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54 **Package containing a powdered detergent.**

57 The invention relates to a package containing a powdered detergent. According to the invention, the powdered detergent is packaged in compressed form in a gas and/or vapour tight foil. The invention further relates to a powdered detergent suitable for use in the package according to the invention, consisting of agglomerates comprising a core, in which the more hygroscopic ingredients are located, and a shell, in which the less hygroscopic ingredients are located. The invention further relates to a method for the preparation of a powdered detergent suitable for use in the package according to the invention, comprising agglomerating a part of the components of the detergent to form primary agglomerates, followed by agglomerating the primary agglomerates with the remaining component(s).

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This invention relates to a package containing a powdered detergent, more particularly to a gas and/or vapour tight package.

Heretofore, powdered detergents have always been packed as loose powder, i.e. the contents of the package were, at least partly, filled with air. Also because of the low density of powdered detergents, such packages occupy a large volume.

A recent development in the field of powdered detergents concerns detergents of high density. By omitting fillers and other less active components, a detergent with a high concentration of detergent-active components is obtained, so that less volume and weight are required to achieve the same detergency. Thus, a saving of volume has already been achieved.

One object of the invention is to provide a package containing a powdered detergent which occupies little volume, without necessarily using the high-density formulation for the powdered detergent.

The invention relates to a package containing a powdered detergent, which is characterized in that the powdered detergent is packed in compressed form in a gas and/or vapour tight foil.

Surprisingly, it has been found that it is possible to package a powder detergent in compressed form without problems arising that involve its stability or its caking into large lumps.

The invention basically comprises three variants of packaging detergents utilizing compression, namely under vacuum, in a shrink-foil or under external overpressure.

In all of these cases the detergent is compressed, so that it occupies a minimal volume and does not set during storage and transport.

According to the first variant of the invention, the detergent is packaged under vacuum, which means that a vacuum is produced after filling of the package, so that the powder is compressed after the package has been closed. This system has long been known for the packaging of ground coffee. A requirement for this variant is that the foil should be reasonably gas tight. Preferably, an underpressure of 0.1 to 0.9 bar is used in this package.

In the second variant of the invention, a shrink-foil is used. The powder is packaged in a foil, which is heated after packaging and then shrinks, thereby compressing the powder. Optionally, after shrinking, the package can be sealed hermetically. However, this is not necessary.

The third variant of the invention comprises packaging the powder under pressure. In practice, this means that the powder is put in the package and, subsequently, the assembly is placed under external pressure and sealed, or an overpressure is created during packaging. A particular requirement for the package in this variant is that it must be resistant to the forces generated as a result of the pressure applied. The overpressure employed preferably ranges from 0.5 to 15 bar.

A surprising advantage of the invention is that for packaging, relatively inexpensive, and often environment-friendly, materials can be used, such as paper or synthetic foils, for example surlyn foil, polypropylene foil or polyethylene foil, optionally coated and/or laminated with a lacquer, nylon, polyester or aluminum.

In principle, the invention can be used for all types of powdered detergents. In view of storage stability and caking tendencies, however, it is preferred to use an adapted formulation. In this formulation the powdered detergent preferably consists of agglomerates which are composed of a core, in which the more hygroscopic ingredients are located, and a shell, in which the less hygroscopic ingredients are located.

The present invention also relates to a powdered detergent which is suitable for use in compressed form, as defined according to the invention.

Such a powdered detergent consists of agglomerates having a core, in which the more hygroscopic ingredients are located, and a shell, in which the less hygroscopic ingredients are located.

More particularly, detergent-active agents and bleaching agents are located in the core, while bicarbonate is incorporated in the shell. The shell can also contain a metal soap.

The proportions of the amounts of the core and the shell can vary within broad limits. These proportions also depend strongly on the composition of the detergent. In general, the core constitutes about 20-80% by weight of the total detergent and the shell about 20-80% by weight.

According to a preferred composition (calculated on a waterfree basis, but including water of crystallization), the detergent consists of

0.1 - 3 parts by weight of metal soap
 5 - 30 parts by weight of detergent
 0 - 20 parts by weight of sodium perborate,
 0 - 5 parts by weight of other components and
 20 - 65 parts by weight of sodium bicarbonate.

10 The detergent agents used are preferably anionic or non-ionic detergents, such as fatty alcohol ethoxylate and/or sodium dodecyl benzene sulfonate. More particularly, fatty alcohol ethoxylate and sodium dodecyl benzene sulfonate are used in equal amounts by weight. As a bleaching agent, preferably sodium perborate is used, preferably with 4 molecules of water of crystallization, although the modification with one molecule of water of crystallization can also be used.

15 Within the scope of the present invention, supplementary additives may be used as well, as is conventional for powdered detergents of the present type. Examples are colourings, optical whitening agents, perfume, enzymes and the like.

The invention also relates to a method for preparing such a powdered detergent, which comprises agglomerating a part of the components of the detergent to form primary agglomerates, followed by
 20 agglomerating said primary agglomerates with the remaining component(s).

First, the more hygroscopic components are agglomerated to form primary agglomerates, whereupon these primary agglomerates are agglomerated with the less hygroscopic components.

This method can be carried out in the conventional apparatus for powdered detergents, using the conventional conditions.

25 The invention is explained in and by the following examples, which are not intended to limit the application.

Example 1: An enzymatic soaking detergent

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	I	II
A Sodium bicarbonate	60	60
B Sodium lauryl sulfate	15	15
35 C Fatty alcohol ethoxylate 7EO	10	10
D Fatty alcohol ethoxylate 25EO	10	10
E Protease granulate	1	1
40 F Cellulase granulate	1	1
G Minors including perfume, and silicon oxide	3	3
45 H Sodium sulfate		30
	100	130

The dose for I is 90 g detergent for a normal washing machine load that is to be soaked. The dose for II is
 50 130 grams.

Half of the amount of A is applied to a fluid bed apparatus. Added thereto is the mixture of C and D in melted state, so finely divided and so slowly that a pulpy, granular product is obtained. During the subsequent cooling period, B is added first, and then the enzyme granulates and the rest of A and the
 55 minors are admixed.

For formulation II, the sodium sulfate together with half the amount of the sodium carbonate is initially present.

After cooling, the product formed is stored in a closed vessel in a room of low humidity. The

temperature of the room is 15 ° C and the humidity is less than 40%. The cooled product is finally packaged in a vacuum-packaging machine in an externally lacquered polyethylene foil package, formed in the machine.

After three months of storage under severe conditions - 30 ° C and 80% relative humidity - the activity of the enzymes in the vacuum-packed product has remained constant within the margin of measurement error, while the enzyme activity in the normally packed product has decreased by well over 10%. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump. This applies to both the new formulation I and the 'old' formulation II with sodium sulfate.

Example 2: An enzymatic prewash detergent

	I	II
A Sodium bicarbonate	39	39
B Sodium carbonate	19	19
C Sodium lauryl sulfate	10	10
D Fatty alcohol ethoxylate 7EO	10	10
E Fatty alcohol ethoxylate 25EO	15	15
F Protease granulate	1	1
G Amylase granulate	1	1
H Minors including perfume, copolymers and tertiary amine (for foam inhibition)	5	5
I Sodium sulfate		30
	100	130

The dose for I is 60 g detergent for a normal washing machine load that is to be soaked.
The dose for II is 87 g.

The total amount of B and half the amount of A is applied to a fluid bed apparatus. Added thereto is the mixture of D and E in a melted state, so finely divided and so slowly that a pulpy, granular product is obtained. During the subsequent cooling period, C is added first, and then the enzyme granulates and the residue of A and the minors are admixed.

For formulation II, the sodium sulfate together with half of the amount of the sodium carbonate is initially present.

After cooling, the product formed is cooled in a closed vessel in a room of low humidity. The temperature of the room is 15 ° C and the humidity is less than 40%. The cooled product is finally packaged in a vacuum-packaging machine in a package that is formed in the machine from a polyethylene foil coated with aluminum.

After three months of storage under severe conditions - 30 ° C and 80% relative humidity - the activity of the enzymes in the vacuum-packed product has remained constant within the margin of measurement error, while the enzyme activity in the normally packed product has decreased by well over 10%. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump. This applies to both the new formulation I and the 'old' formulation II with sodium sulfate.

Example 3: An enzymatic soaking detergent

	I	II
A Sodium bicarbonate	41	41
5 B Soda Oaq	25	25
C Sodium disilicate	3	3
D Sodium dodecyl benzene sulfonate *	13	13
10 E Fatty alcohol ethoxylate 7EO	3	3
F Protease granulate	1	1
G Amylase granulate	1	1
H Minors including perfume,		
15 and silicon oxide	13	13
I Sodium sulfate		30
20 * waterfree	100	130

The dose for I is 90 grams of detergent for a normal washing machine load to be soaked.
The dose for II is 130 grams.

25 Half of the amount of A and the total amount of B are initially present in a Z-blade mixer of sufficient capacity, after which C and D are successively added. After C, D and F have been added, E, heated to about 90 °C, is added so slowly that a product of the desired grain size is obtained. After cooling, the minors are added first, followed by the enzyme granulates and, finally, the residue of A.

30 For formulation II, the sodium sulfate together with half of the amount of the sodium carbonate is initially present. The product formed is stored in a closed vessel in a room of low humidity. The temperature of the room is 15 °C and the humidity is 40%. The finished product is finally packed in a vacuum-packaging machine in an externally lacquered polyethylene foil package, which is formed in the machine.

35 After three months of storage under severe conditions - 30 °C and 80% relative humidity - the activity of the enzymes in the vacuum-packed product has remained constant within the margin of measurement error, while the enzyme activity in the normally packed product has decreased by well over 10%. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump. This applies to both the new formulation I and the 'old' formulation II with sodium sulfate.

40 Example 4: An enzymatic prewash detergent

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	I	II
A Sodium bicarbonate	37	37
B Soda Oaq	21	21
C Sodium disilicate	3	3
D Sodium dodecyl benzene sulfonate *	13	13
E Fatty alcohol ethoxylate 7EO	8	8
F Tertiary amine (methyl, tallow fat alcohol) **	3	3
G Protease granulate	1	1
H Lipolase granulate	3	3
I Minors including perfume, and silicon oxide	11	11
J Sodium sulfate		30
	100	130

* waterfree

** methyl and two tallow fat alcoholsThe dose for I is 60 grams of detergent for a normal washing machine load to be soaked.

The dose for II is 87 grams.

B together with half of the amount of A is initially present in a Z-blade mixer of sufficient capacity, after which C and D are successively added. After this addition, E, heated to about 90 ° C, is added so slowly that a product of the desired grain size is obtained. After cooling, first the minors are added, followed by the enzyme granulates and finally B.

For formulation II, the sodium sulfate together with half of the amount of the sodium bicarbonate and the soda is initially present.

The product formed is stored in a closed vessel in a room of low humidity. The temperature of the room is 15 ° C and the humidity is 40%. The finished product is finally packed in a vacuum-packaging machine in an externally lacquered polyethylene foil package, formed in the machine.

After three months of storage under severe conditions - 30 ° C and 80% relative humidity - the activity of the enzymes in the vacuum-packed product has remained constant within the margin of measurement error, while the enzyme activity in the normally packed product has decreased by well over 10%. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump. This applies to both the new formulation I and the 'old' formulation II with sodium sulfate.

Example 5: An enzymatic main wash detergent

The advantages of the new method are particularly evident in the packaging of main wash detergents based on modern bleaching agents which are oversensitive to traces of moisture, such as D(odecane)-P-(eroxy)-D(ioxy)-C(arboxylic acid):

	A DPDC	20
	B Soda.0aq	40
5	C Sodium disilicate	3
	D Sodium dodecyl benzene sulfonate (waterfree)	13
10	E Fatty alcohol ethoxylate 7EO	8
	F Tertiary amine (methyl, tallow fat alcohol) **	3
15	G Protease granulate	1
	H Minors including perfume and silicon oxide	12
20		100

** (see Example 4)

- 25 The dose is 90 grams of detergent for a normal washing machine (taking in 18 liters of water for washing).
 A is initially present in a Z-blade mixer of sufficient capacity, after which B, D, and C are successively added. Subsequently, E is heated to about 90° C and sprayed on so slowly that a product of the desired grain size is obtained. After cooling, first the minors are added, followed by the enzyme granulates and finally F.
- 30 The product formed is stored in a closed vessel in a room of low humidity. The temperature of the room is 15° C and the humidity is 40%. The finished product is finally packed in a vacuum-packaging machine in an externally lacquered polyethylene foil package, formed in the machine.
- After three months of storage under severe conditions - 30° C and 80% relative humidity - the hydrogen peroxide content and the activity of the enzymes in the vacuum-packed product have remained constant within the margin of measurement error, while they have practically disappeared from the normally packed product. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump.

Example 6: An enzymatic main wash detergent

- 40 The advantages of the new method are also evident in the packaging of main wash detergents on the basis of modern bleach intensifiers which are oversensitive to traces of moisture, such as phthalic anhydride:

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	A Phthalic anhydride	30
	B Perborate.laq	29
5	C Soda	5
	D Bicarbonate	20
	E Fatty alcohol ethoxylate 7EO	10
10	F Tertiary amine (methyl, tallow fat alcohol) **	2
	G Protease granulate	1
15	H Minors including perfume and amine oxide	3
		100
20		

** see: example 4

25 The dose is 90 grams of detergent for a normal washing machine (taking in 18 liters of water for washing).

A is initially present in a Z-blade mixer of sufficient capacity, after which C and D are successively added. Subsequently, E, heated to about 90°C, is sprayed onto the prepared powder mixture, so slowly that a product of the desired grain size is obtained. After cooling, the minors are added first, followed by the enzyme granulates and, finally, B and F.

30 The product formed is stored in a closed vessel in a room of low humidity. The temperature of the room is 15°C and the humidity is less than 40%. The finished product is finally packed in a vacuum-packaging machine in an externally lacquered polyethylene foil package, formed in the machine.

After three months of storage under severe conditions - 30°C and 80% relative humidity - the hydrogen peroxide content and the activity of the enzymes in the vacuum-packed product have remained constant within the margin of measurement error, while they have practically disappeared from the normally packed product. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump.

Example 7: A concentrated, enzymatic main wash detergent for lower temperatures. The new method is also very useful in this modern trend in the detergent industry.

	A Sodium dodecyl benzene sulfonate	42
	B Fatty alcohol ethoxylate 7EO	5
5	C Soda	6
	D Soap	2
	E Disilicate	15
10	F Sodium aluminum silicate (NAS)	25
	G Protease granulate	2
	H Cellulase granulate	2
15	H Minors including perfume	
	and optical whitening agent	1
		100

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The dose is 45 grams of detergent for a normal washing machine (taking in 18 liters of water for washing).

F is initially present in a fluid bed of sufficient capacity, after which A, C, D, and E are successively added. Subsequently, B, heated to about 90 °C, is added so slowly that a product of the desired grain size is obtained. After cooling, the minors are added first, and finally the enzyme granulates.

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The product formed is stored in a closed vessel in a room of low humidity. The temperature of the room is 15 °C and the humidity is about 40%. The finished product is finally packed in a vacuum-packaging machine in a package of externally lacquered polyethylene foil, formed in the machine.

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After three months of storage under severe conditions - 30 °C and 80% relative humidity - the hydrogen peroxide content and the activity of the enzymes in the vacuum-packed product have remained constant within the margin of measurement error, while they have largely disappeared from the normally packed product. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump.

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Example 8: A concentrated, enzymatic main wash detergent for lower temperatures. The new method is also eminently useful in this modern trend in the detergent industry. Also for a less conventional composition.

	A Sodium lauryl sulfate	42
	B Fatty alcohol ethoxylate 7EO	5
40	C Bicarbonate	6
	D Tertiary amine	2
	E Disilicate	15
45	F Bentonite	20
	G Protease granulate	2
	H Cellulase granulate	2
50	H Minors including perfume,	
	polymers and optical	
	whitening agents	6
55		100

The dose is 45 grams of detergent for a normal washing machine (taking in 18 liters of water for washing).

F is initially present in a fluid bed of sufficient capacity, after which A, C, D, and E are successively added. Subsequently, B, heated to about 90° C, is added so slowly that a product of the desired grain size is obtained. After cooling, the minors are added first, and finally the enzyme granulates.

5 The product formed is stored in a closed vessel in a room of low humidity. The temperature of the room is 15° C and the humidity is about 40%. The finished product is finally packed in a vacuum-packaging machine in an externally lacquered polyethylene foil package, formed in the machine.

10 After three months of storage under severe conditions - 30° C and 80% relative humidity - the hydrogen peroxide content and the activity of the enzymes in the vacuum-packed product have remained constant within the margin of measurement error, while they have largely disappeared from the normally packed product. The vacuum-packed powder is reasonably free-flowing, while the normally packed product has coagulated into a lump.

Claims

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1. A package containing a powdered detergent, characterized in that the powdered detergent is packaged in compressed form in a gas and/or vapour tight foil.

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2. A package as claimed in claim 1, characterized in that the detergent is packaged under vacuum, in a shrink-foil or under external overpressure.

3. A package as claimed in claim 2, characterized in that in the package an underpressure of 0.1 to 0.9 bar is maintained.

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4. A package as claimed in claim 2, characterized in that in the package an overpressure of 0.5 to 15 bar is maintained.

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5. A package as claimed in claims 1-4, characterized in that the powdered detergent consists of agglomerates which are composed of a core, in which the more hygroscopic ingredients are located, and a shell, in which the less hygroscopic ingredients are located.

6. A package as claimed in claim 5, characterized in that the shell contains a metal soap.

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7. A package as claimed in claims 1-6, characterized in that the gas and/or vapour tight foil is paper or synthetic foils, for example surlyn foil, polypropylene foil or polyethylene foil, optionally coated and/or laminated with a lacquer, nylon, polyester or aluminum.

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8. A powdered detergent suitable for use in the package as claimed in claims 1-7, consisting of agglomerates comprising a core, in which the more hygroscopic ingredients are located, and a shell, in which the less hygroscopic ingredients are located.

9. A powdered detergent as claimed in claim 8, characterized in that the shell contains a metal soap.

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10. A powdered detergent as claimed in claim 8 or 9, characterized in that detergent-active substances and bleaching agents are incorporated in the core, while bicarbonate is incorporated in the shell.

11. A powdered detergent as claimed in claims 8-10, characterized in that the detergent consists of

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0.1 - 3 parts by weight of metal soap

5 - 30 parts by weight of detergent agent

1 - 20 parts by weight of sodium perborate, and

20 - 65 parts by weight of sodium bicarbonate.

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12. A method for the preparation of a powdered detergent suitable for use in the package as claimed in claims 1-7, characterized by first agglomerating a part of the components of the detergent to form

primary agglomerates, followed by agglomerating said primary agglomerates with the remaining component(s).

- 5 **13.** A method as claimed in claim 12, characterized by first agglomerating the more hygroscopic components to form primary agglomerates, followed by agglomerating said primary agglomerates with the less hygroscopic components.

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