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(54) **SELF-DISSOLUBLE CAPSULE FOR PREPARATION OF WASHING SOLUTIONS**

(57) A self-dissolving capsule for preparation of washing solutions is disclosed, containing chemical materials and probiotic microorganisms.

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Description

[0001] The invention relates to a self-dissolving capsule for preparation of washing solutions, containing chemical materials and probiotic microorganisms. Structure - the capsule is comprised of two or more cavities, wherein one of them is filled with a tablet composed partially of microorganisms.

[0002] Known household chemistry formulations, appearing on the market in form of capsules enclosed in self-dissolving films, and formulations containing microorganisms are mono- and multi-cavity self-dissolving capsules in liquid/liquid, liquid/powder, powder/liquid phase, used for laundry washing, dishwashing, cleaning of hard surfaces such as floors or windows (to a small extent). They contain concentrated, active chemical substances. Whereas the formulations containing microorganisms are products in bottles with an atomizer (trigger) containing nonionic surfactants of natural origin, soaps of natural origin and probiotic strains. These formulations are usually very expensive and directed towards environmentally-friendly households, mainly for cleaning of hard surfaces.

[0003] The object of the invention is improving the available cleaning agents by providing formulations preventing harmful changes in composition of the microbial flora resulting from using aggressive detergents as cleaning agents that firstly cause sterilisation of cleaned surfaces and appliances, which later leads to uncontrolled colonisation of such ecological niches by undesirable microorganisms. This often leads to arising of hard to remove, unpleasant odours (biofilms), e.g. in spaces of washing or laundry washing devices.

[0004] It is especially desired to provide cleaning agents constituting a connection between the classic cleaning method having a disinfecting character and the cleaning abilities of useful bacteria.

[0005] Another technological problem directly involving the invention is providing a cleaning agent of this type in a form fit to apply in currently used washing and laundry washing appliances, especially in dishwashers and automatic washing machines, which are usually configured to use a cleaning agent in a form having a particular tablet shape or self-dissolving capsule containing a cleaning agent. In case of self-dissolving capsules, they are currently manufactured, generally, with a film dissolving in water, containing polyvinyl alcohol (PVOH), and the cleaning agent contained within is usually in liquid form. An appropriate PVOH film is known to people skilled in the art, e.g. from the application WO2014110356. In case of developing such capsules, it is necessary to achieve compatibility between the cleaning agent and the polyvinyl film. This means that the liquid part and eventual tablet part of the capsule should be compatible with the self-dissolving PVOH film. Moreover, the cleaning agent formula should be stable (not subjected to physicochemical changes), and the product should be effective at the same time.

[0006] Unexpectedly, the problems mentioned above have been solved by the present invention.

[0007] The invention relates to a self-dissolving capsule for preparation of washing solutions, characterised in that it contains a washing agents and probiotic microorganisms, wherein the capsule consists of two separate, sealed cavities, the first of which contains the washing agent, whereas the second one contains a lyophilised or concentrated liquid formulation of *Bacillus* species probiotic bacteria, forming spores and displaying growth in temperature up to 65°C, wherein the washing agent is a composition containing the following components: a nonionic surfactant, a polycarboxylate, an anionic surfactant, a complexing agent aiding the washing process and providing gloss, plasticisers, a phosphonate, hydrolytic enzymes, a preservative, a fragrance, functional additives, and water.

[0008] Preferably, the capsule contains probiotic bacteria chosen from the following: *Bacillus subtilis, Bacillus pumilus, Bacillus vallismortis, Bacillus coagulans, Bacillus lactis,* and *Bacillus mojavensis,* preferably spores of *Bacillus coagulans* PCM 1843 strain.

[0009] Preferably, the washing agent contained in the capsule contains the following: a nonionic surfactant in amount of 5 to 30 wt%, a polycarboxylate in amount below 7 wt%, an anionic surfactant in amount of 0 to 35 wt%, a complexing agent in amount of 50 to 75 wt%, a plasticiser in amount below 20 wt%, a phosphonate in amount below 3 wt%, a hydrolytic enzyme in amount below 1 wt%, a preservative in amount below 1 wt%, a fragrance in amount below 1 wt%, a functional additive in amount below 1 wt%, and water in amount below 4 wt%.

[0010] Preferably, the nonionic surfactant is chosen from the group containing the following: C12-C14 ethoxylated alcohols, C8 alkyl glucoside, capryl decyl glucoside, alkyldimethylamine oxide solution, sorbitan sesquioctanoate, alkoxylated fatty alcohol, 2-propylheptanol ethoxilate, 2-ethylhexyl glucoside, alcohol alkoxylate, C8/10 methyl ester, preferably chosen from the group containing: C12-C14 ethoxylated alcohols and C8 alkyl glucoside.

[0011] Preferably, the anionic surfactant is chosen from the group containing the following: sodium p-cumenesulphonate; potassium 4-isopropylbenzenesulphonate; sodium 2-ethylhexyliminodipropionate; sodium cocopropylenediamine propionate; decyl phosphoric acid; potassium salt, sodium alkyl sulphate; sodium 2-ethylhexyl sulphate; ABS acid MEA salt; ABS acid TEA salt; C12-C14 ethoxylated alcohols (1-2.5 EO), sulphates, sodium salts; MEA cocoate; TEA cocoate, preferably chosen from the group consisting of: ABS acid MEA salt; ABS acid TEA salt; C12-C14 ethoxylated alcohols (1-2.5 EO), sulphates, sodium salts; MEA cocoate; TEA cocoate.

[0012] Preferably, the complexing agent is chosen from the group containing the following: sodium carboxymethyl inulin; tetrasodium EDTA; N,N-bis(carboxymethyl)alanine trisodium salt; glutamic acid, N,N-diacetic acid tetrasodium salt; sodium citrate, preferably chosen from the group containing: sodium carboxymethyl inulin; glutamic acid, N,N-

diacetic acid tetrasodium salt.

[0013] Preferably, the polycarboxylate is chosen from the group containing the following: aqueous solution of partially neutralised polycarboxylic acid (sodium salt); aqueous solution of a polyacrylic acid sodium salt; preferably is an aqueous solution of partially neutralised polycarboxylic acid (sodium salt).

[0014] Preferably, the functional additive is chosen from the group containing the following: vinylpyrrolidone/vinylimidazole copolymer; sodium metasilicate; acylamide carboxylic acid, alkanolamine salt; PEG-8 GMIS; Fluorescent Brightener 28; Undeceth-5, undecyl alcohol, sodium lauryl sulphate, caprylyl pyrrolidone.

[0015] Preferably, the plasticiser is chosen from the group containing the following: glycerine, propylene glycol, and sorbitol.

[0016] Preferably, the phosphonate is chosen from the group containing the following: proprietary mixture of organophosphonic acid, sodium salts; aqueous solution of aminophosphonic acid salt.

[0017] Preferably, the preservative is chosen from the group containing the following: 2-bromo-2-nitropropane-1,3-diol, methylchloroisothiazolinone, methylisothiazolinone; methylisothiazolinone, benzisothiazolinone. Preferably, the hydrolytic enzyme is chosen from the group containing the following: protease, lipase, amylase, and cellulase.

[0018] Preferably, a substance from the following table is chosen as a dye, wherein the particularly preferred substances are underlined.

Dye name
Dysol Turquoise AXN50 Liquid
Brilliant Blue E133
Violet 656271
Dysol Red 4B
Rhodamine conc. 500%
Acid Blue FG200%
Acid Pure Green
Dysol Light Blue
Dysol Green
Sanolin Blue AE90
Sanolin Blue NBL
Puricolor Blue FBL
Acid Amaranth
Acid Orange II 143%
Puricolor Red ARE 14
Puricolor Red ARE 52
Alizarin Blue RN-200
Acid Blue R150%
Helion Turquoise Blue FGL166
SensiRinse Orange
SensiRinse Green
SensiRinse Blue
SensiRinse Yellow
SensiRinse Red
SensiRinse Violet
SensiRinse Black
Liquitint Bright Yellow HP

(continued)

Dye name
Liquitint Cyan 15HP
Liquitint Blue HP

[0019] Preferably, a commercially available substance from the following table is chosen as a fragrance, wherein the particularly preferred substances are underlined.

Fragrance name
Colour Action 9904P
Spring Scent 7954P
Olipic 5693P
Calnival 6364P
Active Fresh 5215
Active Fresh 8360
Sensi Fresh 4812
Active Fresh 5214
Sensi Fresh 0912
Air Color 1 (8730006)
Air Color 2 (8730007)
Air Color 3 (8730008)
Air Color 4 (8730012)
Air White 1 (8730009)
Air White 2 (8730010)
Air White 3 (8730011)
Citrus Clean 3779
Citrus Clean 7699
Citrus Clean 3070
EFFPL 42314 Citrus Fizz
EFFPL 42318 Platinum Flower
EFFPL 42341 Platin Freshness
EFFPL 42342 Aloe&Green Fruits
EFFPL 42343 Green Tea&Rose
EFFPL 42344 Pineapple&Tangerine
EFFPL 42345 Strawberry&Rhubarb
EFFPL 42346 Apple Garden
EFFPL 42347 Black Grape

[0020] The invention relates to a self-dissolving capsule for cleaning soiled hard surfaces characterised in that, the soiled surface is washed with an aqueous solution obtained by diluting the capsule of the present invention, defined above, in 2 to 10 litres of water, wherein the cleaned surface is left to dry, after the washing process.

[0021] The proposed form of application of microorganisms enables isolating the components acting antagonistically

in separate cavities, and thus maintaining their high activity and effectiveness. Simultaneously, the product protects the microorganisms from the influence of concentrated materials during the step of enclosing inside the capsule and under practical conditions (administration routes of biocomponents).

[0022] Product according to the invention contains a tablet, which allows obtaining a maximal concentration of substances contained therein.

[0023] The application of microorganisms' technology enables introducing microorganisms along with chemical substances in a single washing bath, also in automatic washing systems (dishwashers, washing machines), by which they aid the cleaning cycle, and by colonisation of outlets and surfaces of the device, they promote the hygiene maintenance.

[0024] The natural microbial enzymes carry out decomposition of organic substances, which provides a possibility of decreasing the amount of chemical substances in the cleaning agents - environmental aspect.

[0025] The microorganisms aid the cleaning processes by extending the washing cycle. By colonisation of the washing surface, the 'good microbes' compete with pathogenic microorganisms present on the washing surface, driving them out and ultimately improving the microflora of the cleaning surface.

[0026] The embodiments of the invention have been described below, although they should not be identified with the scope of the invention defined in the attached claims.

[0027] In regard to the classic cleaning, capsules according to the invention have a corresponding level to that of available traditional products. Their advantage is performing a microbiological cleaning process. In comparison to products not containing microorganisms, in case of capsules according to the invention, a biofilm reduction and improvement of bacterial flora by eliminating pathogenic microorganisms are observed. Applying the capsule with probiotic on hard surfaces reduced survival of potentially pathogenic Gram+ bacteria including *Staphylococcus aureus* and *Enterococcus faecalis.*

[0028] Bacteria inside appliances colonise hard to reach places and elements, and are not fully washed out during rinsing processes. Capsules according to the invention induce a change in the flora in direction of eliminating pathogenic microbes.

Example 1. Administration routes of biocomponents

[0029] Following methods of biocomponents application were verified:

- Single-cavity capsule - the formula basis and biocomponents are encapsulated in the same cavity. The ingredients and the basis are applied jointly from a single cavity.
- Multi-cavity capsule (liquid/liquid) - antagonistic ingredients of the formula are separated. Ingredients are applied from two cavities.
- Multi-cavity capsule (liquid/tablet) - antagonistic ingredients of the formula are separated. Ingredients are applied from two cavities.

[0030] For the concept of multi-cavity liquid/tablet and liquid/liquid capsule, two variants of the test were carried out:

Test package no. 1

[0031] A multi-cavity capsule of self-dissolving film was formed, in liquid/liquid liquid/tablet (12 mm dimeter) phase according to the following criteria: capsule size 30 mm $\times$ 40 mm or approximate, total tablet mass not exceeding 24 g.

Test package no. 2

[0032] A multi-cavity capsule of self-dissolving film was formed, in liquid/liquid liquid/tablet (16 mm dimeter or approximate) phase according to the following criteria: capsule size 30 mm $\times$ 40 mm or approximate, total tablet mass shout not exceed 24 g.

[0033] At the same time, the cleaning processes efficiency test was performed, as well as the washing bath parameters and individual formula components influence on stability and maintaining the activity of biocomponents. For each concept, the possibility of technological manufacturing of the product and the possibility of placing the recommended biocomponent dosage inside a capsule were verified.

[0034] Introducing the tablet also provides a possibility of higher concentration of the compounds, introducing peroxide substances into the formula, mutual separation of peroxide compound and probiotics, which are negatively affected by them, and possible introduction of probiotics in pressed form into the product.

Example 2. Technological capacity assessment of individual capsule forms

[0035] Single-cavity capsule - the formula basis and biocomponents are encapsulated in the same cavity. The components and the basis are applied jointly from a single cavity. There is no possibility of enclosing the water suspension of biocomponents available on the market in a self-dissolving film - dissolving of the film occurs.

[0036] In a preferred embodiment, probiotic in powder form should be enclosed in a self-dissolving film.

1. Multi-cavity capsule (liquid/liquid)

[0037] (Liquid/liquid) capsules using a cavity having 12 mm and 16 mm diameter have been presented in fig. 1. The smaller cavity (intended for biocomponents) has a volume of 5 ml, whereas the liquid part (the larger cavity intended for chemical substances) has a volume of 12 ml. Total mass of the liquid/liquid capsule was about 19 g.

[0038] Due to significant differences in volume, and therefore the dosing time required, the product is extremely difficult technologically. There is no possibility of enclosing the water suspension of biocomponents available on the market in a self-dissolving film - dissolving of the film occurs. The probiotic content contained in the smaller cavity is too low comparing to that recommended required in a washing bath.

[0039] A multi-cavity liquid/12 mm diameter tablet capsule allows using an about 2 g tablet and obtaining a liquid part volume of about 12 ml in the first, and 17 ml in the second case. The total mass of the capsule with a cavity of 12 ml volume is 16 g, and of the capsule with 17 ml cavity is 21 g.

[0040] Both sachet types fit into the dishwasher detergent cup. The tablet position inside the capsule is suitable for its shape. The tablet placement in subsequent samples in relation to the liquid cavity is constant. The film is sealed. The mass in relation to the assumed capsule size in both cases is below 24 g. The capsule with a cavity of 17 ml volume looks 'bulgy', the capsule does not touch the table with the whole surface. Folds are forming around the tablet - further development on flattening them is required. The cavity having 12 ml volume for the liquid and 12 mm for the tablet has been assessed as too small to contain the proper biocomponent dose.

[0041] Liquid/tablet (16 mm) capsule: Increasing the tablet diameter allows obtaining a more stable tablet while cutting the heat-seal. A smaller cavity required for the tablet/liquid is less cone-like, which positively affects the stage of enclosing the tablet/liquid in a film. The amount of active substance that can be contained in a capsule of this form was increased to 3 g and corresponds to the recommended dose.

[0042] As a result of performed tests, a most preferred capsule form was determined: 16 mm (smaller cavity) and 16 ml (cavity with liquid). An exemplary capsule of this type has been presented in Fig. 2.

[0043] Figures 3-5 illustrate different views of the capsule according to the invention, having a coating made of PVOH

Example 3. Tested variants of the capsules

[0044] Generally, the capsule is filled with an aqueous solution containing:

nonionic surfactants, polycarboxylates, anionic surfactants, phosphonates, enzymes, a preservative, a fragrance composition and microorganisms (preferably about 1 wt%).

[0045] Microorganisms are contained in the tablet portion. The capsule is made of a PVOH self-dissolving film.

[0046] The method of administering the microorganisms in dry form ensures their stability in the system. Microorganisms contained in the tablet in form of spores are activated by contact with water.

[0047] The capsule manufacture process proceeds in two steps. The first step is preparing the tablet. This stage involves the process of ingredient mixing, and next pressing on the device to a tablet form. The pressing step is carried out under pressure, which ensures intactness of cell wall structures. The second step involves a process of forming the capsule, in which the dosing step of the liquid part and tablet incorporation is achieved at the same time. The process is carried out under strictly controlled conditions of about 23°C and 45% humidity. Performed tests of film stability confirm the stability of its structure, and therefore the stability of the capsule under these formulating conditions.

[0048] Depending on the liquid part composition, the capsule can be applied in appliances for dishwashing, laundry washing, and, after diluting, also for cleaning other hard surfaces.

[0049] Various formula compositions were tested:

A dishwasher capsule (comparative example)

nonionic surfactants in amount of 15-30%
polycarboxylates in amount of 5-15%
anionic surfactants in amount of <5%

phosphonates
enzymes
preservative
fragrance composition
microorganisms <1%

[0050] The tablet contained in the capsule displayed a change of parameters - bulging (about 1 mm height change, which consequently results in tearing of the film.

[0051] The liquid part penetrates into the tablet part, which consequently leads to exposure of the microorganisms on aggressive chemical substances and their elimination.

[0052] A dishwasher capsule with an addition of a peroxide substance (comparative example):

nonionic surfactants in amount of 15-30%
polycarboxylates in amount of 5-15%
anionic surfactants in amount of <5%
peroxide compounds <5%
phosphonates
enzymes
preservative
fragrance composition
microorganisms <1%

[0053] The peroxide substances are desired in the formula systems due to the fact that they enhance their effectiveness and efficiency. The tests employed sodium percabonate. However, peroxide substances are highly hygroscopic. One of the stages of forming the capsule involves binding (gluing) the upper and lower self-dissolving film by spraying it with water. The tablet with an addition of a peroxide compound displayed a strong bulging effect and was tearing the film. Product was not stable in time. The negative effect was observed already 2 weeks after the product manufacture.

[0054] Applying standard materials such as Marlox 154, Not Surf, Rokanol L7W, Rokanol IT in anionic systems, anionic compound systems with hydrotropes, resulted in lack of formula stability and their delamination.

[0055] The preferred embodiments of capsules according to the present invention are the following:

1. A capsule for automatic dishwashing. The form of the capsule itself is highly preferred. It allows application of precisely specified and validated amount of detergent, thus preventing an excess of chemical agents from being introduced to the environment. The capsule is characterised by simplicity and quickness of use. It is ready to use immediately after taking out of the package. The application is performed by placing the capsule in a dishwasher cup intended for this use and choosing an appropriate program. The capsule can be used for all washing programs. One capsule is sufficient and adequate for performing an effective washing process.
2. A capsule for automatic laundry washing promoting the maintenance of hygiene of the appliance. The microorganisms are introduced jointly with the washing agents. The capsule form is highly preferred. It allows application of precisely specified and validated amount of detergent, thus preventing an excess of chemical agents from being introduced to the environment. The capsule is characterised by simplicity and quickness of use. It is ready to use immediately after taking out of the package. The application is performed by placing the capsule in the washing machine drum and choosing an appropriate program. The capsule can be applied in all washing programs. One capsule is sufficient and adequate for performing an effective washing process of typically dirty laundry. In case of very dirty laundry, using two capsules is required.
3. A capsule for cleaning of hard surfaces. Application is performed by dissolving a single capsule in about 5 l of water, and subsequent washing of soiled surface. The process does not require rinsing of the washed surface.

[0056] Tests described in the following example employ the following washing agent formulas:

WASHING GEL

No.	Formula component	Content [%]	Effect on bacteria
1.	Alkyl alcohol ethoxylate (C10-C16)	15-30	Bacteriostatic / bactericidal
2.	Propylene glycol	15-30	Bacteriostatic / bactericidal

(continued)

No.	Formula component	Content [%]	Effect on bacteria
3.	Glycerine	15-30	Stabilizes the bacteria / utilized as carbon and energy source
4.	Dodecylbenzene sulfonic acid	15-30	Highly bactericidal
5.	Water	< 15	In low concentration (9%) increases the effectiveness of bactericidal components
6.	Coconut acid	< 5	Bacteriostatic / can be utilized as carbon and energy source
7.	Monoethanolamine	<5	Bacteriostatic / bactericidal
8.	Proprietary mixture of organophosphonic acid, sodium salts	< 1	Bactericidal
9.	Stain remover	1-5	Bacteriostatic / bactericidal
10.	Fragrance	< 1	Bacteriostatic
11.	Protease	< 1	Bacteriostatic / bactericidal
12.	Lipase	< 1	Bacteriostatic / bactericidal
13.	Cellulase	< 1	No effect / can be utilized as carbon and energy source
14.	EASY WET : Undeceth-5 Undecyl alcohol Sodium lauryl sulphate Caprylyl Pyrrolidone	< 1	Highly bactericidal
15.	Vinylpyrrolidone/ Vinylimidazole Copolymer	< 1	No effect
16.	Carboxymethyl inulin, sodium salt solution	< 1	Bacteriostatic / bactericidal
17.	Dye	<0.1	Bacteriostatic / bactericidal
18.	2-Bromo-2-Nitropropane-1,3-Diol Methylchloroisothiazolinone, Methylisothiazolinone	<0.5	Highly bactericidal

DISHWASHING GEL

No.	RAW MATERIAL	Content [%]	Effect on bacteria
1.	Glycerine	15-30	Stabilizes the bacteria / utilized as carbon and energy source
2.	Sorbitol	< 15	Stabilizes the bacteria / utilized as carbon and energy source
3.	Water	< 15	In low concentration (9%) increases the effectiveness of bactericidal components
4.	Propylene glycol	< 15	Bacteriostatic / no effect
5.	Sodium citrate	< 15	Can be utilized as carbon and energy source. However it also chelates essential divalent ions, thus inhibiting survival of bacteria.
6.	Polyacrylic acid, partially neutralized, sodium salt in water	15-30	Bacteriostatic
7.	Polyacrylic acid, neutralized, sodium salt in water	15-30	Bacteriostatic

(continued)

No.	RAW MATERIAL	Content [%]	Effect on bacteria
8.	Monoethanolamine	5-10	Bacteriostatic / bactericidal
9.	Alcohols, C12-C14 (even numbers), ethoxylated propoxylated (>2.5 moles EO/PO)	< 5	Bacteriostatic / can be utilized as carbon and energy source
10.	Mixture of organophosphonic acid, sodium salt in water	< 5	Bactericidal
11.	Protease	< 5	Bacteriostatic / bactericidal
12.	Amylase	< 5	No effect / can be utilized as carbon and energy source
13.	Polyacrylic acid (carbomer)	< 5	Bacteriostatic
14.	Disodium metasilicate	< 1	Bacteriostatic / no effect
15.	Fragrance	< 1	Bacteriostatic
16.	2-Bromo-2-nitropropane-1,3-diol 2-octyl-2H-isothiazol-3-one	< 0.5	Highly bactericidal
17.	Dye	< 0.1	Bacteriostatic / no effect

Example 4. Influence of physicochemical parameters, changing environment and washing agent formula components on biocomponent activity in changing environments

[0057] Tests of pH influence on microbial survival in the probiotic were performed. The maximum survival temperature of microbes isolated from the probiotic was determined - the maximum temperature growth for probiotics was 58°C, as well as the thermal death time of the probiotics in temperature of 100°C. Moreover, test of probiotics' survival in different washing agent formulas were performed. It has been determined that washing mixture components in high concentrations totally inhibit probiotic growth, displaying very high bactericidal properties towards them.

[0058] At the same time, unexpectedly, the tests demonstrated a possibility of survival, especially in spore form, of chosen *Bacillus sp.* bacteria constituting the probiotic during washing process using the washing agent diluted to the working concentration. For this reason, in the finished product, microbes should be contained in a separate cavity.

[0059] A summary of preformed tests is presented in the following table.

Table 1. A summary report of thermostability, pH influence and microbial survivability tests in concentrated washing agents (so called formulas) and washing baths in working dilutions.

No.	Tested strain	pH	Temperature	Survivability in the formula	Survivability in the washing bath
1	Commercial liquid probiotic Badlox^{R} XL100x Ospray Biotechnics Inc. (mixture of *Bacillus* sp.	Bacteria incubated in a growth medium, as well as in the tap water, are resistant to a wide	Bacteria are highly thermostable, exhibiting cell division up to the temperature of 58°C. In hibernation state	Death after mixing with tested formulas of washing and cleaning gels.	Probiotic bacteria used in the temperature range of dishwasher washing and in the chemical environment generated by the capsules survive a
	probiotic strains)	range of pH, at least 4-10.	they survive 100°C for over 18h (low percent of living cells).		multiple washing cycle.

(continued)

No.	Tested strain	pH	Temperature	Survivability in the formula	Survivability in the washing bath
2	Commercial probiotic in powder form BPB-100 Ospray Biotechnics Inc. (mixture of *Bacillus* sp. probiotic strains)	Bacteria incubated in a growth medium, as well as in the tap water, are resistant to a wide range of pH, at least 4-10.	Bacteria are highly thermostable, exhibiting cell division up to the temperature of 58°C. In hibernation state they survive 100°C for over 18h (low percent of living cells).	Death after mixing with tested formulas of washing and cleaning gels.	Probiotic bacteria used in temperature range of dishwasher washing and in the chemical environment generated by the capsules survive a multiple washing cycle.
3	*Bacillus coagulans* strain - live bacteria Polish Collection of Microorganisms, access number PCM 1843	The bacterial strain exhibits good survivability in pH 5, 7, and 8, in temperatures of 30, 40, and 50°C. In temperature of 60°C, a satisfactory survivability was recorded only in pH 8. Higher temperatures act bactericidally on tested bacteria.		Test were not performed - bacteria strains died out already in diluted solutions.	The washing gel exhibits a highly bactericidal effect on tested bacteria strains. No satisfactory survivability of bacterial cells was observed in any tested conditions (pH and temperature) in the presence of washing gel.
4	*Lactococcus lactis* strain- live bacteria Polish Collection of Microorganisms, access number PCM 476	The bacterial strain exhibits good survivability in pH 5, 7, and 8, in temperatures of 30, 40, and 50°C. In temperature of 60°C, a satisfactory survivability was recorded only in pH 8. Higher temperatures act bactericidally on tested bacteria.		Test were not performed - bacteria strains died out already in diluted solutions.	The washing gel exhibits a highly bactericidal effect on tested bacteria strains. No satisfactory survivability of bacterial cells was observed in any tested conditions (pH and temperature) in the presence of washing gel.
5	*Bacillus coagulans* - spores Polish Collection of Microorganisms, access number PCM 1843	*Bacillus coagulans* spores are characterised by a good survivability in tested washing and cleaning formulas, in tested pH and temperature range.		Not performed	Tested temperature did not exhibit an inhibiting effect on spore count, except for the temperature of 100°C. However, even after incubation in temperature of 100°C, bacterial spores capable of growing were found.

Commercial liquid or solid probiotic (points 1 and 2 in Table 1)

[0060] Stability tests of *Bacillus* sp. bacteria in various temperatures and various pH of the laundry washing and washing bath were performed (concerning pts. 1 and 2 of Table 1).

[0061] The tests involved the following steps:

- preliminary determination of the optimal temperature for liquid and solid probiotic samples,
- selection of type and nature of microbiological medium (composition and form) - the growth medium recommended by the manufacturer was chosen (LA) as typically averagely nutrient-rich medium with average sodium chloride concentration and neutral pH, on which typically *Bacillus* sp. genus bacteria exhibit good growth. Firstly, streaking was performed using an inoculation loop from a 10 microlitre droplet of thoroughly mixed probiotic in order to isolate the microbial species and strains present therein,

- temperature selection (scale, minimum, maximum, step) - a wide range of temperature was chosen, starting from room temperature, 30°C and 37°C for mesophiles, 55°C for thermophiles, and 65°C and 70°C for hyperthermophiles.

[0062] Observed results concerning the optimal temperature for liquid probiotic bacteria (1) are summarised in the following table.

Temperature	Growth
Room temperature	+
30°C	+++
37°C	+++
55°C	++
65°C	-
70°C	-

[0063] Observed results concerning the optimal temperature for solid probiotic bacteria (1) are summarised in the following table.

Temperature	Growth
Room temperature	+
30°C	+++
37°C	+++
55°C	++
65°C	-
70°C	-

[0064] A pH influence on survivability of microbes present in probiotics - solid and liquid, was tested. For this reason a series of liquid growth media was prepared, having an increasing pH: 5, 6, 7, 8, 9, 10. The probiotic sample was incubated in growth media prepared in this manner, in room temperature in an increasing pH environment, mixed in 1:100 ratio. Afterwards, 0.1 ml of such suspension was inoculated, in order to obtain a bacterial lawn, on a LA growth medium and incubated under optimal conditions of 37°C for 24 hours. Established incubation times in different pH: 1 hour, 24 h, 48 h, 2 × 48 h (4 days) and 7 days.

[0065] Results: After 4 day incubation in 5-10 pH range, an intense growth was observed for each of tested pH values for both probiotic samples (liquid 2 and solid) - no change in relation to 1 hour incubation - tested microbes tolerate a wide range of growth medium pH: 5, 6, 7, 8, 9, 10, even despite the 100-fold dilution applied.

[0066] Afterwards, a survivability test of the probiotic microbes in concentrated formulas (washing liquid, laundry washing liquid) was performed.

[0067] In order to carry out the survivability test, 1:10 was arbitrarily chosen as maximal possible probiotic dilution in the tested medium, which equals a 10% content of the microbial medium in the tested formula (10+90). Next, two approaches were applied in order to verify the survivability of the microbes in this environment in comparison with the double control: a 1:10 dilution of probiotic in the growth medium and an inoculation immediately after mixing with the tested medium (washing liquid or laundry washing liquid). The inoculations were performed at time: 0, 1 h, 2 h, 3 h, 6 h, 20 h, accordingly. Two analytical approaches were applied:

A mixture of the probiotic and the tested medium was incubated at time: 0 (inoculated immediately after mixing), 1 h, 2 h, 3 h, 6 h, 20 h. Each time after the determined incubation time in the tested medium elapsed, plate inoculations on optimal growth media were performed in order to obtain the bacterial lawn (0.1 ml) and incubated at optimal temperature.

[0068] Test results of the influence of washing liquid and laundry washing liquid on microbes used in the probiotics are presented below:

Incubation time in the tested medium [room temp.]	Growth intensity on a solid medium - P2 AFTER INCUBATION IN THE WASHING LIQUID	Growth intensity on a solid medium - P2 AFTER INCUBATION IN THE LAUNDRY WASHING LIQUID
0	no growth	no growth
1 h	no growth	no growth
2 h	no growth	no growth
3 h	no growth	no growth
6 h	no growth	no growth
20 h	no growth	no growth

[0069] The obtained results were verified in a subsequent experiment in liquid growth media. The tested samples were inoculated in liquid growth media by means of an inoculation loop, thus transporting about 10 microlitres of the tested sample into 5 ml of the growth medium (1:500 dilution). This approach would enable catching even individual microbial cells surviving in the laundry washing/washing liquid formula or their spores, and effective growth under optimal conditions, which would by demonstrated by turbidity of the liquid growth medium.

[0070] The observed results are presented in the following table:

Incubation time in the tested medium [room temp.]	Growth intensity in a liquid medium - P2 AFTER INCUBATION IN THE WASHING LIQUID	Growth intensity in a liquid medium - P2 AFTER INCUBATION IN THE LAUNDRY WASHING LIQUID
0	no growth	no growth
1 h	no growth	no growth
2 h	no growth	no growth
3 h	no growth	no growth
6 h	no growth	no growth
20 h	no growth	no growth

[0071] The culture samples in the liquid growth medium were also analysed by a spectrophotometric method (absorbance change for 600 nm light wavelength). Obtained results confirmed lack of bacterial growth.

[0072] CONCLUSIONS: The concentrated laundry washing liquid and washing liquid fully eliminate the microbial growth, displaying very high bactericidal properties.

[0073] Probiotic survivability (pos. 1 and 2) in dilutions used in the washing process.

[0074] In order to perform the survivability test, 3 g weighted amounts of the powder probiotic (S) were prepared. For effective suspension in the tested medium, the suspension in tap water was prepared in 1:5 ratio. Whole suspensions were mixed into the previously prepared working dilutions according to the following scheme:

1) 13 ml of washing liquid for 2.5 l

2) 13 ml of washing liquid for 5.0 l

3) 24 ml of washing liquid for 2.5 l

4) 24 ml of laundry washing liquid for 5 l

5) 2.5 l of the growth medium - control

6) 5.0. l of the growth medium - control

[0075] Next, three experimental approaches were applied, in order to test the microbial survivability in this environment comparing to a double control of probiotic suspension in a growth medium, and the inoculation immediately after mixing

with the tested medium (washing liquid or laundry washing liquid). The inoculations were performed at times: 0, 1 h, 2 h, 3 h, 6 h, 24 h, accordingly.

[0076] After mixing the probiotic with the tested working dilution, the mixture was incubated at time: 0 (inoculation immediately after mixing), 1 h, 2 h, 3 h, 6 h, 24 h. Each time after the incubation time in the tested medium has elapsed, microbiological inoculations on a plate (0.1 ml), on optimal growth media were performed, and incubated at optimal temperature.

[0077] Results of observations concerning the influence of washing liquid and laundry washing liquid in a working dilution of 13 ml (washing) / 24 ml (laundry washing) for 2.5 or 5.0 l of tap water, determining the microbiological stability (survivability) of the probiotic compared to the control in the growth medium. The observations are summarised in the following table.

Incubation time in the tested medium [room temp.]	Growth intensity in the liquid growth medium - 3 g of the solid probiotic after mixing with 13 ml of the washing liquid /2.5 l of water	Growth intensity in the liquid growth medium - 3 g of the solid probiotic after mixing with 13 ml of the washing liquid /5 l of water	Growth intensity in the liquid growth medium - 3 g of the solid probiotic after mixing with 24 ml of the washing liquid /2.5 l of water	Growth intensity in the liquid growth medium - 3 g of the solid probiotic after mixing with 24 ml of the washing liquid /5 l of water
0	+++	+++	+++	+++
1 h	+++	+++	+++	+++
2 h	+++	+++	+++	+++
3 h	+++	+++	+++	+++
6 h	+++	+++	+++	+++
24 h	+++	+++	+++	+++

Conclusions:

[0078] All tested dilutions of the washing liquid and the laundry washing liquid allow for survival of *Bacillus* sp. bacteria of the probiotic. Thus, it is possible to apply a combination of washing/laundry washing liquid and the probiotics, as long as they are physically separated in the applied package and their release will occur with simultaneous diluting in water.

Test concerning the chosen probiotic strains (see points 3, 4 and 5 in Table 1)

Tests of bacterial stability in various temperatures and various pH of the laundry washing and washing bath (concerning pts. 3 and 4 of Table 1)

[0079] The tests employed cell suspension of chosen bacteria having a cell count of an order of 10^6 cells/ml. An appropriate volume of the suspension was taken and transported to a laundry washing bath with a given pH, prepared beforehand. The final cell count in the bath was 10^4 cells/ml. Afterwards, the bath along with the cells were placed in a defined temperature for 30 minutes. After this time, 1 ml of the suspension was taken and a pour plate prepared, using an earlier developed, best growing mixture as a growth medium.

Table 2. *Lactococcus lactis* bacteria cell count after 30 minutes incubation in the laundry washing bath under the tested conditions.

pH	Control, without the laundry washing gel			Tested sample, with the laundry washing gel		
	Temperature [°C]			Temperature [°C]		
	30	40	60	30	40	60
5	9.7×10^4	6.8×10^4	0.5	0.0	0.0	0.0
7	6.8×10^4	1.7×10^4	0.0	0.0	0.0	0.0
8	9.6×10^4	6.6×10^4	1.7×10^4	0.0	0.0	0.0

Table 3. *Bacillus coagulans* bacteria cell count after 30 minutes incubation in the laundry washing bath under the tested conditions.

pH	Control, without the laundry washing gel			Tested sample, with the laundry washing gel		
	Temperature [°C]			Temperature [°C]		
	30	40	60	30	40	60
5	7.0×10^4	5.0×10^4	0,0	0.0	0.0	0.0
7	11.5×10^4	5.7×10^4	4,5	0.0	0.5	0.0
8	8.0×10^4	6.2×10^4	31	2.0	0.5	0.0

Table 4. *Lactococcus lactis* bacteria cell count after 30 minutes incubation in the washing bath under the tested conditions.

pH	Control, without the washing gel				Tested sample, with the washing gel			
	Temperature [°C]				Temperature [°C]			
	50	60	70	100	50	60	70	100
5	11	0.0	0.0	0.0	14.5	0.0	0.0	0.0
7	3	0.0	0.0	0.0	3	0.0	0.0	0.0
8	7.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Table 5. *Bacillus coagulans* bacteria cell count after 30 minutes incubation in the washing bath under the tested conditions.

pH	Control, without the washing gel				Tested sample, with the washing gel			
	Temperature [°C]				Temperature [°C]			
	50	60	70	100	50	60	70	100
5	9.0×10^2	1.0	0.0	0.0	0.0	0.0	0.0	0.0
7	8.5×10^2	1.0	0.5	0.0	0.0	0.0	0.0	0.0
8	8.0×10^2	0.5	0.0	0.0	0.0	0.0	0.0	0.0

Tests of bacterial stability in various temperatures and various pH of the laundry washing and washing bath (concerning pt. 5 of Table 1).

[0080]

Table 6. *Bacillus coagulans* bacteria spore cell count [spores/ml] after 30 minutes incubation in the laundry washing bath under the tested conditions.

pH	Control, without the laundry washing gel			Tested sample, with the laundry washing gel		
	Temperature [°C]			Temperature [°C]		
	30	40	60	30	40	60
5	5.5×10^5	5.4×10^5	7.1×10^5	6.9×10^5	9.8×10^5	10.6×10^5
7	3.8×10^5	5.8×10^5	7.0×10^5	9.1×10^5	10.3×10^5	10.4×10^5
8	6.0×10^5	6.8×10^5	6.8×10^5	8.2×10^5	10.2×10^5	9.4×10^5

Table 7. *Bacillus coagulans* bacteria spore cell count [spores/ml] after 30 minutes incubation in the washing bath under the tested conditions.

pH	Control, without the washing gel				Tested sample, with the washing gel			
	Temperature [°C]				Temperature [°C]			
	50	60	70	100	50	60	70	100
5	10.1×10^5	9.3×10^5	8.3×10^5	3.0×10^0	10.3×10^5	10.8×10^5	10.0×10^5	3.0×10^0
7	9.0×10^5	8.8×10^5	8.5×10^5	6.5×10^0	9.2×10^5	10.2×10^5	11.3×10^5	1.5×10^2
8	8.7×10^5	7.9×10^5	8.3×10^5	6.7×10^0	10.9×10^5	8.3×10^5	10.0×10^5	9.2×10^0

[0081] The tests involved:

- proliferation of *Bacillus coagulans* bacteria,
- formation of spores of these bacteria,
- separation of spores from the vegetative cells,
- freezing and lyophilisation of these spores,
- stability tests of obtained spores in various pH and temperatures, in the washing and laundry washing formulas (analogically to the laundry washing process: 30, 40, 60 °C; analogically to the washing process in the dishwasher: 50-60-70°C, drying at about 100°C).

[0082] The tests were performed under the so called dirty conditions, that is with an addition of 3 g of lecithin for 1 litre of the mixture (the added lecithin simulates the organic matter load, similarly to the organic contaminations).

[0083] The originally prepared suspension contained 1.5×10^7 of bacterial spores. Subsamples were taken from this suspension, and introduced to the reaction mixtures (washing and laundry washing formulas with various pH values).

[0084] The final spore count in the laundry washing formula was of order 10^5, whereas in the washing formula it was 10^6.

Conclusions:

[0085]

1. The chosen bacterial strains can be easily grown in laboratory and semi-industrial conditions.
2. The tested bacterial strains display good survivability in pH 5, 7 and 8 in temperatures of 30, 40 and 50°C. In temperature of 60°C, a satisfying survivability was recorded only for pH 8. Higher temperatures are highly bactericidal for tested bacteria.
3. The laundry washing gel displays highly bactericidal influence on the tested bacterial strains. Good survivability of the bacterial cell was not recorded in any of the testing conditions (pH and temperature), in the presence of the laundry washing gel.
4. The washing gel also displays highly bactericidal influence on the tested bacterial strains. Good survivability of the bacterial cell was not recorded in any of the testing conditions (pH and temperature), in the presence of the washing gel.
5. Solutions of washing and laundry washing gels display high bactericidal influence on the microbes in the vegetative form.
6. Solutions of washing and laundry washing gels do not display a negative influence on the microbes in spore form.
7. The composition may employ *Bacillus sp.* probiotic strains of Risk Group 1, forming spores, displaying growth in temperature up to of 65°C.

Example 5. Washing tests

[0086] For the chosen preferred variants of embodiments of the capsule according to the invention, washing tests were performed.

[0087] An experimental dirty surface was washed with the water solution obtained by dissolving the capsule according to the invention in a 2 to 10 litres volume of water, wherein after finishing the washing process, the cleaned surface was left to dry. The tests were performed in commercially available dishwasher machines.

[0088] Obtained results are summarised in the following table.

Table 8. Washing tests results

Formulation no.	Probiotic administration	Classical washing efficiency results	Survivability of microorganisms after the washing process
20/2014	Probiotic added in powder form to the liquid form	milk - 52.77% yolk - 80.00% plates/cereal - 72.87%	According to the survivability tests, the formulation components acted bactericidally on BPB-100 OSPREY microorganisms
8/2015	Probiotic in a tablet	Visual evaluation: Surface without streaks, dirt, after washing with gloss	Applying the washing gel with addition of probiotic to washing the tested surfaces lowered the survivability of potentially pathogenic G+ bacteria. Decrease of the cell count of these bacteria was of one order of magnitude.
13/2015	Probiotic in a tablet	milk - 93.07% yolk - 52.51% plates/cereal - 87.51%	The probiotics inhabit sides and hard to read areas of the appliances. Tested dilutions of the laundry washing liquid and the washing liquid enable bacterial survivability.
36/2015	Probiotic in a tablet with an addition of percarbonate	milk - 89.44% yolk - 77.8% plates/cereal - 72.5%	The active oxygen displays highly bactericidal influence on microorganisms.
46/2015	Probiotic in a tablet	milk - 95.00% yolk - 78.86% plates/cereal - 92.49%	The probiotics inhabit sides and hard to read areas of the appliances. Tested dilutions of the laundry washing liquid and the washing liquid enable bacterial survivability.
167/2016	Probiotic in a tablet	milk - 93.02% yolk - 79.14% plates/cereal - 92.49%	The probiotics inhabit sides and hard to read areas of the appliances. Tested dilutions of the laundry washing liquid and the washing liquid enable bacterial survivability.

[0089] In case of dishwasher products, the washing efficiency was determined by means of an IKW test. The following criteria of washing efficiency evaluation were assumed: milk about 90%, yolk about 75%, plates (starch dirt) - about 90%. Survivability of microorganisms after the washing process, at the level not lower than 80% of the control amount.

Formulation 20/2014

No.	LIQUID	[%]
1	Propylene glycol	**6.98**
2	Sorbitol	**9.60**
3	Glycerine	**32.00**
4	Carbopol	**0.53**
5	Distilled water	**8.21**
6	Sodium citrate	**4.31**
7	Sokalan PA25	**10.27**
8	Sokalan PA30	**10.27**
9	Disodium metasilicate	**0.16**
10	Monoethanolamine	**3.69**

(continued)

No.	LIQUID	[%]
11	Cublen A4015	**1.03**
12	Everlase	**1.31**
13	Termamyl ultra	**1.07**
14	NatSurf	**10.27**
15	Clean Citrus fragrance composition	**0.01**
16	Parmetol N20	**0.04**
17	Dysol Light Blue (0.5%) dye	**0.26**
18	Badlox® XL100x probiotic	**1.00**

Formulation 36/2015

No.	LIQUID	[%]
1	Propylene glycol	**5.00**
2	Sorbitol	**4.75**
3	Glycerine	**16.90**
4	Carbopol	**0.65**
5	Distilled water	**3.60**
6	Sodium citrate	**6.00**
7	Dissolvine GL47S	**19.00**
8	Sokalan PA25	**16.00**
9	Sokalan PA30	**16.00**
10	Disodium metasilicate	**0.30**
11	Monoethanolamine	**5.80**
12	Cublen A4015	**1.50**
13	Marlox	**4.00**
14	Clean Citrus fragrance composition	**0.01**
15	Acticide MBS	**0.09**
16	Dysol Green (0.5%) dye	**0.40**
No.	**TABLET**	**[%]**
1	Sodium percarbonate	**50.00**
2	Heavy sodium carbonate	**15.36**
3	Sodium bicarbonate	**7.00**
4	Citric acid	**7.14**
5	Heweten 101	**4.00**
6	Zinc stearate	**0.5**
7	Cellulose	**4.00**
8	Detergent	**3.00**
9	Polyethylene glycol	**2.00**

(continued)

No.	TABLET	[%]
10	TAED	**2.00**
11	Heweten 101	**4.00**
12	BPB-100 probiotic	**1.00**

Formulation 13/2015

No.	LIQUID	[%]
1	Propylene glycol	**6.40**
2	Sorbitol	**9.20**
3	Glycerine	**23.00**
4	Carbopol	**0.80**
5	Distilled water	**8.00**
6	Sodium citrate	**7.00**
7	Sokalan PA25	**15.00**
8	Sokalan PA30	**15.00**
9	Disodium metasilicate	**0.30**
10	Monoethanolamine	**5.40**
11	Cublen A4015	**1.50**
12	Excellenz P100	**2.20**
13	Effectenz S100	**1.70**
14	Marlox	**4.00**
15	Fresh Odor fragrance composition	**0.05**
16	Parmetol N20	**0.05**
17	Dysol Light Blue dye	**0.40**

No.	TABLET	[%]
1	Heavy sodium carbonate	**35.93**
2	Anhydrous sodium sulphate	**21.43**
3	Marvellenz1000/T-blend	**8.00**
4	PROBIOTIC	**7.00**
5	Sodium bicarbonate	**7.00**
6	Citric acid	**7.14**
7	Cellulose	**4.00**
8	Detergent	**3.00**
9	Polyethylene glycol	**2.00**
10	Cellulose	**4.00**
11	Zinc stearate	**0.50**

Formulation 46/2015

No.	LIQUID	[%]
1	Propylene glycol	**6.00**
2	Glycerine	**10.00**
3	Syntapon OD	**2.00**
4	Sokalan PA25	**22.50**
5	Dissolvine GL47S	**28.00**
6	Monoethanolamine	**5.70**
7	Cublen A4015	**3.00**
8	Hostacor IT	**2.20**
9	Caflon APG810	**9.30**
10	AG6202	**9.30**
11	Carboxyline 25 D Powder	**1.50**
12	Acticide MBS	**0.0944**
13	Citrus Clean 3779 fragrance composition	**0.10**
14	Tap water	**0.30**
15	Liquitint Bright Yellow	**0.0050**
16	Puricolor FBL 5	**0.0006**

No.	TABLET	[%]
1	Magnesium stearate	**0.50**
2	Sodium sulphate	**32.50**
3	Sodium citrate	**35.00**
4	Sodium bicarbonate	**5.00**
5	Citric acid	**5.00**
6	Arbocel TF 0210	**4.00**
7	HEWETEN 101	**4.00**
8	Pluriol E 4000 Powder	**2.00**
	Polyglycol 4000 PS	
9	MARVELLENZ 1000	**8.00**
	T-Blend Evity Blaze/Stainzyme Plus 70/3.6	
10	Purafac LF300	**3.00**
11	*Bacillus coagulans* probiotic	**1.00**

Formulation 167/2016

No.	LIQUID	[%]
1	Glutamic acid, N,N-diacetic acid tetrasodium salt	**74.00**
2	Alkyl glucoside	**8.50**

(continued)

No.	LIQUID	[%]
3	Polyacrylic acid, partially neutralized, sodium salt in water	**6.30**
4	Propylene Glycol	**4.30**
5	Glycerine	**3.20**
6	Mixture of organophosphonic acid, sodium salt in water	**2.30**
7	Water	**0.61**
8	Acylamide carboxylic acid, salt	**0.40**
9	Carboxymethyl inulin	**0.25**
10	Preservative:	**0.09**
	1,2-benzisothiazol-3(2H)-one	
	2-methyl-2H-isothiazol-3-one	
11	Fragrance	**0.05**
12	Dye Blue	**0.00035**
13	Dye Yellow	**0.005**

No.	TABLET	[%]
1	Magnesium stearate	**0.50**
2	Sodium sulphate	**32.50**
3	Sodium citrate	**35.00**
4	Sodium bicarbonate	**5.00**
5	Citric acid	**5.00**
6	Arbocel TF 0210	**4.00**
7	HEWETEN 101	**4.00**
8	Pluriol E 4000 Powder	**2.00**
	Polyglycol 4000 PS	
9	MARVELLENZ 1000	**8.00**
	T-Blend Evity Blaze/Stainzyme Plus 70/3.6	
10	Purafac LF300	**3.00**
11	*Bacillus coagulans* probiotic	**1.00**

Formulation 167/2016

No.	LIQUID	[%]
1.0	Glycerine	**13.00**
1.0	Propylene glycol	**10.00**
1.1	Distilled water	**6400**
2.0	ABS acid	**20.00**
3.0	Monoethanolamine	**6.8**
4.0	Rokanol L7A	**20**

(continued)

No.	LIQUID	[%]
6.0	Isopropyl alcohol	**2**
7.0	Sensi Rinse 745301 Red Liquid (2,5% r-r) dye	**0.50**
8.0	Fragrance composition	**1.00**
9.0	Parmetol A28	**0.05**

No.	TABLET	[%]
1	Magnesium stearate	**0,50**
2	Sodium sulphate	**32.50**
3	Sodium citrate	**35.00**
4	Sodium bicarbonate	**5.00**
5	Citric acid	**5.00**
6	Arbocel TF 0210	**4.00**
7	HEWETEN 101	**4.00**
8	Pluriol E 4000 Powder	**2.00**
	Polyglycol 4000 PS	
9	MARVELLENZ 1000	**8.00**
	T-Blend Evity Blaze/Stainzyme Plus 70/3.6	
10	Purafac LF300	**3.00**
11	*Bacillus coagulans* probiotic	**1.00**

Example 6. Stability of the liquid part of the formula and its compatibility with the film

[0090] According to the invention, the capsule is filled with a mixture containing the following: nonionic surfactants, polycarboxylates, anionic surfactants, complexing compounds, plasticisers, phosphonates, enzymes, a preservative, a fragrance composition, functional additives, water, and microorganisms (preferably about 1 wt%). Each group of components listed above has a certain function on each step of washing process, i.e. wetting the surface, which allows penetrating the dirt particles and detaching them from the surface, water softening and removal of metal ions, acting as dispergators of dirt surrounding the individual dirt particles and preventing their agglomeration, and preventing the redeposition of dirt, cutting the dirt chains on smaller fragments, or additives protecting the cleaned surfaces.

[0091] The nonionic surfactants chosen in the first step of the formula development have not led to obtaining stable systems, they had low effectiveness in the washing process. Attempts to additionally introduce anionic surfactants into the formula also did not yield good results. The obtained formulation were characterised by excessively high foam, which is not recommended for automatic washing systems.

[0092] A compound for wetting the surface was sought that would maintain compatibility with the other elements of the formula.

[0093] A difficulty in developing a stable formulation of the liquid part of the capsule was posed by its compatibility with the self-dissolving PVOH (polyvinyl alcohol) film, having a natural, desired capability of dissolving in water. A system having the water content in the formulation that would allow maintaining its proper structure at the product stage and simplicity of dissolving in the washing baths, was searched for. The research led to reduction of the water content introduced directly to the formula from 7% to below 4%. Simultaneously, as part of the performed research, a method of reducing the water content in the formula in a form contained in the materials was investigated, by means of replacing them with materials having a higher concentration or introducing thickeners and complexing compounds that bond water.

[0094] As part of the development; the next group of compounds found, having influence on the film structure, were plasticisers. Their amount in the final formulas, in comparison with the starting formulas, was lowered by about 50% and was on a level <20%. The starting, high content of plasticisers originated from their additional function as a solvent,

which allowed introducing of thickeners into the formulas. Analysing the water content and the plasticisers' share in the formulation was an essential element for capsules functionality of keeping and maintaining its shape and proper properties during the whole shelf life. Decision not to use the glucose-fructose syrup, as well as reduction of glycerine and propylene glycol content in the formula, resulted in an improvement of elasticity and reduction of plasticity of the capsule surface. The choice of fundamental and auxiliary materials is based, on one hand, on achieving the desired working properties, and on the other, minimising the amount of bound water introduced into the formulation, allowed reducing the occurence of substance migration through the PVOH film, and thus achieving the effect of dry capsule surface. This was achieved also by applying more concentrated material replacements: replacing the MGDA compound with the GLDA material - having a higher concentration and fulfilling a similar function in the formula, reducing the amount of water introduced directly to the basis. Attempts to apply a thickener that binds water contained in the formulation were not fully successful, due to the fact that although a stable basis was obtained, the effect of dry capsule surface was not achieved. Finally, the decision to use a thickener was revoked. As a result of performed experiments, the following composition of the optimal formula of the cleaning agent was determined:

	Washing systems	Laundry
Nonionic surfactants	6.2-10 %	20-30%
Complexing materials, aiding the washing process and providing gloss	50-75%	3%
Plasticisers	<20%	<20%
Polycarboxylates	<7%	<7%
Anionic surfactants	-	35-30%
Phosphonates	<3%	<3%
Preservative	<1%	<1%
Fragrance composition	<1%	<1%
Dyes	<1%	<1%
Water	<4%	<4%
Functional additives	<1%	<1%
Enzymes	-	<1%**

Tested formulation systems, content percentage

Formula no.	6	55	43	46	51	77	128	146	160
Nonionic surfactants	21.6	27	3	4	5	0	0	0	15.2
Complexing materials, aiding the washing process and providing gloss	0.15	0.15	11	12.5	18.3	34.1	30	35.67	28
Plasticisers	27.3	16	38.9	42.5	35.5	27.75	23	16	16
Polycarboxylates	0.2	1.3	28	26	28	27	32	32.33	22
Anionic surfactants	17.8	27					8	10.63	7.49
Phosphonates	0.7	0.7	2	2.15	2.2	2.15	3	3	3
Preservative	1.2	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09
Fragrance composition	0.05	1		0.01	0.01	0.01	0.01	0.01	0.01
Dyes	0.28	0.01	0.4	0.46	0.4	0.5	0.51	0.38	0.32
Water	4.92	3.7	7	6	6.6	3	3	1.5	0
Functional additives	7.2	10.25	6.5	3.2	3.9	5.4	0.4	0.4	7.9
Enzymes	0.8	0.8	3.21	3.21					

(continued)

Formula no.	6	55	43	46	51	77	128	146	160
Soaps**	17.8	12							
**concerns the systems for laundry washing									

Claims

1. A self-dissolving capsule for preparation of washing solutions, **characterised in that** it contains a washing agents and probiotic microorganisms, wherein the capsule consists of two separate, sealed cavities, the first of which contains the washing agent, whereas the second one contains a lyophilised or concentrated liquid formulation of *Bacillus* species probiotic bacteria, forming spores and displaying growth in temperature up to 65°C, wherein the washing agent is a composition containing the following components: a nonionic surfactant, a polycarboxylate, an anionic surfactant, a complexing agent aiding the washing process and providing gloss, plasticisers, a phosphonate, hydrolytic enzymes, a preservative, a fragrance, functional additives, and water.

2. Capsule according to claim 1, **characterised in that** it contains probiotic bacteria chosen from the following: *Bacillus subtilis, Bacillus pumilus, Bacillus vallismortis, Bacillus coagulans, Bacillus lactis,* and *Bacillus mojavensis,* preferably spores of the *Bacillus coagulans* PCM 1843 strain.

3. Capsule according to claim 1, **characterised in that** the washing agent contains the following: a nonionic surfactant in amount of 5 to 30 wt%, a polycarboxylate in amount below 7 wt%, an anionic surfactant in amount of 0 to 35 wt%, a complexing agent in amount of 50 to 75 wt%, a plasticiser in amount below 20 wt%, a phosphonate in amount below 3 wt%, a hydrolytic enzyme in amount below 1 wt%, a preservative in amount below 1 wt%, a fragrance in amount below 1 wt%, a functional additive in amount below 1 wt%, and water in amount below 4 wt%.

4. Capsule according to claim 1, **characterised in that** the nonionic surfactant is chosen from the group containing the following: C12-C14 ethoxylated alcohols, C8 alkyl glucoside, capryl decyl glucoside, alkyldimethylamine oxide solution, sorbitan sesquioctanoate, alkoxylated fatty alcohol, 2-propylheptanol ethoxilate, 2-ethylhexyl glucoside, alcohol alkoxylate, C8/10 methyl ester, preferably chosen from the group containing: C12-C14 ethoxylated alcohols and C8 alkyl glucoside.

5. Capsule according to claim 1, **characterised in that** the anionic surfactant is chosen from the group containing the following: sodium p-cumenesulphonate; potassium 4-isopropylbenzenesulphonate; sodium 2-ethylhexyliminodipropionate; sodium cocopropylenediamine propionate; decyl phosphoric acid; potassium salt, sodium alkyl sulphate; sodium 2-ethylhexyl sulphate; ABS acid MEA salt; ABS acid TEA salt; C12-C14 ethoxylated alcohols (1-2.5 EO), sulphates, sodium salts; MEA cocoate; TEA cocoate, preferably chosen from the group consisting of: ABS acid MEA salt; ABS acid TEA salt; C12-C14 ethoxylated alcohols (1-2.5 EO), sulphates, sodium salts; MEA cocoate; TEA cocoate.

6. Capsule according to claim 1, **characterised in that** the complexing agent is chosen from the group containing the following: sodium carboxymethyl inulin; tetrasodium EDTA; N,N-bis(carboxymethyl)alanine trisodium salt; glutamic acid, N,N-diacetic acid tetrasodium salt; sodium citrate, preferably chosen from the group containing: sodium carboxymethyl inulin; glutamic acid, N,N-diacetic acid tetrasodium salt.

7. Capsule according to claim 1, **characterised in that** the polycarboxylate is chosen from the group containing the following: aqueous solution of partially neutralised polycarboxylic acid (sodium salt); aqueous solution of a polyacrylic acid sodium salt; preferably is an aqueous solution of partially neutralised polycarboxylic acid (sodium salt).

8. Capsule according to claim 1, **characterised in that** the functional additive is chosen from the group containing the following: vinylpyrrolidone/vinylimidazole copolymer; sodium metasilicate; acylamide carboxylic acid, alkanolamine salt; PEG-8 GMIS; Fluorescent Brightener 28; Undeceth-5, undecyl alcohol, sodium lauryl sulphate, caprylyl pyrrolidone.

9. Capsule according to claim 1, **characterised in that** the plasticiser is chosen from the group containing the following: glycerine, propylene glycol, and sorbitol.

10. Capsule according to claim 1, **characterised in that** the phosphonate is chosen from the group containing the following: proprietary mixture of organophosphonic acid, sodium salts; aqueous solution of aminophosphonic acid salt.

11. Capsule according to claim 1, **characterised in that** the preservative is chosen from the group containing the following: 2-bromo-2-nitropropane-1,3-diol, methylchloroisothiazolinone, methylisothiazolinone; methylisothiazolinone, benzisothiazolinone.

12. Capsule according to claim 1, **characterised in that** the hydrolytic enzyme is chosen from the group containing the following: protease, lipase, amylase, and cellulase.

13. A method for cleaning soiled surfaces, **characterised in that** the soiled surface is washed with an aqueous solution obtained by dissolving the self-dissolving capsule defined in claims 1-12 in 2 to 10 litres volume of water, wherein, after finishing the washing process, the cleaned surface is left to dry.

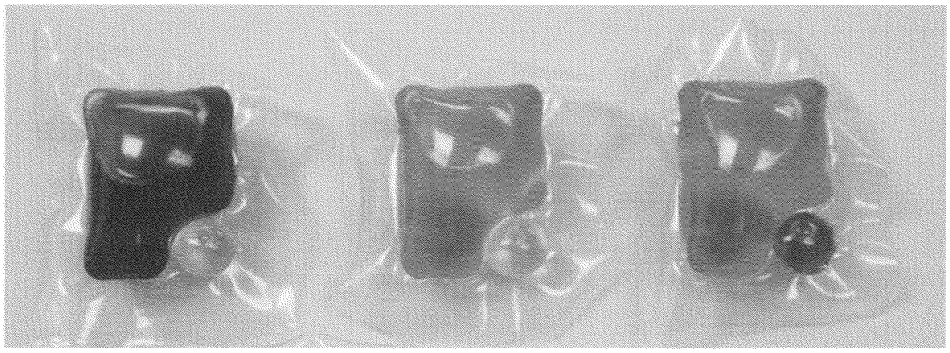

Fig. 1

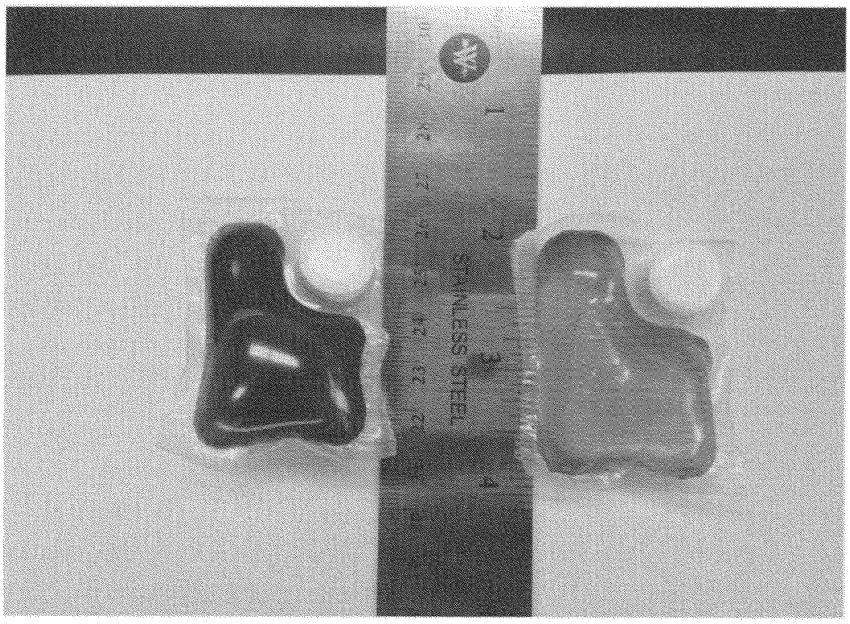

Fig. 2

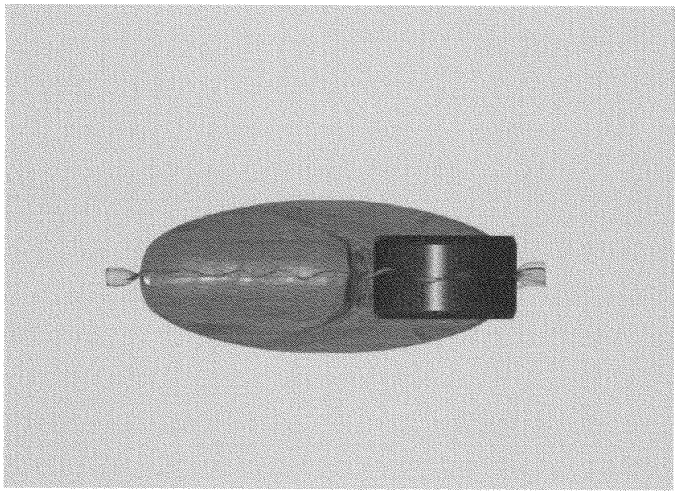

Fig. 3

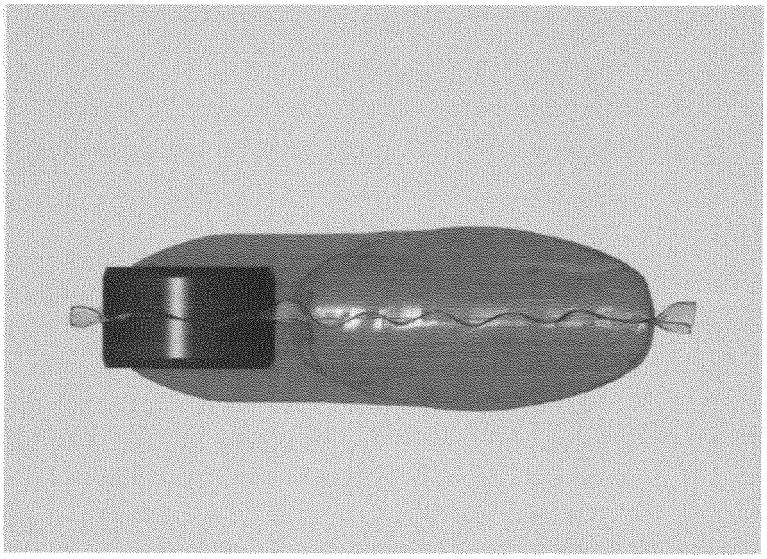

Fig. 4

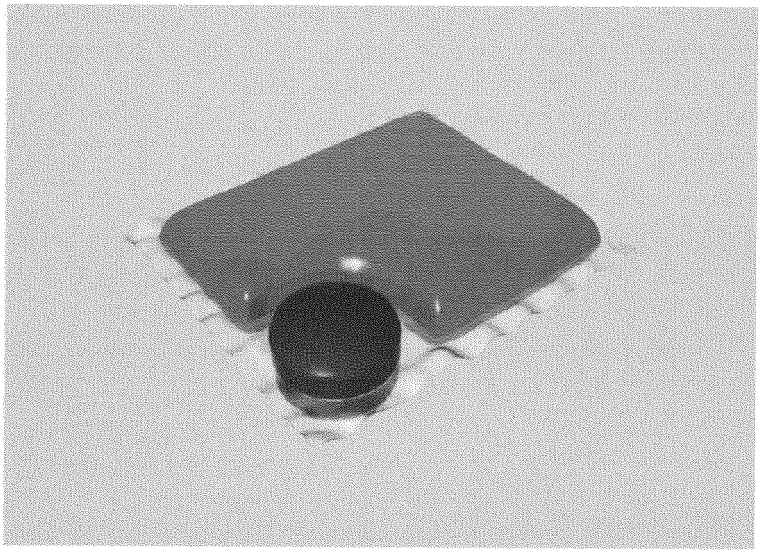

Fig. 5

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EUROPEAN SEARCH REPORT

Application Number
EP 16 20 7662

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	12 April 2017	Agra-Gutierrez, C

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