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(21) International Application Number: PCT/G (22) International Filing Date: 8 July 1999 (30) Priority Data: 9814968.5 10 July 1998 (10.07.98) (71)(72) Applicant and Inventor: HINTON, Gerald [GB/GB]; Eastfields, Stonebyres, Lanark ML11.5 (74) Agent: MURGITROYD & COMPANY; 373 Scotl Glasgow G5 8QA (GB).	G , Thom PUW (GE	BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAP patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR NE, SN, TD, TG).
blend comprises a sulphate, carboxymethyl cellulose and and does not include a zeolite, a perborate or a phosp packaging for at least nine months and wherein the cor	d a nonio hate. The mposition	ate and a blend which encapsulates the percarbonate is described. The ic surfactant. The detergent composition comprises sodium metasilicate composition is capable of being stored in a water-soluble PVA film comprises between 1 % and 15 % percarbonate. The composition can sition can be compressed into a tablet format and used as a laundering

"Detergent" 1 2 3 This invention relates to a detergent product 4 formulations which can be packaged in water soluble 5 film. The invention also relates to detergent formulations excluding phosphates. 6 7 8 A product of the invention is ideally for use in the 9 laundering and conditioning of industrial and domestic 10 man-made and/or natural fabrics in semi-automatic or automatic washing machines. It may also be used in 11 12 dishwashers. For convenience purposes it is useful if 13 this can be achieved by means of a soluble single compartment sachet containing varying amounts of 14 15 bleaching detergents including for example sodium 16 percarbonate. Typically a sachet may be made from a 17 water soluble film such as PVA. 18 19 Conventional laundering detergents comprise perborates 20 and zeolites and these compounds are not stable in 21 water soluble film. Previous attempts have been made 22 to manufacture fully built detergent and conditioner 23 systems in a sachet have required a twin compartment 24 sachet manufactured from a perforated film. These 25 fully built detergent and conditioner systems generally contain bleaching agents. Sodium percarbonate is recognised in this field as a bleaching agent. However, use of percarbonate in sachets is not popular as it is unstable when combined with other components of a high moisture content.

Twin compartment sachets have a disadvantage in that they require greater mechanical action to dissolve the sachet and thus have long dispersion times. Also, they are expensive to manufacture.

Additionally, the perforated film used in these twin compartment sachets does not confer a significant shelf life to the components contained within the sachet, wherein the oxidising power of the bleaching agent is reduced.

10 Also, typically these formulations contain zeolites. These have high moisture content which affects the mechanical properties of the film e.g. the pliability.

Pollution problems arise from the use of phosphates in detergent compositions.

Phosphates are required as solubilisers and aid detergency. However, they have a detrimental effect on the environment. Forthcoming European legislation will require the amount of phosphate released into the environment to be minimised.

It is an object of the present invention to provide a detergent or bleaching agent for cleaning in laundries, or in domestic washing machines or dishwashers which is phosphate free.

Throughout the specification, unless the context requires otherwise, the word "comprise" or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated integer or group of integers but not the exclusion of any other integer or group of integers.

The preceding discussion of the background art is intended to facilitate an understanding of the present invention only. It should be appreciated that the discussion is not an acknowledgement or admission that any of the material referred to was part of the common general knowledge in Australia as at the priority date of the application.

According to one aspect of the present invention there is provided a detergent composition comprising a

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1 granulated percarbonate and a blend which encapsulates the percarbonate, the blend comprising a sulphate, 3 carboxymethyl cellulose and a nonionic surfactant, 4 wherein the detergent composition comprises sodium 5 metasilicate and does not include a zeolite, a perborate or a phosphate, and wherein the composition 6 7 is capable of being stored in a water-soluble PVA film 8 packaging for at least nine months and wherein the 9 composition comprises between 1% and 15% percarbonate. 10 Preferably the detergent formulation uses sodium 11 percarbonate, carboxy methyl cellulose, sodium 12 13 sulphate, a nonionic surfactant blend, sodium silicate 14 and a phosphate substitute, ie an alternative to the phosphates used in detergent formulations. 15 16 17 The phosphate substitute may be selected from the group 18 comprising silicates, carbonates and polycarboxylates, 19 which should be wholly or substantially anhydrous. 20 21 The invention specifically excludes polyphosphates and 22 preferably includes polyacrylates in powdered or liquid 23 form. In particular formulations the amount of emulsifier metasilicate is also increased and the 24 25 amount of carboxymethyl cellulose is decreased and bleach activator is added when liquid polyacrylate is 26 27 used. 28 29 Preferably the detergent further comprises bleach 30 activator such as TAED. 31 32 The detergent may further comprise at least one ingredient chosen from the group comprising linear 33 34 alkylbenzene sulphonate, sodium lauryl sulphate, sodium 35 carbonate, low foam wetting agent, perfumes, optical 36 brighteners, salts, pigments and enzymes.

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1 In one embodiment the detergent formulation is a 2 laundering product. 3 4 In an alternative embodiment the detergent is a machine 5 dishwashing product. 6 7 Suitably the laundry or dishwashing product according 8 to the present invention is packaged in PVA film. A 9 product of the invention is stable in PVA film compared 10 to other products containing zeolites and perborates. 11 12 Suitably the film is 20-80 microns thick. 13 14 The product may be incorporated into a tablet form. 15 16 The granulated form of percarbonate in the above permits efficient bleaching action of the laundry 17 18 product whilst not effecting the stability of the 19 product in storage. 20 21 While modifications and improvements may be made 22 without departing from the scope of this invention, the following is a description of the invention, with 23 24 reference to the accompanying diagrams: 25 26 Figures 1a and 1b illustrate a soluble single 27 compartment sachet produced from a polyvinyl alcohol 28 (PVA) film filled with product and heat sealed. 29 30 Figures 2a and 2b illustrate a soluble single 31 compartment sachet produced from PVA film by 32 thermoforming. 33 The sachets are sealed such that they contain a laundry 34 35 and conditioning powder without spillage or air borne 36 contamination which can cause irritation to eyes and/or

-6-

5

1 skin etc.

2

3 Example 1

4

The laundry and conditioning powder can be in the form of a super concentrate with a bulk density of not less than 0.75kg/l. The laundry and conditioning powder is preweighed and packed in 50g batches which is sufficient to launder 4.5kg dry weight of mixed fibres (normal soiling) in either hard or soft water

11 conditions.

12

13 In order to determine the storage and durability of 14 sachets containing laundry and conditioner, the sachets 15 were treated as follows:

16

17 1. Laundry and conditioner products including the 18 granulated percarbonate compound were sealed in 19 PVA sachets under atmospheric conditions and 20 stored in various temperatures.

21 22

23

24

 Sachets containing the laundry and conditioner products were sealed in a PVC container under atmospheric conditions as stored at various temperatures.

25 26

The samples of both 1 and 2 above were stored for nine
months whereupon they were added to separate washing
cycles. In both cases the samples were found to be
stable (both before use and after storage) with no
deterioration of the product or the sachet containing
the product.

33

34 Sachets were dissolved in cold water (20°C) using a 35 combination of water flow and mechanical agitation 36 whereupon sachets and contents were typically

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completely dissolved with no residue within 90 seconds.

1 2

3 The polyvinyl alcohol film was 30-85 microns (+/- 10-

15%) thick. The polyvinyl alcohol film is both 4 5

biodegradable and nonhazardous.

6

The process for producing the sachets according to 7

figures 1a and b containing the dishwashing, laundry

9 and/or conditioner product requires a form filling

machine modified such that the sachet is produced with 10

a minimum number of folds and seals. 11

12

13 Alternatively thermoforming of film can be used to

14 produce filed sachets as illustrated in Figure 2.

15

1	Example 2		
2			
3 .	Typical detergent product formulations		
4			
5	Linear alkylbenzene sulphonate (LABS)	0.5%	
6	Sodium Percarbonate	1-15%	
7	Carboxy Methyl Cellulose (CMC)	1-5%	
8	Sodium Sulphate Anhydrous	5-35%	
9	Sodium Carbonate	0-35%	
10	Nonionic Surfactant Blend	1-10%	
11	Low Foam Wetting Agent	0-2%	
12	Sodium Metasilicate	1-30%	
13	Perfumes	0-1.5%	
14	Optical Brighteners	0-1%	
15	Salts	0-10%	
16	Enzymes (blended)	0-5%	
17	Copolymer	0-10%	
18	Water Soluble Dye Pigment	0-2%	
19	Sodium Polyacrylate	1-30%	
20	(eg Sandoperol)	(preferably	
21		2-12%)	
22	Bleach Activator	0.1-2%	
23			
24	Minor ingredients as required.		
25			
26	Linear alkylbenzene sulphonate may be r	eplaced with 0.5	
27	to 10% sodium lauryl sulphate to produce a more		
28	environmentally friendly product.		
29			
30	Varying amounts of the above components	may be used	
31	depending on the type of product required, i.e. for		
32	laundering, dishwashing or conditioning	J•	
33			
34	In the following examples nonoinic sur	factant blend and	
35	low foam wetting agent are together re	ferred to as	
36	liquid blend.		

```
1
      Example 3
2
3
     Laundry Product using Liquid Polyacrylate
      A laundering product was prepared and packaged in PVA
5
6
     film.
7
8
      The formulation consisted of
9
10
     Linear alkylbenzene sulphate
                                         2%
11
      Sodium Percarbonate
                                         3%
12
      Carboxy Methyl Cellulose
                                         1%
13
      Sodium Sulphate Anhydrous
                                         25%
14
      Sodium Carbonate
                                         28%
15
     Liquid blend
                                         2%
16
     Sodium Metasilicate
                                         30%
17
      Anionic Sodium Polyacrylate
                                         10%
18
      Perfumes
                                         0.8%
19
      Optical Brighteners
                                         0.5%
20
      Salts
                                         2%
21
      Enzymes (blended)
                                         1.5%
22
      Copolymer
                                         2%
23
      Bleach Activator (TAED)
                                         1%
24
25
      Prior to mixing the liquid polyacrylate is blended with
26
      approximately 60% of the anhydrous sulphate and dried.
27
28
      Alternatively, the polycrylate may be mixed with any of
29
      the powdered materials, dispersed through the powder.
30
31
      Example 4
32
33
      Laundry Product
34
35
      A laundering product was prepared and packaged in PVA
      film.
36
```

-10-

1 The formulation consisted of 3 Linear alkylbenzene sulphate 2% 4 Sodium Percarbonate 5.1% Carboxy Methyl Cellulose 1.0% Sodium Sulphate Anhydrous 6 20% Sodium Carbonate 33.0% 8 Liquid blend 2.0% Sodium Metasilicate 9 30% 10 Perfumes 0.8% 11 Optical Brighteners 0.5% 12 Salts (NTA Powder) 2₹ Enzymes (blended) 13 1.5% Copolymer 14 2.0% 15 Bleach Activator (TAED) 1% 16 17 Inclusion of copolymer improved redeposition. 18 19 Production of Formulation 20 The percarbonate was added to the sachet as shown in 21 22 Figure 1 in the form of granules. These granules 23 comprised percarbonate, sulphate and carboxy methyl cellulose in varying amounts together with a blend of 24 25 nonionic surfactants to create a binding agent. These 26 components were processed in order to produce a dust 27 free granule of a diameter not less than 150 microns. 28 29 In order to produce the granules a horizontal type 30 mixer was used. A liquid blend of the abovementioned 31 laundry components was added to the mixer from a high 32 pressure vessel incorporating an agitator. The liquid 33 blend was fed in at a pressure of 60 pounds per square 34 35

36

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10

```
The finished granulated detergent is fully
1
2
     biodegradable and has a stable pH range of 10-11, which
3
     does not affect the PVA film stability as used in this
     invention.
5
6
     Trials have shown that using nonionic surfactants
     comprising alkyl aryl polyglycol ethoxylates through
8
     the alkyl group C_{6\text{--}12} (typically C_{8\text{--}10}) is stable and
9
      gives the best results even after storage in excess of
10
      9 months.
11
12
      A typical encapsulation blend is as follows:
13
14
      Sodium Sulphate (Anhydrous)
                                     5-98%
15
      Carboxy Methyl Cellulose
                                     1-25%
16
      Nonionic Surfactant blends
                                     1-40%
17
18
      Alternative nonionic surfactant blends comprising
19
      alcohol polyglycol ethoxylate oxide in the range of
20
      0.5-5% have been used successfully.
21
22
      The advantages of the invention and of the ways in
23
      which the disadvantages of the previously known
24
      arrangements are overcome include encapsulation of a
25
      percarbonate with a powder/liquid blend forming a
26
      granular product of suitable size and strength for use
27
      in a hot or cold process.
28
29
      A single component sachet sealed such that the
30
      percarbonate does not decompose in the detergent
31
      contained within the sachet.
32
33
      Upon dissolution the PVA leaves no residues i.e. it is
34
      fully dissolved.
35
36
```

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```
1
      No mechanical action is required to dissolve the PVA
2
      film.
3
     The encapsulation process extends the shelf life of
4
5
      fully built detergent within the PVA sachet.
6
7
     In the super concentrated form, a laundering
8
      formulation normally requires 50g per 4.5kg (dry
9
      weight) wash with normal soiling.
10
11
      Example 5(a)
12
13
      Sodium Sulphate
                                        20 %
14
      Sodium Carbonate
15
      Sodium Metasilicate Anhydrous
                                        15 %
16
      Sodium Percarbonate Peroxyhydrate 10 %
17
18
      Alcohol Polyglycol Ethoxylate
19
      Oxide + Cationic Resin Dye
20
      Transfer Inhibitor
                                         5 %
21
      LABS
                                         1.2%
      NTA Powder/EDTA Powder
22
23
      Enzymes (optional)
                                         0.5%
24
      Co Polymer
                                         1 %
25
      TAED
                                         1 %
26
      OBA (optional)
                                          0.3%
27
      Perfume (optional)
28
29
      Example 5(b)
30
31
      Sodium Sulphate
                                         14 %
32
      Sodium Carbonate Dense
                                         50 %
33
      Sodium Metasilicate Anhydrous
                                         10 %
34
      Alcohol Polyglycol ether +
35
      Cationic resin Dye
36
       Transfer Inhibitor
                                         10 %
```

-13-

34

35 36 TAED

-14-

1.3%

13

1	Example 7	
2		
3 .	Sodium Metasilicate 20 %	
4	Sodium Sulphate 20 %	
5	Sodium Carbonate Dense 30 %	
6	Sodium Percarbonate 10 %	
7	LABS 1.5%	
8	CMC 2 %	
9	NTA/EDTA 5 %	
10	Perfume 0.5%	
11	TAED 2 %	
12	Co Polymer 2 %	
13	Liquid Ethoxylate Alcohol/Alcohol	
14	Polyglycol Oxide Dye Transfer	
15	Inhibitor 7 %	
16		
17	This formulation uses no OBAs for use on coloured	
18	fabrics, and no enzymes, such that it is non-	
19	biological.	
20		
21	The formulations in Examples 5(a), (b), 6 and 7 can be	рe
22	prepared and packaged in PVA film in a similar manner	:
23	to that in Examples 1-4.	
24		

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14

1	CLAIM	ıs
2		
3 .	1.	A detergent composition comprising a granulated
4		percarbonate and a blend which encapsulates the
5		percarbonate, the blend comprising a sulphate,
6		carboxymethyl cellulose and a nonionic surfactant,
7		wherein the detergent composition comprises sodium
8		metasilicate and does not include a zeolite, a
9		perborate or a phosphate, and wherein the
10		composition is capable of being stored in a water-
11		soluble PVA film packaging for at least nine
12		months and wherein the composition comprises
13		between 1% and 15% percarbonate.
14		
15	2.	A composition as claimed in claim 1 wherein the
16		percarbonate is sodium percarbonate.
17		
18	3.	A composition as claimed in claim 1 or claim 2
19		wherein the sulphate is sodium sulphate.
20		
21	4.	A composition as claimed in any of the preceding
22		claims wherein the surfactant is alkyl (C_6 to C_{12})
23		aryl polyglycol ethoxylate.
24		
25	5.	A composition as claimed in any of the preceding
26		claims wherein the composition further comprises
27		at least one of the ingredients chosen from the
28		group comprising linear alkylbenzene sulphonate,
29		sodium carbonate, low foam wetting agent,
30		perfumes, cationic surfactant, optical
31		brighteners, salts and enzymes.
32		
33	6.	A composition as claimed in any one of the
34		preceding Claims wherein the composition further
35		includes a phosphate substitute.
36		

- A composition as claimed in Claim 6 wherein the phosphate substitute is selected from the group comprising silicates, carbonates and polycarboxylates.
- 8. A composition as claimed in Claim 7 wherein the phosphate substitute is apolyacrylate.
 - 9. A composition as claimed in Claim 8 wherein the phosphate substitute is sodium polyacrylate.
 - 10. A composition as claimed in any of the preceding claims wherein the composition is a laundering product.
- 10 11. A composition as claimed in any of the preceding claims wherein the composition is a machine dishwashing product.
 - 12. A composition as claimed in any one of the preceding claims wherein the PVA film is 20-80 microns thick.
- 13. A composition as claimed in any one of the preceding claims wherein theproduct is compressed into a table format.
 - 14. A detergent composition substantially as hereinbefore described with reference to Figures 1a and 1b, or 2a and 2b.
 - 15. A detergent composition substantially as hereinbefore described with reference to any one of the examples.

20

Dated this Seventeenth day of November 2003.

Gerald Thomas HINTON Applicant

Wray & Associates Perth, Western Australia Patent Attorneys for the Applicant

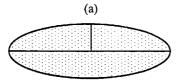
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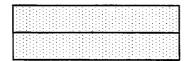
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Figure 1



(b)

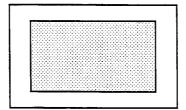


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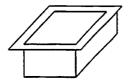
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Figure 2

(a)



(b)



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