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54 **Powder compositions and detergent compositions.**

57 A powder composition for blending into detergent powder compositions comprises agglomerates of sodium perborate hydrate (usually tetrahydrate) fines together with one or more other ingredients of detergent powders, for example builders, water-softeners, bulking agents, sodium silicate, surfactants and chelaing agents. Methods for producing agglomerates including a falling curtain technique and an extrusion technique are provided and detergent powder compositions, including powder dishwashing compositions, comprising the agglomerates are also provided.

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POWDER COMPOSITIONS AND DETERGENT COMPOSITIONS

This invention relates to powder compositions for detergents and particularly to formulations which can be dry-blended into detergent powder compositions and comprising agglomerates of sodium perborate hydrate (usually tetrahydrate) fines. The invention further relates to detergent compositions for dishwashers and to a method for the manufacture of agglomerates of sodium perborate (tetra)hydrate fines for use in detergent powder compositions.

Detergent powder compositions, including dishwashing compositions are well known and comprise dry-blended mixtures of several ingredients selected typically from bleaching agents, surfactants, alkali water-softening agents, builders, bulking agents, binders and dyestuffs or pigments.

Standard sodium perborate tetrahydrate, of particle size about 300 microns, dissolves only slowly in water and is generally deemed to be rather too slow dissolving to be satisfactory at low wash temperature. Sodium perborate tetrahydrate in the form of fines, of particle size about 500 microns, dissolves rapidly in water but the fine particle size presents problems of handling since the material is 'dusty' and tends to segregate and cake in the detergent powder pack. Also, the fines tend to lack thermal in-pack stability in hot climates. For these reasons, sodium perborate monohydrate is now being used to a significant extent in detergent powder compositions. The monohydrate comprises porous particles which dissolve rapidly in water at low temperatures to provide a rapid release of bleach in the washing process. However, the monohydrate is much more expensive than the tetrahydrate.

The present invention resides in the discovery that the valuable rapid-dissolving property of sodium perborate tetrahydrate fines can be utilized in detergent powder compositions whilst the handling and segregation problems associated with it are obviated, by agglomerating the fines into larger particles which contain one or more other ingredients of a detergent powder composition.

According to the invention there is provided a powder composition for blending into detergent powder compositions which comprises agglomerates of sodium perborate hydrate, preferably tetrahydrate, fines together with one or more other ingredients of the detergent powder compositions.

According to the invention also there is provided a detergent powder composition containing agglomerates of sodium perborate hydrate, preferably tetrahydrate, fines together with one or more other ingredients of the detergent powder composition. A particular detergent powder composition provided as a feature of the invention is a powder dishwashing composition.

By sodium perborate tetrahydrate fines is meant a powder material in which the particles are of size from 6 microns to 160 microns. Sodium perborate tetrahydrate fines are temperature sensitive and cannot be agglomerated by spray-drying agglomeration techniques which employ temperatures of above 100° C. A further feature provided according to the invention is a process for producing agglomerates of sodium perborate tetrahydrate fines together with one or more other ingredients of detergent powder compositions, which comprises moistening the fines powder or powder mixture with water or an aqueous solution or dispersion of a substance to be incorporated in the agglomerates and agitating the moistened powder whilst removing water from it at a temperature of from about 20° C to about 60° C. The other ingredient or ingredients of detergent powder compositions to be included in the agglomerates can be blended with the fines powder prior to moistening thereof and/or can be dissolved in the water with which the fines powder is moistened. A preferred moistening solution is water glass solution.

A preferred process for producing the agglomerates comprises tumbling the fines powder or fines powder blends in a rotating inclined vessel to create a falling curtain of the particles, applying a predetermined amount of water, water glass solution or an aqueous solution in the form of a fine spray to the falling curtain of particles such that essentially all of the powder particles are moistened and tumbling the moistened powder particles until agglomerates of the desired size are produced. An alternative process for forming agglomerates comprises moistening the powder mixture with water, water glass solution or an aqueous solution and extruding the moist powder through die plates to produce particles (agglomerates) of uniform size.

The agglomerates are then dried under conditions whereby they are prevented or at least inhibited from sticking together, for example by agitating them during drying or by laying them out in a thin layer in shallow trays. Fluid bed drying is preferred. Both the production and the drying of the agglomerates can be carried out at low temperatures at which the tetrahydrate fines are stable and in particular at temperatures below about 60° C. Agglomerate production and drying can be carried out at normal ambient temperature if desired although in general we prefer to heat the agglomerates at about 30° C to increase the rate of drying them. After drying, the agglomerates powder can be sieved to eliminate unwanted large or small particles.

In a modification of the processes described hereinbefore, the agglomerates may be dried at higher

temperatures, e.g. above 60° C, such that the sodium perborate tetrahydrate is converted into sodium perborate monohydrate. In this way agglomerates are provided which in fact are novel particles of sodium perborate monohydrate or mixed tetrahydrate/monohydrate together with one or more other ingredients of detergent powder compositions. Detergent powder compositions comprising these modified agglomerates are provided according to a further feature of the invention.

The agglomerates are in the form of a powder material, particle size say about 100 to 1000 microns, which can be dry-blended with other powders to form the final detergent powder compositions. Detergent powder compositions containing the agglomerates are provided according to a further feature of the invention.

The other substances which may be incorporated in the agglomerated fines include any of the conventional ingredients of detergent powder compositions. Examples of such substances include the so-called precipitant builders, e.g. phosphates such as sodium tripolyphosphate, zeolites water-softeners (alkaline materials) e.g. sodium carbonate, the so-called bulking agents such as sodium sulphate, sodium silicate, active detergent surfactants and transition metal chelating agents such as EDTA and DTPA.

In addition, substances which presently are not commonly incorporated as ingredients of conventional detergent powder compositions may also be included in the agglomerated sodium perborate tetrahydrate fines, for example sodium bicarbonate and partially-calcined sodium sesquicarbonate and sodium bicarbonate or sesquicarbonate mother liquors.

By way of illustration only, some specific powder composition formulations according to the invention are agglomerates comprising:

- sodium perborate tetrahydrate fines + partially-calcined sodium sesquicarbonate (available under the trade name "Crex" from Imperial Chemical Industries plc)
- sodium perborate tetrahydrate fines + partially-calcined sodium sesquicarbonate ("Crex") + sodium sulphate
- sodium perborate tetrahydrate fines + sodium silicate liquor optionally + partially calcined sodium sesquicarbonate ("Crex").
- sodium perborate tetrahydrate fines + partially-calcined sodium sesquicarbonate ("Crex") + sodium sesquicarbonate mother liquor
- sodium perborate tetrahydrate fines + sodium carbonate + sodium sulphate + sodium silicate liquor .
- sodium perborate tetrahydrate fines + sodium silicate liquor (water glass solution).

It will be appreciated that any of the above specific formulations may include other ingredients such as a surface active agent.

A preferred specific powder composition for dry-blending into a dishwashing powder are agglomerates comprising:

- sodium perborate tetrahydrate fines + sodium carbonate + sodium sulphate + sodium silicate liquor

A preferred dishwashing composition incorporating this agglomerate formulation comprises the agglomerates dry-blended with a bulking agent such as sodium tripolyphosphate, a surfactant and sodium metasilicate. This composition may also contain one or both of an amylolytic enzyme and a proteolytic enzyme.

It will readily be appreciate that many other combinations of sodium perborate tetrahydrate fines with additives are possible and that the formulations of the invention may provide a range of functions in the final detergent powder compositions. We have found that incorporation of sodium silicate or sodium sesquicarbonate mother liquor or both in the agglomerates has the advantage of providing particles exhibiting very low dusting properties. By selecting the detergent powder ingredients employed and the levels thereof, the agglomerates can be tailored to meet particular market requirements and provide:-

- (i) - a rapid rate of dissolution for high bleach delivery at low temperatures,
- (ii) - high porosity to allow loading of the agglomerates with liquid detergent ingredients and/or heat sensitive materials,
- (iii) - controlled particle friability to control dust, and
- (iv) - improved in-pack stability at higher ambient temperatures compared with standard tetrahydrate crystals.

The amount of the other additive in the agglomerated sodium perborate tetrahydrate fines may vary within wide limits but will usually be less than about 50% i.e. from 30% to 50% by weight of the agglomerates. The amount of the agglomerates formulation incorporated in the final detergent powder composition will be such that the final composition contains the usual amount of sodium perborate bleaching additive and the amount of any other additive incorporated in the partial detergent composition

will be such that the final composition contains the usual amount of said other additive.

According to a particular feature of the present invention there is provided a powder dishwashing composition for use in automatic dishwashing machines comprising -

- (a) 25 to 75%, preferably 50 to 60%, by weight of agglomerates of sodium perborate tetrahydrate
- 5 fines, sodium carbonate, a bulking agent and sodium silicate,
- (b) 10 to 40%, preferably 20 to 30%, by weight of a phosphate builder,
- (c) 10 to 40%, preferably 20 to 30%, by weight of sodium metasilicate,
- (d) 0 to 5% by weight of each of an amylolytic enzyme and a proteolytic enzyme, and
- (e) 0 to 5% by weight of a low-foaming surfactant.

10 The amounts of the ingredients in the agglomerate should be such as to provide in the dishwashing composition -

- 1 to 10% by weight of sodium perborate tetrahydrate,
- 10 to 40% by weight of sodium carbonate,
- up to 40% by weight of bulking agent(s), and
- 15 - 2 to 15% by weight of sodium silicate.

The composition is alkaline and in particular has a solution pH of about 12.0 to 13.0, this being the pH of a solution of 3 g/litre of the composition in water. The alkalinity or solution pH of the composition, which should generally be maintained above about 9 if an enzyme is included in order that the enzyme be effective, is largely established and maintained by components (a) and (b) and (c) which exhibit a

20 builder/buffer effect on the solution and are used in sufficient amounts to provide the desired solution pH. The optimum relative proportions of components (a), (b) and (c) are readily determined by simple trial for any particular composition but as a guide the ratio by weight is typically about 2:1:1 of agglomerates (a) : sodium tripolyphosphate (b) : sodium metasilicate (c).

The composition will usually contain one or more bulking agents in an amount of from 20% to 40% by weight contained in the agglomerates. Any bulking agent may be used which does not appreciably inhibit the effectiveness of any enzymes present or adversely modify the alkalinity of the composition. Sodium sulphate is a typical and preferred bulking agent and sodium chloride may also be used. The compositions may contain both sodium sulphate and sodium chloride, for example in a ratio of about 1:0.5 by weight.

The preferred phosphate, component (b), is sodium tripolyphosphate.

30 The composition may contain a low-foaming surfactant and advantageously does contain a small amount, for example up to 5% by weight, of such a component. Any low-foaming surfactant may be employed but will usually be a non-ionic surfactant such as an alkylene oxide condensate in which the alkoxy moiety is derived from ethylene oxide or propylene oxide or a mixture thereof. Examples of suitable surfactants are the well-known ethoxylated straight-chain alcohols. Preferably, the surface active agent is

35 biodegradable.

The composition may contain an amylolytic enzyme and a proteolytic enzyme. Preferably at least one of the enzymes and especially preferably both of the enzymes, if present, are in granular form. The enzymes are used in mixtures in which the ratio by weight of amylolytic enzyme: proteolytic enzyme is from 1:4 to 4:1 and usually from 1:2 to 2:1. If present approximately equal amounts (by weight) of

40 proteolytic enzyme and amylolytic enzyme may be used, typically from 0.5 to 1.5% by weight of each enzyme based on the weight of the composition.

Any amylolytic enzyme and any proteolytic enzyme may be used which at pH10 exhibits at least 50% and preferably at least 60% of its maximum activity. The enzyme mixture is present in the composition in an amount such that the composition has an amylolytic activity of at least 150 KNU (Kilo Novo units),

45 preferably at least 300 KNU, per kilogram and a proteolytic activity of at least 6 Anson units per kilogram. Amylolytic activity in KNU is determined as described in United States Patent Specification No 3,931,034. Proteolytic activity in Anson units is determined as the amount of enzyme which degrades haemoglobin under standard conditions as described in Journal of General Physiology, Vol 22 (1938), page 79.

The compositions of the invention may if desired contain other ingredients known for use in automatic

50 dishwasher compositions, for example sequestering agents, soil-suspending agents, corrosion inhibitors, drainage-promoting agents, crystal modifiers, dyes and perfumes.

The compositions of the invention are presented as powders which can be produced by dry-mixing the powdered/granular materials and spraying any liquid components, e.g. a surfactant, on the the mixture during mixing.

55 The invention is illustrated but in no way limited by the following examples.

EXAMPLE 1

Sodium perborate tetrahydrate fines (PB4 fines) of mean particle size 50 microns (210 g) were dry-blended with partially-calcined sodium sesquicarbonate of mean particle size 120 microns (90 g). The partially-calcined sodium sesquicarbonate is available under the trade name "Crex" from ICI Chemicals & Polymers Ltd. The blended powder mix was introduced into a pan-granulator of diameter 400 mm and depth 100 mm inclined at an angled of 30-40 degrees to the horizontal and rotating at 25-30 rpm. The mixture was tumbled for 2 minutes at ambient temperature using a wall scraper to remove particles adhering to the wall and create a falling curtain of the particles, whilst demineralised water (50 ml) was sprayed onto the falling curtain from a spray nozzle of diameter 0.2 mm. The moistened powder mixture was then tumbled for a further 2 minutes to produce agglomerate granules which were then removed from the pan-granulator, placed in thin layers in shallow trays and dried overnight in an oven at 30 °C. The dry agglomerate granules were then removed from the trays and sieved to remove particles larger than 850 microns and particles smaller than 150 microns.

The agglomerates exhibited the following properties

Mean particle size = 300 microns

Dissolution time (1) = 15 seconds

% available oxygen (2) = 6.8

Friability (3) = 18%

(1) - ISO test method 3123; 2 g/l, 90% of full conductivity at 25 °C.

(2) - ISO test method 1917; the free oxygen of the dissolved agglomerates at 2 g/l and 25 °C, determined by titration against standard potassium permanganate solutions.

(3) - % breakdown of agglomerates to below 150 microns caused by a high-velocity air jet.

EXAMPLE 2

PB4 fines of mean particle size 50 microns (150 g) was dry-blended with "Crex" (64 g) and anhydrous sodium sulphate (86 g) and the mixture was agglomerated using 50 ml of demineralised water and the agglomerates dried as described in Example 1.

The following properties were determined:-

Mean particle size = 400 microns

% available oxygen = 6.1

Dissolution time = 6 seconds

Friability = 23%

EXAMPLE 3

The procedure described in Example 2 was repeated except that instead of 50 ml of demineralised water there was used 30 ml of a 50% by weight solution of water glass in demineralised water. The water glass had the composition:-

18% Na₂O : 29.5% SiO₂ : 52.4% water

The following properties were determined.

Mean particle size = 560 microns

% available oxygen = 5.8

Dissolution time = 28 seconds

Friability = 15%

EXAMPLE 4

The procedure described in Example 1 was repeated except that instead of 50 ml of demineralised water there was employed 50 ml of a 5% aqueous solution of sodium sesquicarbonate.

The following properties were determined:-

Mean particle size = 560 microns
 % available oxygen = 6.3
 Dissolution time = 11 seconds
 Friability = 26%

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EXAMPLE 5

PB4 fines of mean particle size 50 microns (220 g) were dry-blended with sodium carbonate of size 120
 10 microns (40 g) and anhydrous sodium sulphate (140 g). The mixture was agglomerated as described in
 Example 1 using 50 ml of a water glass solution as described in Example 3.

The following properties were determined:-

Mean particle size = 345 microns
 % available oxygen = 7
 15 Dissolution time = 28 seconds
 Friability = 27%

A powder dishwashing composition was then blended to the following formulation:-

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	% by weight
Agglomerates (as above)	60
Sodium metasilicate powder	18
Sodium tripolyphosphate powder	20
Surfactant ("Ukanil" 2136)	2
	100

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"Ukanil" 2136 is an ethoxylated alcohol and is available from Imperial Chemical Industries plc.

30 The liquid surfactant was sprayed on to the sodium tripolyphosphate and then the three powders were
 dry-blended together in a Kenwood food mixer.

A sample of the dishwashing composition was used to clean dirty plates in a domestic automatic
 dishwashing machine (Indesit 2260E). The water softener reservoir in the machine was kept topped-up with
 sodium chloride and a rinse-aid was employed during the rinse cycle of the cleaning operation. 15 g of the
 35 powder dishwashing composition was loaded into the machine in each trial.

The dirty plates being cleaned were soiled by coating them with both a greasy soil and a protein soil
 and baking them at 150 °C in an oven. Some plates were baked for 5 minutes, some for 7.5 minutes and
 others for 10 minutes.

The greasy soil was a mixture of peanut butter (5), butter (2), lard (2.5) and mineral oil (2), the figures
 40 indicating parts by weight.

The protein soil was a mixture of raw egg (5) peanut butter (1), lard (1), butter (1) and milk (1), the
 figures being parts by weight.

After a full wash-cycle (including drying) in the machine, all of the dry plates were removed and found
 to be clean.

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EXAMPLE 6

The porosity of the agglomerates produced in Example 5 was determined by spraying a liquid non-ionic
 50 surfactant (known as A7) onto the agitated powder and measuring the amount of the surfactant which could
 be loaded onto the powder before its flow properties were adversely affected. An adverse effect on flow
 properties was quantified by measuring the change in the angle of repose of the powder.

Results:-

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% loading of Surfactant (w/w)	Angle of Repose (Degrees)
0	33
10	34
15	36
20	40
25	42

EXAMPLE 7

The storage stability at high ambient temperature of the agglomerates produced in Examples 1, 3 and 5 was assessed by placing 20 g samples of the agglomerates in sealed containers and maintaining them at 35°C for 50 days. For purposes of comparison, a Control test was carried out using standard sodium perborate tetrahydrate of mean particle size 300 microns.

Results:-

Powder from Example	Time (days)				
	2	5	10	30	50
1	0	1	1	2	2
3	0	0	0	0	0
5	0	0	0	0	0
Control	1	1	2	3	3

Code: 0 = no caking, powder unchanged and free-flowing
 1 = slight caking, dispersed with shaking
 2 = severe caking, not dispersed with shaking
 3 = caking of sample to a lump of solid

The results show the high in-pack stability of the agglomerates compared with standard sodium perborate tetrahydrate crystals at 35°C and the results for the agglomerates of Examples 3 and 5 compared with those for the agglomerates of Example 1 indicate the beneficial effect upon resistance to caking of incorporating sodium silicate in the agglomerates.

The procedural steps described in Example 1 represent a general procedure for agglomerating fine powders and are illustrative of the process according to the invention for making agglomerates. The procedure has now been scaled up and operated with 1 tonne batches per hour on a number of commercially-available powders.

EXAMPLE 8

A powder dishwashing composition was blended to the following formulation:-

	% by weight
Agglomerates (as in Example 5)	50
Sodium metasilicate	23
Sodium tripolyphosphate	25
Surfactant ("Ukanil" 2136)	2
	100

The composition was used to clean dishes as described in Example 5 and after the treatment all of the plates had a clean appearance.

EXAMPLE 9

PB4 fines of mean particle size 50 microns (200 gm) were sprayed with a solution of water glass and then dried. The amount and type of water glass (sodium silicate solution) was varied (see attached table) and the properties measured.

Sodium Silicate Solutions Used						
Type	Wt Ratio	% By Weight				Viscosity cp
		SiO ₂ +Na ₂ O	Na ₂ O	SiO ₂	Water	S.G.
A120	1.6	18.1	29.5	52.4	1.6	1400
E100	2.21	13.1	29.2	57.6	1.5	220
Q79	3.3	8.9	29.2	61.9	1.4	330

Agglomerate Properties						
Samples	Water glass type	% amount by wt	Mean Particle Size	% O ₂	R.O.S	Friability
1	A120	10	350	9.8	22	21
2	A120	15	420	9.7	24	18
3	E100	10	370	9.7	37	19
4	E100	15	500	9.6	36	20
5	Q79	10	420	9.7	95	14
6	Q79	15	550	9.8	114	12

EXAMPLE 10

The agglomerate of Example 9 Sample 4 was further dried by heating the powder with hot air at 120 °C till the powder temperature reached 60 °C for a period of one hour.

The available oxygen was 12.2%. This was then sprayed with a liquid nonionic surfactant (A7) as in Example 6.

The angle of repose was measured at various loadings of the surfactant.

% Loading of surfactant w/w	Angle of Repose (Degrees)
0	35
5	35
10	36
15	37
20	38

The agglomerate remained free flowing up to a substantial loading of the surfactant.

Drying of the agglomerate is not limited to Oxygen of 12.2% and covers all levels from 9.5% to 15% oxygen.

EXAMPLE 11

The combination of PB4 fines and E 100 water glass sample 4 in Example 9 was extruded whilst in the damp state through a 2 mm dye plate. This formed extrudates of 2 mm in diameter and on average about 2 mm in length. These extrudates were dried in the normal manner by fluidising in hot air.

The extrudates (agglomerates) demonstrated the following properties:

Mean particle size 200 μ - uniform

% Available oxygen 9.6

Dissolution time 32 seconds

Friability hard, none dusty

The needle shaped particles showed excellent dissolution rates for a large size particle together with uniform granulometry on handling.

Claims

1. A powder composition for blending into detergent powder compositions which comprises agglomerates of sodium perborate hydrate fines together with one or more other ingredients of the detergent powder compositions.

2. A powder compositions as claimed in claim 1 wherein the sodium perborate hydrate is sodium perborate tetrahydrate.

3. A powder compositions as claimed in claim 1 wherein the sodium perborate hydrate is sodium perborate monohydrate.

4. A powder composition as claimed in claims 1, 2 or 3 wherein the amount of the other additive(s) in the agglomerates is from 30% to 50% by weight of the agglomerates.

5. A powder composition as claimed in any one claims 1 to 4 wherein the other ingredient(s) in the agglomerate is/are selected from precipitant builders, water-softeners, bulking agents, sodium silicate, surfactants and chelating agents.

6. A powder composition as claimed in any one of the preceding claims wherein the agglomerates are of size 100 microns to 1000 microns.

7. A powder composition as claimed in any one of the preceding claims wherein the other ingredients of the agglomerates are sodium carbonate, sodium sulphate and sodium silicate liquor.

8. A detergent powder compositions containing a powder composition as claimed in any one of claims 1 to 7.

9. A powder dishwashing composition containing a powder composition as claimed in any one of claims 1 to 7.

10. A powder dishwashing composition as claimed in claim 9 comprising agglomerates as claimed in claim 7 dry-blended with a bulking agent, a surfactant and sodium metasilicate.

11. A dishwashing composition as claimed in claim 10 which contains in addition one or more enzymes.

12. A powder dishwashing composition comprising:-

(a) 25 to 75%, by weight of agglomerates of sodium perborate tetrahydrate fines, sodium carbonate, a bulking agent and sodium silicate,

(b) 10 to 40%, preferably 20 to 30%, by weight of a phosphate builder,

- (c) 10 to 40%, preferably 20 to 30%, by weight of sodium metasilicate,
- (d) 0 to 5% by weight of each of an amylolytic enzyme and a proteolytic enzyme, and
- (e) 0 to 5% by weight of a low-foaming surfactant.

13. A powder dishwashing composition as claimed in claim 12 wherein the amounts of the ingredients
5 in the agglomerates is such as to provide in the dishwashing composition:

- 1 to 10% by weight of sodium perborate tetrahydrate
- 10 to 40% by weight of sodium carbonate,
- up to 40% by weight of bulking agent(s), and
- 2 to 15% by weight of sodium silicate.

14. A process for producing agglomerates of sodium perborate tetrahydrate fines together with one or
10 more other ingredients of detergent powder compositions which comprises moistening fines powder or a
fines powder mixture with water or an aqueous solution or dispersion of a substance to be incorporated in
the agglomerates and agitating the moistened powder whilst removing water from it at a temperature of
from 20°C to 60°C.

15. A process as claimed in claim 1 which comprises tumbling the fines powder or fines powder
15 mixture in a rotating inclined vessel to create a falling curtain of the particles, applying the liquid in the form
of a fine spray to the falling curtain of particles and tumbling the moist powder until agglomerates of the
desired size are produced.

16. A process as claimed in claim 14 which comprises moistening the fines powder or fines powder
20 mixture and extruding the moistened powder through holes in a die plate.

17. A process as claimed in claims 14, 15 or 16 in which the agglomerates are dried under conditions
whereby they are inhibited from sticking together.

18. A process as claimed in claim 17 wherein the agglomerates are dried by fluid bed drying.

19. A process as claimed in claim 17 or 18 wherein the agglomerates are subsequently heated at a
25 temperature above 60°C to convert the sodium perborate tetrahydrate to sodium perborate monohydrate
and thereby produce agglomerates of sodium perborate monohydrate with one or more other ingredients of
detergent powder compositions.

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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. 4)												
X	US-A-3 154 496 (A.S. ROALD) * whole document * ---	1,2,4-8 ,15	C 11 D 3/39 C 11 D 3/386												
X	US-A-2 975 142 (J. SCHMIDT et al.) * column 1, lines 18-22,65-71; column 2; column 3, lines 3-6; claims 1,2,7 * ---	1,2,5-8													
X	US-A-2 979 464 (H. PISTOR et al.) * column 2, lines 3-8,19-22,30,32-33,64-72; column 3, lines 5-8; claims 5,7-11 * ---	1,2,4-8													
A	* claims 1-4 * ---	9-13													
X	FR-A-1 129 870 (UNILEVER N.V.) * whole document * ---	1,2,4-8													
X	US-A-3 161 597 (J.H. YOUNG) * column 1, lines 52-58,67-72; column 2, lines 33-35; column 3, lines 41-71 * ---	1,2,4-8													
A	* claims * ---	9-13													
A	US-A-2 863 835 (H.A. GOLDSCHMITH) * column 1, lines 50-72; column 2, line 1; claims * ---	1,2,5-8	TECHNICAL FIELDS SEARCHED (Int. Cl.4) C 11 D 3/00												
X	US-A-3 664 961 (R. NORRIS) * abstract; column 1, lines 71-75; column 2, lines 1-2,32-66; column 16, lines 19-32; claims 1,2,4 * ---	1,2,8													
A	---	9-13													
X	US-A-3 451 935 (A.S. ROALD) * abstract; column 4, lines 23-25; claims 1,3,7,8,10 * -----	8													
The present search report has been drawn up for all claims															
Place of search BERLIN		Date of completion of the search 25-08-1988	Examiner PELLI B												
<table border="0"><tr><td>CATEGORY OF CITED DOCUMENTS</td><td>T : theory or principle underlying the invention</td></tr><tr><td>X : particularly relevant if taken alone</td><td>E : earlier patent document, but published on, or after the filing date</td></tr><tr><td>Y : particularly relevant if combined with another document of the same category</td><td>D : document cited in the application</td></tr><tr><td>A : technological background</td><td>L : document cited for other reasons</td></tr><tr><td>O : non-written disclosure</td><td>.....</td></tr><tr><td>P : intermediate document</td><td>& : member of the same patent family, corresponding document</td></tr></table>				CATEGORY OF CITED DOCUMENTS	T : theory or principle underlying the invention	X : particularly relevant if taken alone	E : earlier patent document, but published on, or after the filing date	Y : particularly relevant if combined with another document of the same category	D : document cited in the application	A : technological background	L : document cited for other reasons	O : non-written disclosure	P : intermediate document	& : member of the same patent family, corresponding document
CATEGORY OF CITED DOCUMENTS	T : theory or principle underlying the invention														
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