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EUROPEAN PATENT APPLICATION

⑲ Application number: **80303673.0**

⑤① Int. Cl.³: **C 11 D 3/06, C 11 D 3/10,**
C 11 D 11/02

⑳ Date of filing: **17.10.80**

③① Priority: **19.10.79 GB 7936351**

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④③ Date of publication of application: **27.05.81**
Bulletin 81/21

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⑧④ Designated Contracting States: **AT BE CH DE FR IT LI**
NL SE

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⑤④ **Detergent composition and process for its production.**

⑤⑦ A detergent composition, particularly for fabric washing or dishwashing, contains a detergent active compound, an orthophosphate builder and an alkalimetal bicarbonate to reduce the initial pH. The bicarbonate is in particulate form with a preferred particle size of 50 to 500 microns. A typical composition contains 5 to 30% detergent active, 10 to 30% of a mixture of tripolyphosphate and orthophosphate in a ratio of 10:1 to 1:5 and 2 to 15% bicarbonate. Preferably the compositions also include an alkalimetal silicate in similar amounts to the bicarbonate. In use the composition gives an initial pH below 11.0 and a final pH between 9.0 and 11.0.

DETERGENT COMPOSITIONS

TECHNICAL FIELD

The invention relates to powdered detergent compositions which are adapted for fabric washing or dishwashing.

BACKGROUND ART

- 5 It is common practice for detergent compositions to form fairly alkaline solutions, as this has a significant beneficial effect on detergency by helping to solubilise fatty soils whilst also improving the effect of some ingredients, for example peroxygen bleaching agents.
- 10 However, the alkalinity should of course not be too high for domestic safety, particularly allowing for conditions of mis-use. In addition, very high alkalinity can cause fabric damage and colour-fading during washing, particularly at high wash temperatures. It is usual
- 15 therefore to employ wash solutions having a pH in the range of from about 9 to 11, preferably about 9.5 to 10.5.

The widespread, but often misinformed, criticism of sodium tripolyphosphate as a detergency builder has led to the consideration of several more alkaline materials for its partial or complete replacement, for example sodium carbonate and sodium orthophosphate. The latter, in particular, can act as an efficient detergency builder, especially at high temperatures, whilst it has a relatively low phosphorus content in relation to the amount needed to counteract the calcium ions present in hard water. Although the higher alkalinity of alkali metal orthophosphate salts is not of itself harmful, provided the pH at equilibrium washing conditions is not excessively high, i.e. over about pH 11, we have found that under some circumstances the initial pH on dissolution of such detergent compositions in water can be higher than normally desired, apparently due to the differential rates of solubility of the various detergent ingredients. Thus, depending on the concentration in the solution and temperature, initial pHs of the order of up to 11.5 or even 12 may be observed before the pH then drops to less than 11 for the remainder of the wash cycle.

It is known, particularly from GB 1 412 401, to include alkaline salts in detergent compositions. The alkaline salts may be a bicarbonate in combination with other alkaline material. These bicarbonate-containing compositions may be prepared by spray-drying in which event the bicarbonate becomes converted to the corresponding carbonate in the final composition. Such compositions suffer from the disadvantage outlined above, of a particularly high initial pH.

DISCLOSURE OF THE INVENTION

We have now found it possible to decrease the initial peak pH on using particulate detergent compositions which contain an alkali metal orthophosphate detergency builder, by including therein a proportion of discrete particles of sodium or potassium bicarbonate. Thus, according to a

first aspect of the invention there is provided a detergent composition containing at least one synthetic detergent active compound and an alkalimetal ortho-phosphate detergency builder, wherein the composition
5 further contains discrete particles of sodium or potassium bicarbonate and the composition yields an initial pH of not more than about 11.0 and an equilibrium pH of between about 9.0 and about 11.0, preferably 9.5 to 10.5 when dissolved in distilled water at 25°C at a
10 concentration of 1% by weight.

BEST MODE OF CARRYING OUT THE INVENTION

The amount of the bicarbonate used is normally within the range of about 1% to about 20%, preferably from about 2% to 15%, especially about 4% to 10%, by
15 weight of the composition. The higher levels within this range tend to be of greater value with compositions likely to be used for fabric washing by hand, for example in the developing countries. If desired, the bicarbonate may be used partly in the form of a sesquicarbonate,
20 which is a 1:1 mixture of bicarbonate and carbonate salts.

The alkalimetal bicarbonate used is preferably of small particle size so as to facilitate its prompt dissolution in water, i.e. not later than other alkaline detergent ingredients are dissolved. In particular it is
25 preferred to use an alkalimetal bicarbonate having a mean particle size diameter of less than about 1000 microns, preferably from about 50 to 500 microns. Whilst bicarbonate particles of smaller size than 50 microns have good dissolution properties, they can cause dustiness
30 during powder handling, and particles over about 1000 microns can contribute to segregation.

The benefit of adding the bicarbonate is widely applicable to the detergent compositions comprising an alkalimetal orthophosphate salt, usually at levels
35 between about 2% and about 30%, especially about 5-20%

by weight of the composition. However, the invention is particularly applicable to the compositions described and claimed in our UK patent No 1 530 799. Such compositions comprise from about 5% to about 30% of a
5 synthetic anionic, nonionic, amphoteric or zwitterionic detergent compound or mixture thereof, and from about 10% to about 30% of mixed sodium tripolyphosphate and alkalimetal orthophosphate in the ratio of from 10:1 to 1:5 parts by weight, wherein the amount of the sodium
10 tripolyphosphate is at least about 5% and the amount of any alkalimetal pyrophosphate is not more than about 5%, all these percentages being expressed by weight of the total detergent composition, and the pH of a 0.1% aqueous solution of the composition being from 9 to 11.

15 Potassium tripolyphosphate could be used instead of sodium tripolyphosphate.

Whilst the ratio of the sodium tripolyphosphate to the alkalimetal orthophosphate can be varied from 10:1 to 1:5 parts by weight, it is preferred to have a ratio
20 of from 8:1 to 1:2, especially with an excess of the sodium tripolyphosphate over the alkalimetal orthophosphate, within the ratio of from 5:1 to 1:1 parts by weight, for example from about 4:1 to about 3:2, and more especially from about 3:1 to about 2:1 parts by weight,
25 respectively. These ratios of sodium tripolyphosphate to alkalimetal orthophosphate are especially suitable for detergent compositions used at relative high product concentrations, i.e. about 0.3% to about 0.8% by weight, as is common practice in Europe, especially in front-
30 loading automatic washing machines, and where relatively high levels of phosphates are allowed in the products, i.e. equivalent to about 4% to about 7% P.

However, for detergent compositions which are to be used at relatively low product concentrations, i.e.
35 from about 0.1% to about 0.3%, as is common practice

under North American washing conditions, especially in top-loading automatic washing machines, and where relatively low phosphate levels are permitted in the products, i.e. equivalent to less than about 4% P, it may be desirable to increase the proportion of the alkalimetal orthophosphate in the products. The ratio of sodium tripolyphosphate to alkalimetal orthophosphate can then be from 2:1 to 1:5 parts by weight, preferably from 1:1 to 1:5, for example from 1:1 to 1:2 or 1:3 parts by weight, respectively. Alternatively, when especially low phosphate levels are enforced it may be beneficial to have a supplementary non-phosphate builder, whilst still achieving optimum detergency building from the phosphate builders in the compositions according to the invention.

It will be appreciated that the actual amounts of sodium tripolyphosphate and alkalimetal orthophosphate are chosen according to the overall phosphate detergency builder level which is desired in the detergent compositions or according to the maximum permitted phosphorus content. Within the requirements of a total sodium tripolyphosphate and alkalimetal orthophosphate level of about 10% to about 30% by weight of the product, it is preferable to have a sodium tripolyphosphate content of from about 10% to about 20%, and an alkalimetal orthophosphate content of from about 3% to about 15%, especially about 5% to about 10%, by weight of the product. The total amount of sodium tripolyphosphate and alkalimetal orthophosphate is preferably at least about 15%, up to about 25% by weight of the composition.

It is preferable that the only phosphate detergency builders used to make the compositions of the invention should be alkalimetal orthophosphate and optionally the sodium tripolyphosphate. In particular, it is desirable to add no alkalimetal, i.e. sodium or potassium, pyrophosphates to the compositions as they tend to increase

inorganic deposition. Moreover, the present of significant levels of the alkalimetal pyrophosphates instead of the sodium tripolyphosphate or alkalimetal orthophosphate leads to lower detergency building capacities within the limited phosphate levels permitted. However, as mentioned earlier, some sodium pyrophosphate is produced with a lesser amount of sodium orthophosphate by hydrolysis of sodium tripolyphosphate under the hot alkaline conditions met during spray drying, so low levels, i.e. up to about 5%, of sodium pyrophosphate are unavoidable in spray dried powders. Preferably the total amount of phosphate materials present in the detergent compositions is not more than about 30% by weight of the compositions. It should be noted that the amounts of the hydratable phosphate salts in the compositions are to be determined on an anhydrous basis.

Other detergent compositions comprising alkalimetal orthophosphate salts which can benefit by the addition of the sodium bicarbonate include compositions with the orthophosphate as the sole detergency builder or in admixture with other non-phosphate builders such as NTA or sodium carbonate.

The alkalimetal orthophosphate used is potassium or preferably sodium orthophosphate, as the latter is cheaper and more readily available. Normally the tri-alkyl metal salts are used, but orthophosphoric acid or the di- or monoalkali metal salts, e.g. disodium hydrogen orthophosphate or monosodium dihydrogen orthophosphate, could be used if desired to form the compositions. In the latter event other more alkaline salts would also be present to maintain a high pH in the end product, with full neutralisation to the trialkali metal orthophosphate salts. The use of a mixture of the monosodium and disodium hydrogen orthophosphates in the ratio of about 1:3 to 2:3, especially about 1:2, is particularly

advantageous, as such a mixture is made as a feedstock for the production of sodium tripolyphosphate and is therefore readily available. The alkalimetal orthophosphate can be used initially as the hydrated salt, for example as trisodium orthophosphate dodecahydrate, or in anhydrous state in which case hydration normally takes place during detergent powder production. The amount of the salt is, however, calculated in anhydrous form.

10 The use of an alkalimetal hydrogen phosphate such as disodium monohydrogen phosphate, instead of using the fully neutralised alkalimetal orthophosphate salts can help to decrease the alkalinity of the wash solution, assuming of course that the other ingredients are
15 otherwise the same, whilst maintaining the same orthophosphate salt content in solution. However, in compositions which have a sufficient alkaline pH for effective fabric washing, the use of the alkalimetal hydrogen phosphates has been found to be less effective
20 in controlling the peak pH on initial dissolution of the compositions in water.

It will be appreciated that addition of the alkalimetal bicarbonate will tend to decrease the overall pH of the wash liquor, besides decreasing the initial pH peak.,
25 It may be desirable therefore to add to the composition an amount of a more alkaline ingredient which does not affect the initial pH appreciably, but which raises the final pH of the wash liquor to the optimum level which would be achieved by the composition but for the addition of the
30 bicarbonate. For example, it has been found that an extra amount of about 3% of sodium alkaline silicate included in the detergent composition compensates in this way for the addition of about 5% of sodium bicarbonate. The total amount of sodium silicate used in the
35 compositions for such pH control during the wash, as well

as giving improved powder properties and corrosion resistance, is about 5% to about 15% by weight of the composition.

5 The detergent compositions include about 5% to 40%, especially about 15% to 30% of an anionic, nonionic, amphoteric or zwitterionic detergent compound. Such compounds are well known in the art and are amply described in the literature, for example in "Surface Active Agents and Detergents" by Schwartz, Perry and
10 Berch, Volumes I and II.

The preferred detergent compounds which can be used are synthetic anionic and nonionic compounds. The former are usually water soluble alkalimetal salts of organic sulphates and sulphonates having alkyl radicals containing
15 from about 8 to about 22 carbon atoms, the term alkyl being used to include the alkyl portion of higher acyl radicals. Examples of suitable synthetic anionic detergent compounds are sodium and potassium alkyl sulphates, especially those obtained by sulphating higher (C_8-C_{18}) alcohols
20 produced for example from tallow or coconut oil; sodium and potassium alkyl (C_9-C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl ($C_{10}-C_{15}$) benzene sulphonates; sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived
25 from tallow or coconut oil and synthetic alcohols derived from petroleum; sodium coconut oil fatty acid monoglyceride sulphates and sulphonates; sodium and potassium salts of sulphuric acid esters of higher (C_9-C_{18}) fatty alcohol-alkylene oxide, particularly ethylene oxide, reaction
30 products; the reaction products of fatty acids such as coconut fatty acids esterified with isethionic acid and neutralised with sodium hydroxide; sodium and potassium salts of fatty acid amides of methyl taurine; alkane monosulphonates such as those derived by reacting alpha-
35 olefins (C_8-C_{20}) with sodium bisulphite and those derived

by reacting paraffins with SO_2 and Cl_2 and then hydrolysing with a base to produce a random sulphonate; and olefin sulphonates, which term is used to describe the material made by reacting olefins, particularly
5 C_{10} - C_{20} alpha olefins, with SO_3 and then neutralising and hydrolysing the reaction product. The preferred anionic detergent compounds are sodium (C_{11} - C_{15}) alkyl benzene sulphonates and sodium (C_{16} - C_{18}) alkyl sulphates.

Examples of suitable nonionic detergent compounds
10 which may be used include in particular the reaction products of alkylene oxides, usually ethylene oxide, with alkyl (C_6 - C_{22}) phenols, generally 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule; the condensation products of aliphatic (C_8 - C_{18}) primary or
15 secondary linear or branched alcohols with ethylene oxide, generally 6 to 30 EO, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic
20 detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides.

Mixtures for detergent compounds, for example mixed anionic or mixed anionic and nonionic compounds may be used in the detergent compositions, particularly in the
25 latter case to provide controlled low sudsing properties. This is beneficial for compositions intended for use in suds-intolerant automatic washing machines. We have also found that the use of some nonionic detergent compounds in the compositions tends to decrease the tendency of
30 insoluble phosphate salts to deposit on the washed fabrics.

Amounts of amphoteric or zwitterionic detergent compounds can also be used in the compositions of the invention but this is not normally desired due to their relatively high cost. If any amphoteric or zwitterionic
35 detergent compounds are used it is generally in small

amounts in compositions based on the much more commonly used synthetic anionic and/or nonionic detergent compounds. For example, mixtures of amine oxides and ethoxylated nonionic detergent compounds can be used.

5 Some soaps may also be used in the compositions of the invention, but not as the sole detergent compounds. They are particularly useful at low levels in binary and ternary mixtures together with nonionic or mixed synthetic anionic and nonionic detergent compounds,
10 which have low sudsing properties. The soaps which are used are the sodium, or less desirably potassium, salts of C₁₀-C₂₄ fatty acids. It is particularly preferred that the soaps should be based mainly on the longer-chain fatty acids within this range, that is with at least half
15 of the soap having a carbon chain length of 16 or over. This is most conveniently accomplished by using soaps from natural sources such as tallow, palm oil or rapeseed oil, which can be hardened if desired, with lesser amounts of other shorter-chain soaps, prepared from nut
20 oils such as coconut oil or palm kernel oil. The amount of such soaps can be varied between about 0.5% and about 25% by weight, with lower amounts of about 0.5% to about 5% being generally sufficient for lather control. Amounts of soap between about 2% and about
25 20%, especially between about 5% and about 15%, are preferably used to give a beneficial effect on detergency.

Apart from the detergent compounds and detergency builders, the detergent compositions of the invention can contain any of the conventional additives in the amounts
30 in which such materials are normally employed in fabric washing detergent compositions. Examples of these additives include lather boosters such as alkanolamides, particularly the monoethanolamides derived from palm kernel fatty acids and coconut fatty acids, lather
35 depressants such as alkyl phosphates and silicones,

antiredeposition agents such as sodium carboxymethyl-cellulose and polyvinyl pyrrolidone, oxygen-releasing bleaching agents such as sodium perborate and sodium percarbonate, per-acid bleach precursors, chlorine-releasing
5 bleaching agents such as trichloroisocyanuric acid and alkalimetal salts of dichloroisocyanuric acid, fabric softening agents, inorganic salts such as sodium sulphate, sodium carbonate and magnesium silicate, and, usually present in very minor amounts, fluorescent agents,
10 perfumes, enzymes such as proteases and amylases, germicides and colourants.

It is particularly beneficial to include in the detergent compositions an amount of sodium perborate, preferably between about 10% and 40%, for example about
15 15% to about 30%, by weight.

It is desirable to include one or more antideposition agents in the detergent compositions of the invention, to decrease any tendency to form inorganic deposits on washed fabrics. The amount of any such antideposition agent is
20 normally from about 0.1% to about 5% by weight, preferably from about 0.2% to about 2% by weight of the composition. The preferred antideposition agents are salts of homo- and co-polymers of acrylic acid or substituted acrylic acids, such as sodium polyacrylate, the sodium salt of
25 copolymethacrylamide/acrylic acid and sodium poly-alpha-hydroxyacrylate, salts of copolymers of maleic anhydride with ethylene, vinylmethylether or styrene, especially 1:1 copolymers, and optionally with partial esterification of the carboxyl groups especially in the case of the
30 styrene-maleic anhydride copolymers. Such copolymers preferably have relatively low molecular weights, e.g. in the range of about 5,000 to 50,000. Other antideposition agents include the sodium salts of polymaleic acid and polyitaconic acid, phosphate esters of ethoxylated
35 aliphatic alcohols, polyethylene glycol phosphate esters,

and certain phosphonates such as sodium ethane-1-hydroxy-1, 1-diphosphonate, sodium ethylene diamine tetramethylene phosphonate, and sodium 2-phosphonobutane tricarboxylate.

Mixtures of organic phosphonic acids or substituted

5 acrylic acids or their salts with protective colloids such as gelatin as described in our Netherlands application 7602082 may also be used. The most preferred anti-deposition agent is sodium polyacrylate having a MW of about 10,000 to 50,000, for example about 27,000.

10 It is also possible to include in the detergent compositions of the invention minor amounts, preferably not more than about 10% by weight, of other non-phosphate detergency builders, which may be either so-called precipitant builders or sequesterant builders. Examples
15 of such other detergency builders are amine carboxylates such as sodium nitrilotriacetate, sodium aluminosilicate ion-exchange materials, sodium citrate, sodium carbonate and soap, which can function as a detergency builder as discussed above.

20 Bearing all the above comments in mind, the preferred compositions according to the invention comprise:-

from about 5% to about 30% of a synthetic anionic, nonionic, amphoteric or zwitterionic detergent compound

25 or mixture thereof;

from about 10% to about 30% of a mixture of sodium tripolyphosphate and an alkalimetal orthophosphate in the ratio of from about 10:1 to about 1:5 parts by weight, the amount of sodium tripolyphosphate being at least about 5% by weight;

30 from about 2% to about 15% by weight of sodium or potassium bicarbonate particles having a discrete particle size from about 50 to about 500 microns;

from about 5% to about 15% by weight of sodium silicate; optionally not more than about 5% by weight of alkali-

35 metal pyrophosphate; and

optionally one or more further ingredients selected from lather boosters, lather depressants, antiredeposition agents, oxygen-releasing bleaching agents, per-acid bleach precursors, chlorine-releasing bleaching agents, 5 fabric softening agents, inorganic salts, fluorescent agents, perfumes, enzymes, germicides and colourants.

The compositions of the invention are in particulate form, especially as free-flowing powders or granules, and they can be produced by any of the techniques commonly 10 employed for making such compositions, normally by slurry making and spray drying processes. Thus, according to a second aspect of the invention there is provided a process for the production of a detergent composition, comprising the steps of

- 15 (i) spray-drying a slurry containing at least one synthetic detergent active compound and an alkali-metal orthophosphate detergency builder to form a spray-dried base powder;
- (ii) admixing with said spray-dried base powder discrete 20 particles of sodium or potassium bicarbonate thereby to form a composition which yields an initial pH of not more than about 11.0 and an equilibrium pH of between about 9.0 and about 11.0 when dissolved in distilled water at 25°C at a concentration of 1% 25 by weight.

The alkalimetal bicarbonate salts cannot be included in the detergent slurry for normal slurry making and spray drying techniques to make the detergent compositions, as the bicarbonate would then react in the 30 slurry to form sodium carbonate. It follows that the alkalimetal bicarbonate must be admixed as discrete particles with the preformed particulate detergent composition, that is to say the bicarbonate must be present in that partial salt form, or partly as sesqui- 35 carbonate, but not otherwise used in the production of the

detergent compositions and thereby present in more highly neutralised form. Although the bicarbonate can be added in its powder form as received, it can be treated if desired to reduce dustiness, for example by admixture
5 of a liquid detergent ingredient such as a nonionic detergent compound, which does not delay the dissolution of the bicarbonate salts in the wash liquor.

The invention is illustrated by the following Examples in which parts and percentages are by weight
10 except where otherwise indicated.

EXAMPLE 1

A detergent composition was made to the following formulation:

	<u>Ingredient</u>	<u>%</u>
15	Sodium alkyl benzene sulphonate	6.0
	Nonylphenol - 10 EO	2.0
	Sodium soap	3.0
	Sodium tripolyphosphate	16.0
	Trisodium orthophosphate	6.0
20	Sodium alkaline silicate	10.0
	Sodium carboxymethylcellulose	0.8
	Sodium sulphate	20.0
	Sodium perborate	20.0
	Sodium bicarbonate	5.0
25	Fluorescent agents, preservatives	1.1
	Water	to 100.0

The composition was made by slurry making and spray drying all of the ingredients except for the sodium perborate and sodium bicarbonate which were separately
30 added in particulate form to the spray dried base powder. The sodium bicarbonate used was a commercially available product having a wide particle size range of from less than 125 microns to about 1,400 microns. The other ingredients of the composition had a mean particle size
35 between 500 and 550 microns.

On addition of the composition to distilled water at 25°C at a concentration of 1% the initial peak pH reached 10.5 and the final pH after 15 minutes was found to be 10.3. This compares very favourably with the initial pH of 11.5 and a final pH of 10.6 for a similar composition which did not contain the sodium bicarbonate, the balance being made up with additional sodium sulphate.

In a further test the level of sodium bicarbonate was decreased to 2.5%, with adjustment to the sodium sulphate content in the composition. It was then found that the initial peak pH of this composition was 10.9 dropping to 10.4 at equilibrium.

The above procedure was repeated except that the particle size range of the sodium bicarbonate was changed and the level of sodium silicate was increased to 13% with consequent adjustment of the sodium sulphate content. The peak and equilibrium pHs were then determined as before with the following results:

<u>Sodium bicarbonate particle size</u>	<u>peak pH</u>	<u>equilibrium pH</u>
less than 150 microns	10.6	10.5
180-250 microns	10.8	10.4
1000-1400 microns	11.1	10.4

These results demonstrate the benefit of using the sodium bicarbonate of smaller particle size.

Each of the above compositions according to the invention were found to have good detergency properties when evaluated for fabric washing.

Further detergent compositions were prepared to the same formulation as above except that the sodium bicarbonate was replaced by 5% and 10% of sodium sesquicarbonate, with consequent reductions to the sodium sulphate contents. The initial peak pHs of these compositions in the 1% concentration at 25°C were found to be 10.9 and 10.7 decreasing respectively to 10.5 and 10.4 at equilibrium. In the absence of the sesquicarbonate the peak pH was 11.5 decreasing to 10.6 at equilibrium, thereby showing a substantial benefit for the addition of the sodium bicarbonate in the form of the sesquicarbonate.

EXAMPLE 2

A powdered detergent composition was prepared to the following formulation by admixing the ingredients:

	<u>Ingredient</u>	<u>%</u>
5	Sodium alkyl benzene sulphonate	28.0
	Trisodium orthophosphate	6.5
	Sodium carbonate	15.0
	Sodium bicarbonate	15.0
	Sodium alkaline silicate	10.0
10	Sodium sulphate	10.0
	Water	to 100.0

The mean particle size of the sodium bicarbonate was about 450 μ while the mean particle size of the remaining components was in the order of 500-550 μ .

15 This composition was found to give an initial pH on addition to water of 10.5, decreasing to 9.6 after 14 minutes, which was acceptable for a product intended for fabric washing by hand. The product was also evaluated under typical Far Eastern wash conditions, in cool water
20 and at a low liquor to cloth ratio, when good results were obtained.

EXAMPLE 3

Three powdered detergent compositions were prepared to the following formulations by admixing the ingredients:

25	<u>Ingredient</u>	<u>% by weight</u>		
		<u>A</u>	<u>B</u>	<u>C</u>
	Sodium alkyl benzene sulphonate	28	28	28
	Trisodium orthophosphate	6.5	-	6.5
	Sodium carbonate	15	30	30
30	Sodium bicarbonate	15	-	-
	Sodium alkaline silicate	10	10	10
	Sodium sulphate	10	10	10
	Water	-----to 100-----		

35 The mean particle size of the sodium bicarbonate was about 450 μ while the mean particle size of the remaining components was in the order of 500-550 μ .

Each of these formulations was used to wash three different test cloths under the same conditions. The detergency of the formulations was compared by measuring the change in reflectance at 460 μ ($\Delta 460^*$) before and after washing. The initial and equilibrium pH of a 1% by weight solution of each formulation was also measured. The results were as follows:

Formulation	<u>A</u>	<u>B</u>	<u>C</u>
$\Delta 460^*$			
10 - Test Cloth I	34.1	28.3	34.3
- Test Cloth II	6.7	4.2	6.9
- Test Cloth III	23.0	20.2	23.2
Initial pH	10.45	11.1	10.95
Equilibrium pH	9.6	10.3	10.3

15 The test cloths used in Example 3 were as follows:

- Test Cloth I - A mixture of sebum fatty acids and carbon black impregnated into cotton poplin.
- 20 Test Cloth II - A mixture of bandy black clay, a non-ionic detergent and a cationic detergent impregnated into cotton cloth.
- Test Cloth III - A mixture of ground nut oil, Indian ink, casein and iron oxide impregnates into cotton sheeting.

C L A I M S

1. A detergent composition containing at least one synthetic detergent active compound and an alkalimetal orthophosphate detergency builder, characterised in that the composition further contains discrete particles of sodium or potassium bicarbonate and in that the composition yields an initial pH of not more than about 11.0 and an equilibrium pH of between about 9.0 and about 11.0 when dissolved in distilled water at 25°C at a concentration of 1% by weight.

2. A detergent composition according to Claim 1, characterised in that the composition contains from about 1% to about 20% by weight of said sodium or potassium bicarbonate.

3. A detergent composition according to Claim 1, characterised in that the mean particle size of said sodium or potassium bicarbonate is less than about 1000 microns.

4. A detergent composition according to Claim 1, characterised in that the composition contains from about 2% to about 30% by weight of said alkalimetal orthophosphate.

5. A detergent composition according to Claim 1, characterised in that said at least one synthetic detergent active compound and said alkalimetal orthophosphate are present in the form of a spray-dried powder.

6. A detergent composition according to Claim 1, characterised in that the composition comprises:

from about 5% to about 30% of a synthetic anionic, nonionic, amphoteric or zwitterionic detergent compound or mixture thereof;

from about 10% to about 30% of a mixture of sodium tripolyphosphate and an alkalimetal orthophosphate in the ratio of from about 10:1 to about 1:5 parts by weight, the amount of sodium tripolyphosphate being at least about 5% by weight;

from about 2% to about 15% by weight of sodium or potassium bicarbonate particles having a discrete particle size of from about 50 to about 500 microns;

from about 5% to about 15% by weight of sodium silicate;

optionally not more than about 5% by weight of alkalimetal pyrophosphate; and

optionally one or more further ingredients selected from lather boosters, lather depressants, anti-redeposition agents, oxygen-releasing bleaching agents, per-acid bleach precursors, chlorine-releasing bleaching agents, fabric softening agents, inorganic salts, fluorescent agents, perfumes, enzymes, germicides and colourants.

7. A process for the production of a detergent composition characterised by comprising the steps of
- (i) spray-drying a slurry containing at least one synthetic detergent active compound and an alkali-metal orthophosphate detergency builder to form a spray dried base powder; and
 - (ii) admixing with said spray-dried base powder discrete particles of sodium or potassium bicarbonate thereby to form a composition which yields an initial pH of not more than about 11.0 and an equilibrium pH of between about 9.0 and about 11.0 when dissolved in distilled water at 25°C at a concentration of 1% by weight.
8. A process according to Claim 7, characterised in that the mean particle size of said sodium or potassium bicarbonate is less than about 1000 microns.
9. A process according to Claim 7, characterised in that before admixing with said spray dried base powder, said sodium or potassium bicarbonate is treated with a nonionic detergent compound.
10. A process according to Claim 7, characterised in that said sodium or potassium bicarbonate is added in the form of the corresponding sesquicarbonate.



European Patent
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EUROPEAN SEARCH REPORT

0029299
Application number

EP 80 30 3673.0

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl.)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
X	DE - A1 - 2 847 826 (UNILEVER N.V.) * claims 1, 5, 7, 10, 13, 18; page 10, paragraph 2; page 13, paragraph 2; pages 17, 18, examples 1, 2 * & GB - A - 2 007 704 --	1,4, 5,6, 9	C 11 D 3/06 C 11 D 3/10 C 11 D 11/02
D	GB - A - 1 530 799 (UNILEVER LTD.) * complete document * --		
A	US - A - 4 115 307 (J.D. McGILVER) * complete document * --		TECHNICAL FIELDS SEARCHED (Int. Cl.)
P,A	US - A - 4 187 190 (R.L. McLAUGHLIN et al.) * complete document * -----		C 11 D 3/00 C 11 D 11/00
			CATEGORY OF CITED DOCUMENTS
			X: particularly relevant A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: conflicting application D: document cited in the application L: citation for other reasons
			&: member of the same patent family, corresponding document
X	The present search report has been drawn up for all claims		
Place of search Berlin		Date of completion of the search 16-12-1980	Examiner SCHULTZE