the rinse water (sometimes referred to as 'rinse conditioners'). 35

Thus, the compositions will contain at least one agent which promotes the cleaning and/or conditioning of the article(s) in question, selected according to the intended application. Usually, this agent will be selected from surfactants, enzymes, bleaches, microbiocides, (for fabrics) fabric softening agents and (in the case of hard surface cleaning) abrasives. Of course in many cases, more than one of these agents will be present, as well as other ingredients commonly used in the relevant product form. 40

The compositions will be substantially free from agents which are detrimental to the article(s) to be treated. For example, they will be substantially free from pigments or dyes, although of course they may contain small amounts of those dyes (colourants) of the kind often used to impart a pleasing colour to liquid cleaning products, as well as fluoescers, bluing agents and the like. 45

All ingredients before incorporation will either be liquid, in which case, in the composition they will constitute all or part of the liquid phase, or they will be solids, in which case, in the composition they will either be dispersed as deflocculated particles in the liquid phase or they will be dissolved in the liquid phase. Thus as used herein, the term "solids" is to be construed as referring to materials in the solid phase which are added to the composition and are dispersed therein in solid form, those solids which dissolve in the liquid phase and those in the liquid phase which solidify (undergo a phase change) in the composition, wherein they are then dispersed. 50

If a deflocculant is incorporated, some liquids are alone, unlikely to be suitable to perform the function of liquid phase for any combination of solids and dispersant/deflocculant. However, they will be able to be incorporated if used with another liquid which does have the required properties, the only requirement being that where the liquid phase comprises two or more liquid ingredients, they are miscible when in the total composition or one can be dispersible in the other, in the form of fine droplets. 55

Thus, where surfactants are solids, they will usually be dissolved or dispersed in the liquid phase. Where they are liquids, they will usually constitute all or part of the liquid phase. Also, some surfactants are eminently suitable as deflocculants.

In general however, surfactants may be chosen from any of the classes, sub-classes and specific materials 60 65

described in 'Surface Active Agents' Vol. I, by Schwartz & Perry, Interscience 1949 and 'Surface Active Agents' Vol. II by Schwartz, Perry & Berch (Interscience 1958), in the current edition of "McCutcheon's Emulsifiers & Detergents" published by the McCutcheon division of Manufacturing Confectioners Company or in 'Tensid-Taschenbuch', H. Stache, 2nd Edn., Carl Hanser Verlag, München & Wien, 1981.

Liquid surfactants are an especially preferred class of material to use in the liquid phase, especially polyalkoxylated types and in particular polyalkoxylated nonionic surfactants.

As a general rule, we have found that the most suitable liquids to choose are those organic materials having polar molecules. In particular, those comprising a relatively lipophilic part and a relatively hydrophilic part, especially a hydrophilic part rich in electron lone pairs, tend to be well suited. This is completely in accordance with the observation that liquid surfactants, especially polyalkoxylated nonionics, are one preferred class of solvent.

Nonionic detergent surfactants are well-known in the art. They normally consist of a water-solubilizing polyalkoxylene or a mono- or di-alkanolamide group in chemical combination with an organic hydrophobic group derived, for example, from alkylphenols in which the alkyl group contains from about 6 to about 12 carbon atoms, dialkylphenols in which each alkyl group contains from 6 to 12 carbon atoms, primary, secondary or tertiary aliphatic alcohols (or alkyl-capped derivatives thereof), preferably having from 8 to 20 carbon atoms, monocarboxylic acids having from 10 to about 24 carbon atoms in the alkyl group and polyoxypropylenes. Also common are fatty acid mono- and dialkanolamides in which the alkyl group of the fatty acid radical contains from 10 to about 20 carbon atoms and the alkyl group having from 1 to 3 carbon atoms. In any of the mono- and dialkanolamide derivatives, optionally, there may be a polyoxyalkylene moiety joining the latter groups and the hydrophobic part of the molecule. In all polyalkoxylene containing surfactants, the polyalkoxylene moiety preferably consists of from 2 to 20 groups of ethylene oxide or of ethylene oxide and propylene oxide groups. Amongst the latter class, particularly preferred are those described in the applicants' published European specification EP-A-225,654, especially for use as all or part of the solvent. Also preferred are those ethoxylated nonionics which are the condensation products of fatty alcohols with from 9 to 15 carbon atoms condensed with from 3 to 11 moles of ethylene oxide. Examples of these are the condensation products of C<sub>11-13</sub> alcohols with (say) 3 or 7 moles of ethylene oxide. These may be used as the sole nonionic surfactants or in combination with those of the described in the last-mentioned European specification, especially as all or part of the solvent.

Another class of suitable nonionics comprise the alkyl polysaccharides (polyglycosides/oligosaccharides) such as described in any of specifications US 3,640,998; US 3,346,558; US 4,223,129; EP-A-92,355; EP-A-99,183; EP-A-70,074, '75, '76, '77; EP-A-75,994, '95, '96.

Nonionic detergent surfactants normally have molecular weights of from about 300 to about 11,000. Mixtures of different nonionic detergent surfactants may also be used, provided the mixture is liquid at room temperature. Mixtures of nonionic detergent surfactants with other detergent surfactants such as anionic, cationic or ampholytic detergent surfactants and soaps may also be used. If such mixtures are used, the mixture must be liquid at room temperature.

Examples of suitable anionic detergent surfactants are alkali metal, ammonium or alkylolamine salts of alkylbenzene sulphonates having from 10 to 18 carbon atoms in the alkyl group, alkyl and alkylether sulphates having from 10 to 24 carbon atoms in the alkyl group, the alkylether sulphates having from 1 to 5 ethylene oxide groups, olefin sulphonates prepared by sulphonation of C<sub>10</sub>-C<sub>24</sub> alpha-olefins and subsequent neutralization and hydrolysis of the sulphonation reaction product.

Other surfactants which may be used include alkali metal soaps of a fatty acid, preferably one containing 12 to 18 carbon atoms. Typical such acids are oleic acid, ricinoleic acid and fatty acids derived from castor oil, rapeseed oil, groundnut oil, coconut oil, palmkernel oil or mixtures thereof. The sodium or potassium soaps of these acids can be used. As well as fulfilling the role of surfactants, soaps can act as detergency builders or fabric conditioners, other examples of which will be described in more detail hereinbelow. It can also be remarked that the oils mentioned in this paragraph may themselves constitute all or part of the liquid phase, whilst the corresponding low molecular weight fatty acids (triglycerides) can be dispersed as solids or function as structurants.

Yet again, it is also possible to utilise cationic, zwitterionic and amphoteric surfactants such as referred to in the general surfactant texts referred to hereinbefore. Examples of cationic detergent surfactants are aliphatic or aromatic alkyl-di(alkyl) ammonium halides and examples of soaps are the alkali metal salts of C<sub>12</sub>-C<sub>24</sub> fatty acids. Ampholytic detergent surfactants are e.g. the sulphobetaines. Combinations of surfactants from within the same, or from different classes may be employed to advantage for optimising structuring and/or cleaning performance.

Non-surfactants which are suitable as solvents include those having the preferred molecular forms referred to above although other kinds may be used, especially if combined with those of the former, more preferred types. In general, the non-surfactant solvents can be used alone or with in combination with liquid surfactants. Non-surfactant solvents which have molecular structures which fall into the former, more preferred category include ethers, polyethers, alkylamines and fatty amines, (especially di- and tri-alkyl- and/or fatty-N-substituted amines), alkyl (or fatty) amides and mono- and di- N-alkyl substituted derivatives thereof, alkyl (or fatty) carboxylic acid lower alkyl esters, ketones, aldehydes, and glycerides. Specific examples include respectively, di-alkyl ethers, polyethylene glycols, alkyl ketones (such as acetone) and glyceryl trialkylcarboxylates (such as glyceryl triacetate), glycerol, propylene glycol, and sorbitol.

In deflocculated systems, many light solvents with little or no hydrophilic character are usually unsuitable on their own (i.e. deflocculation will not occur in them). Examples of these are lower alcohols, such as ethanol, or higher alcohols, such as dodecanol, as well as alkanes and olefins. However, they can be combined with other liquid materials which are surfactants or non-surfactants having the aforementioned 'preferred' kinds of molecular structure.

Preferably, the compositions of the invention contain the liquid phase (whether or not comprising liquid surfactant) in an amount of at least 10% by weight of the total composition. The amount of the liquid phase present in the composition may be as high as about 90%, but in most cases the practical amount will lie between 20 and 70% and preferably between 20 and 50% by weight of the composition.

Preferably also, the compositions of the present invention contain a deflocculant (as hereinbefore defined) which may be any of those referred to in the published prior art, most preferably any described in EP-A-266 199.

The level of any deflocculant material in the composition can be optimised by the means hereinbefore described but in very many cases is at least 0.01%, usually 0.1% and preferably at least 1% by weight, and may be as high as 15% by weight. For most practical purposes, the amount ranges from 2-12%, preferably from 4-10% by weight, based on the final composition.

The compositions according to the present invention preferably also contain one or more other functional ingredients, for example selected from other detergency builders, bleaches or bleach systems, and (for hard surface cleaners) abrasives.

Detergency builders are those materials which counteract the effects of calcium, or other ion, water hardness, either by precipitation or by an ion sequestering effect. They comprise both inorganic and organic builders. They may also be sub-divided into the phosphorus-containing and non-phosphorus types.

In general, other than the STP, optional additional inorganic builders comprise the various phosphate-, carbonate-, silicate-, borate- and aluminosilicate-type materials, particularly the alkali-metal salt forms. Mixtures of these may also be used.

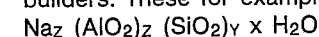
Examples of optional additional phosphorus-containing inorganic builders include the water-soluble salts, especially alkali metal pyrophosphates, orthophosphates, polyphosphates and phosphonates. Specific examples of inorganic phosphate builders include sodium and potassium phosphates and hexametaphosphates, as well as potassium triphosphate.

Examples of optional additional non-phosphorus-containing inorganic builders, when present, include water-soluble alkali metal carbonates, bicarbonates, borates, silicates, metasilicates, and crystalline and amorphous aluminosilicates. Specific examples include sodium carbonate (with or without calcite seeds), potassium carbonate, sodium and potassium bicarbonates, silicates and zeolites.

Examples of optional additional organic builders include the alkali metal, ammonium and substituted, citrates, succinates, malonates, fatty acid sulphonates, carboxymethoxy succinates, ammonium polyacetates, carboxylates, polycarboxylates, aminopolycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates. Specific examples include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, melitic acid, benzene polycarboxylic acids and citric acid. Other examples are organic phosphonate type sequestering agents such as those sold by Monsanto under the tradename of the Dequest range and alkanedihydroxy phosphonates.

Other suitable additional organic builders include the higher molecular weight polymers and co-polymers known to have builder properties, for example appropriate polyacrylic acid, polymaleic acid and polyacrylic/polymaleic acid co-polymers and their salts, such as those sold by BASF under the Sokalan Trade Mark.

The aluminosilicates are an especially preferred class of optional additional non-phosphorus inorganic builders. These for example are crystalline or amorphous materials having the general formula:



wherein Z and Y are integers of at least 6, the molar ratio of Z to Y is in the range from 1.0 to 0.5, and x is an integer from 6 to 189 such that the moisture content is from about 4% to about 20% by weight (termed herein, 'partially hydrated'). This water content provides the best rheological properties in the liquid. Above this level (e.g. from about 19% to about 28% by weight water content), the water level can lead to network formation. Below this level (e.g. from 0 to about 6% by weight water content), trapped gas in pores of the material can be displaced which causes gassing and tends to lead to a viscosity increase also. However, it will be recalled that anhydrous materials (i.e. with 0 to about 6% by weight of water) can be used as structurants. The preferred range of aluminosilicate is from about 12% to about 30% on an anhydrous basis. The aluminosilicate preferably has a particle size of from 0.1 to 100 microns, ideally between 0.1 and 10 microns and a calcium ion exchange capacity of at least 200 mg calcium carbonate/g.

Suitable bleaches include the halogen, particularly chlorine bleaches such as are provided in the form of alkalimetal hypochlorites, e.g. hypochlorites. In the application of fabrics washing, the oxygen bleaches are preferred, for example in the form of an inorganic persalt, preferably with an precursor, or as a peroxy acid compound.

In the case of the inorganic persalt bleaches, the precursor makes the bleaching more effective at lower temperatures, i.e. in the range from ambient temperature to about 60°C, so that such bleach systems are commonly known as low-temperature bleach systems and are well known in the art. The inorganic persalt such as sodium perborate, both the monohydrate and the tetrahydrate, acts to release active oxygen in solution,

and the precursor is usually an organic compound having one or more reactive acyl residues, which cause the formation of peracids, the latter providing for a more effective bleaching action at lower temperatures than the peroxybleach compound alone. The ratio by weight of the peroxy bleach compound to the precursor is from about 15:1 to about 2:1, preferably from about 10:1 to about 3.5:1. Whilst the amount of the bleach system, i.e. peroxy bleach compound and precursor, may be varied between about 5% and about 35% by weight of the total liquid, it is preferred to use from about 6% to about 30% of the ingredients forming the bleach system. Thus, the preferred level of the peroxy bleach compound in the composition is between about 5.5% and about 27% by weight, while the preferred level of the precursor is between about 0.5% and about 40%, most preferably between about 1% and about 5% by weight.

Typical examples of the suitable peroxybleach compounds are alkalimetal peroborates, both tetrahydrates and monohydrates, alkali metal percarbonates, persilicates and perphosphates, of which sodium perborate is preferred.

Precursors for peroxybleach compounds have been amply described in the literature, including in British patent specifications 836,988, 855,735, 907,356, 907,358, 907,950, 1,003,310, and 1,246,339, US patent specifications 3,332,882, and 4,128,494, Canadian patent specification 844,481 and South African patent specification 68/6,344.

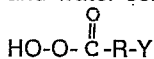
The exact mode of action of such precursors is not known, but it is believed that peracids are formed by reaction of the precursors with the inorganic peroxy compound, which peracids then liberate active-oxygen by decomposition.

They are generally compounds which contain N-acyl or O-acyl residues in the molecule and which exert their activating action on the peroxy compounds on contact with these in the washing liquor.

Typical examples of precursors within these groups are polyacylated alkylene diamines, such as N,N,N',N'-tetraacetylene diamine (TAED) and N,N,N',N'-tetraacetylmethylene diamine (TAMD); acylated glycolurils, such as tetraacetylglucoluril (TAGU); triacetylcyanurate and sodium sulphophenyl ethyl carbonic acid ester.

A particularly preferred precursor is N,N,N',N'-tetraacetylene diamine (TAED).

The organic peroxyacid compound bleaches are preferably those which are solid at room temperature and most preferably should have a melting point of at least 50°C. Most commonly, they are the organic peroxyacids and water-soluble salts thereof having the general formula



wherein R is an alkylene or substituted alkylene group containing 1 to 20 carbon atoms or an arylene group containing from 6 to 8 carbon atoms, and Y is hydrogen, halogen, alkyl, aryl or any group which provides an anionic moiety in aqueous solution.

Another preferred class of peroxygen compounds which can be incorporated to enhance dispensing/dispersibility in water are the anhydrous perborates described for that purpose in the applicants' European patent specification EP-A-217,454.

When the composition contains abrasives for hard surface cleaning (i.e. is a liquid abrasive cleaner), these will inevitably be incorporated as particulate solids. They may be those of the kind which are water insoluble, for example calcite. Suitable materials of this kind are disclosed in the applicants' patent specifications EP-A-50,887; EP-A-80,221; EP-A-140,452; EP-A-214,540 and EP 9,942, which relate to such abrasives when suspended in aqueous media. Water soluble abrasives may also be used.

The compositions of the invention optionally may also contain one or more minor ingredients such as fabric conditioning agents, enzymes, perfumes (including deoperfumes), micro-biocides, colouring agents, fluorescers, soil-suspending agents (anti-redeposition agents), corrosion inhibitors, enzyme stabilizing agents, and lather depressants.

In general, the solids content of the product may be within a very wide range, for example from 1-90%, usually from 10-80% and preferably from 15-70%, especially 15-50% by weight of the final composition. The solid phase, comprising the STP, should be in particulate form and have an average particle size of less than 300 microns, preferably less than 200 microns, more preferably less than 100 microns, especially less than 10 microns. The particle size may even be of sub-micron size. The proper particle size can be obtained by using materials of the appropriate size or by milling the total product in a suitable milling apparatus.

The compositions are substantially non-aqueous, i.e. they little or no free water, preferably no more than 5%, preferably less than 3%, especially less than 1% by weight of the total composition. It has been found by the applicants that the higher the water content, the more likely it is for the viscosity to be too high, or even for setting to occur. However, this may at least in part be overcome by use of deflocculants, especially in relatively high amounts.

Since the objective of a non-aqueous liquid will generally be to enable the formulator to avoid the negative influence of water on the components, e.g. causing incompatibility of functional ingredients, it is clearly necessary to avoid the accidental or deliberate addition of water to the product at any stage in its life. For this reason, special precautions are necessary in manufacturing procedures and pack designs for use by the consumer.

Thus during manufacture, it is preferred that all raw materials should be dry and (in the case of hydratable salts) in a low hydration state, e.g. anhydrous phosphate builder, sodium perborate monohydrate and dry calcite abrasive, where these are employed in the composition. In a preferred process, the dry, substantially

anhydrous solids are blended with the liquid phase in a dry vessel. In order to minimise the rate of sedimentation of the solids, this blend is passed through a grinding mill or a combination of mills, e.g. a colloid mill, a corundum disc mill, a horizontal or vertical agitated ball mill, to achieve a particle size of 0.1 to 100 microns, preferably 0.5 to 50 microns, ideally 1 to 10 microns. A preferred combination of such mills is a colloid mill followed by a horizontal ball mill since these can be operated under the conditions required to provide a narrow size distribution in the final product. Of course particulate material already having the desired particle size need not be subjected to this procedure and if desired, can be incorporated during a later stage of processing.

During this milling procedure, the energy input results in a temperature rise in the product and the liberation of air entrapped in or between the particles of the solid ingredients. It is therefore highly desirable to mix any heat sensitive ingredients into the product after the milling stage and a subsequent cooling step. It may also be desirable to de-aerate the product before addition of these (usually minor) ingredients and optionally, at any other stage of the process. Typical ingredients which might be added at this stage are perfumes and enzymes, but might also include highly temperature sensitive bleach components or volatile solvent components which may be desirable in the final composition. However, it is especially preferred that volatile material be introduced after any step of aeration. Suitable equipment for cooling (e.g. heat exchangers) and de-aeration will be known to those skilled in the art.

It follows that all equipment used in this process should be completely dry, special care being taken after any cleaning operations. The same is true for subsequent storage and packing equipment.

The present invention will now be illustrated by way of the following non-limiting Examples.

#### EXAMPLES 1 AND 2

Four compositions were prepared according to the composition listed below. However, the STP morphology was varied.

The dispensing properties of the compositions were found to improve progressively from A to B to Example 1 to Example 2. They were acceptable in Examples 1 and 2 the latter giving the best result.

Example No.	A	B	1	2
<u>Formulation</u>	<u>wt %</u>			
5 Dobanol 91/6*	<-----		36.6	----->
Glycerol tri-acetate	<-----		5.0	----->
10 ABSA**	<-----		3.0	----->
STP (Phase 1)	24.6	15.9	11.4	-
STP (Phase 2)	5.4	14.1	18.6	30.0
15 Sodium carbonate 0aq	<-----		4.0	----->
Na Perborate monohydrate	<-----		15.0	----->
EDTA	<-----		0.15	----->
20 SCMC	<-----		1.0	----->
TAED	<-----		4.0	----->
Dequest 2041	<-----		0.1	----->
25 Fluorescer (Tinopal DMS-X)	<-----		0.3	----->
Tylose MH20	<-----		0.5	----->
Silicone DB100	<-----		0.25	----->
30 Savinase 8.0 SL	<-----		0.6	----->

\* Nonionic surfactant comprising C<sub>9-11</sub> fatty alcohol  
 35 alkoxyated with an average of 6 moles of ethylene oxide  
 (ex Shell). \*\* Dodecyl benzene sulphonic (free) acid.

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#### Claims

- 45 1. A non-aqueous liquid cleaning composition comprising a dispersion of sodium tripolyphosphate particles in a liquid phase, characterised in that the average Phase II content of the sodium tripolyphosphate is at least 50% by weight.
2. A composition according to claim 1, wherein the average Phase II content is at least 60% by weight.
3. A composition according to claim 1, wherein substantially all of the sodium tripolyphosphate is of the Phase II crystal form.
- 50 4. A composition according to claim 1, wherein the sodium tripolyphosphate comprises at least some seed crystals.
5. A composition according to claim 1, wherein the liquid phase comprises a nonionic surfactant.
6. A composition according to claim 5, wherein the nonionic surfactant comprises both:-
  - (i) a C<sub>12-18</sub> fatty alcohol alkoxyated with from 5 to 20 moles of ethylene oxide and/or propylene
  - 55 oxide; and
  - (ii) a C<sub>5-11</sub> alcohol alkoxyated with from 1 to 8 moles of ethylene oxide and/or propylene oxide.
7. A composition according to claim 1, further comprising a deflocculant for the particles.
8. A composition according to claim 1, containing from 15% to 50% by weight sodium tripolyphosphate, based on the weight of the composition.
- 60 9. A composition according to claim 1, containing at least 15% by weight Phase II sodium tripolyphosphate, based on the weight of the composition.

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