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(54) Title: GRANULATED FRAGRANCED FOAM CONTROL AGENTS

(57) Abstract: A granular fragranced antifoam composition comprising an encapsulated combination of a silicone foam control agent and a fragrance in a protective encapsulating material.



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Granulated Fragranced Foam Control Agents

Brief Description of the Invention

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The present invention relates to fragranced foam control agents and detergent compositions comprising these fragranced foam control agents.

Background Art & Technical Field

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Detergent compositions in powder form are used for washing purposes in machines for washing dishes or for laundering of textiles. These compositions generally contain organic surfactants, builders, for example phosphates, bleaching agents and various organic and inorganic additives. The surfactants usually employed in domestic
15 textile washing powders, when agitated in an aqueous medium during a washing cycle, tend to yield copious quantities of foam. However, presence of excessive amounts of foam during a washing cycle in certain washing machines tends to adversely affect the quality of the washing process.

It has become a practice to include in detergent compositions materials which
20 are intended to control the amount of foam produced during a washing cycle. Various materials have been proposed for this purpose, including certain silicone foam control agents. Silicone foam control agents, especially those based on polydimethylsiloxanes, have been found to be particularly useful foam control agents in a variety of media. The major advantage of silicone antifoam over soaps is their excellent performance at low
25 washing temperatures and their independence on water hardness. Soaps in fact only perform in hard water where they form water-insoluble calcium salts that act as antifoams.

However, generally silicone foam control agents, when incorporated in detergent compositions in powder form, appear to lose their effectiveness after prolonged
30 storage in the detergent compositions. Furthermore, such silicone antifoams are highly viscous compounds, which need to be mixed with organic waxes or solvents prior to encapsulation, in order to obtain liquid blends that can be processed.

It is well described in the public literature that silicone antifoams loose their activity in powder detergents due to the fact that when sprayed on the detergent, they migrate into the powder where they reduce their particle droplet size to an extent that the silicone oil becomes over-emulsified and is not efficient anymore to break foam. Figure 1
5 illustrates the effect of migration of silicone antifoam onto granular powder resulting in reduction of the droplet size of the silicone antifoam and over-emulsification of the antifoam compound.

European patent specification EP 210721 is directed to a silicone foam control agent which is stable on storage. It provides a particulate foam control agent in finely
10 divided form, for inclusion in a powder detergent composition, the agent comprising one part by weight of silicone antifoam and not less than one part by weight of an organic material characterized in that the organic material is a fatty acid or a fatty alcohol having a carbon chain containing from 12 to 20 carbon atoms, or which is a mixture of two or more of these, said organic material having a melting point in the range 45 to 80°C and
15 being insoluble in water, and in that the foam control agent is produced by a process in which the silicone antifoam and the organic material are contacted in their liquid phase. According to the specification it is preferable that the ratio of organic material to silicone antifoam is kept at 3:1 or above, to ensure free-flowing characteristics of the agent and ease the distribution of the foam control agent in the detergent powder. The most
20 preferred ratio of organic material to silicone antifoam is stated to be from 3:1 to 4:1. The process requires heating of the mixture above the melting point of the wax and the wax does not contribute any additional benefit to the antifoam.

European patent specification EP 210731 provides a particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form,
25 characterized in that the agent is wax-free and comprises a silicone antifoam and organic material having a melting point in the range 50 to 85°C and comprising a monoester of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms. Preferably the ratio of organic material to silicone antifoam is stated to be from 2:1 to 2.5:1. Ratios below 1:1 are said to possibly give both manufacturing problems and
30 storage stability problems, while ratios above 5:1 are stated not to contribute any extra benefit.

Both specifications EP 210721 and EP 210731 state that carrier particles may be included in the foam control agents, which provide a solid basis on which the silicone antifoam and the organic material may be deposited during manufacture. This allows easy mixing in a powder detergent, bulking up the foam control agent to facilitate the dispersibility in the powder detergent. It is stated that the carrier particles are preferably water-soluble solid powders, although the examples given include zeolites and clay minerals as well as sodium sulphate, sodium carbonate, carboxymethyl cellulose and most preferably sodium tripolyphosphate particles.

The granular foam control agents described in EP 210721 and EP 210731 perform quite adequately in many situations, but there is a continuous search for foam control agents which are even more storage stable. There is also a desire to limit the amount of organic material, which in itself does not perform a useful function in the laundering process.

European patent EP 040091 describes suds-suppressing granules which comprise a substantially spherical or cylindrical core material, and one or more coatings comprising a mixture of silicone oil and hydrophobic particles. It is claimed that such granules are less quickly deactivated than those granules in which irregularly shaped substances, such as granular tripolyphosphate, are used as solid core materials for impregnating with silicone antifoams. Suitable core materials mentioned include sucrose, spherical enzyme-containing prills and substantially cylindrical enzyme-containing marumes and Alcalase T granules. The specification further mentions that it is preferred to produce a granule which has a core coated with a particulate absorbent, which is impregnated with the silicone oil mixture. The resultant particle is further coated with a protective envelope. Starch and titanium dioxide are stated to be the preferred absorbents. However, there is a need to provide improved foam controlling agents which use a smaller number of materials and which are not dependant on the geometric shape of the core material.

European patent EP 071481 describes a detergent composition comprising an anionic surfactant and a suds-controlling agent characterized in that the suds-controlling agent comprises a core of gelatinized starch having a mixture of silicone oil and hydrophobic silica absorbed thereon. The specification also states that preferably the suds-control agent is coated with a layer of wax, preferably paraffin wax, in order to

improve the storage characteristics. However, producing a gelatinized starch derivative requires extra processing steps and there is a need to be able to use materials, which are more commonly available and are less expensive.

European patent EP 0496510 describes a granular antifoam where a molten
5 blend of silicone antifoam and stearic acid, stearyl alcohol or glyceryl monostearate is deposited onto native starch. The disadvantage of this formulation is that the waxes are water insoluble and do not dissolve particularly well in normal wash conditions and therefore do not release sufficient antifoam particularly at the beginning of the washing cycle. A general disadvantage of native starch carriers is their poor flowability as they
10 tend to cake. This is a major problem for the process where these antifoam granules have to be mixed with the powder detergent. In addition, native starch has a quite low adsorption rate for liquids compared to zeolites for example, which can cause overgranulation, resulting in granules which are too big and cake. Also, native starch gives a slightly yellowish color to the final antifoam granules, which disturbs the
15 perfectly white look of powder detergents.

US patent 6,129,906 describes a spray-dried, granular powder comprising:

- i) from about 50% to about 99% of a water-soluble carrier,
 - ii) from about 1% to about 50% of a hydrophobic silicone oil dispersed within the carrier,
- 20 wherein the spray-dried powder has a volume average particle size in the range from about 20 microns to about 500 microns, the powder being prepared by spray-drying an aqueous dispersion of the silicone oil and the water-soluble carrier, characterized in that the silicone oil is present in the dispersion in the form of discrete droplets having a volume average droplet size in the range from about 0.5 microns to about 20 microns and
25 that the ratio of the average spray-dried particle size to the average droplet size is at least about 2.5:1. The powder is dry, free-flowing and processable into a punched tablet or other compressed forms, yet efficiently delivers the silicone oil on subsequent dissolution in water. The spray-drying process allows better protection of the encapsulated antifoam compared to the granulation methods described in prior patents. However, the silicone
30 antifoam compound still needs to be diluted with solvents like silicone copolymers or other organic solvents in order to allow easy dispersion of the active antifoam compound into the emulsion that is spray-dried.

Powder detergents are fragranced by spray-applying liquid fragrances onto the powders in a final step. Because fragrances are volatile materials, the spray-application of such fragrances onto powders wastes a significant amount of the fragrance during this process, through evaporation. Furthermore, a homogeneous distribution of the liquid
5 fragrance onto the powder is difficult to obtain.

It has become practice to incorporate microencapsulated fragrances in powder detergent compositions by blending the granular microencapsulated fragrance with the powder detergent.

US patent 6,194,375 describes granular fragranced particles where perfume is
10 absorbed within organic polymer particles, which have a further polymer at their exterior. The further polymer incorporates free hydroxyl groups and serves to promote deposition of the particles from a wash or rinse liquor. The further polymer may be part of an encapsulating shell, but, more conveniently, it is used as a stabilizer during polymerization of the particles. Highly hydrolyzed polyvinyl alcohol is preferred. The
15 resulting granular fragrances are not water-soluble and deposit as particles onto the laundry during a wash cycle.

Spray-drying is the most common encapsulation technique used to stabilize volatile substances such as flavors or fragrances, by encapsulating them in a solid form. Granular microencapsulated fragrances that release the fragrance in contact with water
20 are obtained by spray-drying of an emulsion that contains the emulsified fragrance and a water soluble polymeric matrix. Spray-dried powders are commonly made in the usual spray-drying equipment. Spray-drying is usually effected by means of a rotating disc or of multi-component nozzles. Detailed techniques therefor are described for instance in K. Masters, Spray-Drying Handbook, Longman Scientific and Technical, 1991.

25 A particular example of spray-dried fragrances is disclosed in US patent 4,803,195. The microcapsules are formed of a solid film-forming polymer, for example modified starch or polyvinyl alcohol, and an emulsifying agent. The emulsifying agents are chosen from octenyl succinate-substituted starches, mono- or diglycerides of fatty acids, esters derived from fatty acids and sorbitol or a saccharine, or their
30 alkoxylated derivatives, or an ester of tartaric, citric, ascorbic or lactic acid.

European patent EP 1348423 describes that fragrances can be encapsulated with only polyvinyl alcohol as the polymeric matrix and without the addition of further emulsifiers, due to the emulsifying properties of polyvinyl alcohol.

5 The preparation of spray-dried fragrances of reduced explosivity is described in US 2004/0022821 and such products are commercially available under the trade name Fircaps[®] from Firmenich SA, Switzerland. Upon storage in the powder detergent, the fragrance remains fully encapsulated and does not participate to the odor of the powder detergent. It is only released when the detergent comes into contact with water. Hence, such encapsulated fragrance is used in combination with free liquid sprayed-on fragrance
10 that participates in the odor of the dry powder detergent. Preferred applications are hand-wash powder detergents where the encapsulated fragrance is released when the detergent is added to water and is perceived by the consumer as a strong olfactive boost.

Detailed Description of The Invention

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It has now been ascertained that it is possible to encapsulate a fragrance together with a silicone foam control agent in a granular polymeric matrix by spray-drying.

20 According to the invention the matrix consists of a combination of modified starch and polyvinyl alcohol and protects the antifoam upon storage in a powder detergent. The same matrix permits the fragrance to diffuse out off the microcapsules and therefore allows the encapsulated fragrance to contribute to the odor of the dry powder detergent.

To obtain full protection of the encapsulated antifoam, no waxes or other
25 organic liquids need to be mixed with the antifoam.

The fragrance is used as a solvent for the viscous antifoam compound and allows emulsification into the polymeric matrix without addition of further organic solvents and without heating.

30 In parallel, the antifoam performs as a low volatile diluent for the encapsulated fragrance so that the resulting powder has very low explosivity characteristics. Moreover, we have established that the antifoam is beneficial in retaining

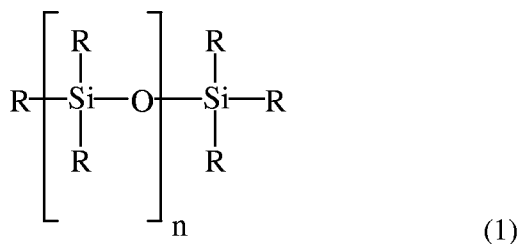
more fragrance on the dry fabrics after the wash treatment. The encapsulated fragranced antifoam is therefore beneficial to the final performance of the powder detergent.

Concerning the process of manufacture, the granules, containing two important actives of a powder detergent can be obtained in one process step with no further coating step required. During the manufacture of the final powder detergent, the two active ingredients can now be added through one type of granule in one step.

The present invention provides a granular fragranced antifoam composition comprising an encapsulated combination of a silicone foam control agent and a fragrance in a protective encapsulating material. According to a particular embodiment of this composition, the foam control agent comprising polydiorganosiloxane and a solid silica.

More preferred embodiments of the invention relate to the use of a matrix formed of polyvinyl alcohol and a dextrose derivative.

The antifoam component of the invention's compositions is a suitable silicone antifoam comprising polydiorganosiloxane and solid silica. Such antifoam compounds are well known in the art and have been described in numerous patent applications. A suitable polydiorganosiloxane is a substantially linear polymer of the average Formula 1 where each R independently can be an alkyl or an aryl radical. Examples of such substituents are methyl, ethyl, propyl, isobutyl and phenyl. A small amount of branching in the chain is possible and small amounts of silicon-bonded hydroxyl groups may also be present.



Preferred polydiorganosiloxanes are polydimethylsiloxanes having trimethylsilyl end-blocking units and having a viscosity at 25°C of from $5 \cdot 10^{-5}$ m²/s to 0.1 m²/s i.e. a value of n in the range of 40 to 1500. These are preferred because of their ready availability and their relatively low cost.

The solid silica of the silicone antifoam can be fumed silica, precipitated silica or silica made by the gel formation technique. The silica particles preferably have an average particle size of from 0.1 to 50 microns, preferably from 1 to 20 microns and a surface area of at least 50 m²/g. These silica particles can be rendered hydrophobic e.g.
5 by treating them with dialkylsilyl groups and/or trialkylsilyl groups either bonded directly onto the silica or by means of a silicone resin. We prefer to employ silica, the particles of which have been rendered hydrophobic, with dimethyl and/or trimethyl silyl groups.

Silicone antifoams employed in a foam control agent according to the invention suitably have an amount of silica in the range of 1 to 30% (more preferably 2.0
10 to 15%) by weight of the total weight of the silicone antifoam. Such silicone antifoams have an average viscosity in the range of from 2 x 10⁻⁴ m²/s to 1 m²/s. Preferred silicone antifoams may have a viscosity in the range of from 5 x 10⁻³ m²/s to 0.1 m²/s. Particularly suitable are silicone antifoams with a viscosity of 2 x 10⁻² m²/s or 5 x 10⁻² m²/s. Such antifoams are commercially available under the trade name Silfoam[®] SC 1132 (Wacker),
15 TP-331 and Y-1462 (General Electric).

The fragrance to be used in the compositions of the invention is any perfuming ingredient in the form of one sole ingredient or in the form of a composition or mixture of ingredients, either as such or, optionally, in a solution or suspension in solvents and adjuvants of current use. The amount of fragrance in the encapsulates of the invention is
20 from 1 to 80%, and preferably from 1 to 50% by weight, relative to the total weight of the microcapsules.

Non limiting examples of solvents that are frequently used in perfume compositions are isopropyl myristate, anozol and propylene glycol. Non limiting examples of adjuvants in perfumes are emulsifiers such as Tween[®]-20 (from ICI) and
25 antioxidants such as butylhydroxy toluene (BHT). The terms "perfume ingredient or composition" as used herein are deemed to define a variety of fragrance materials of both natural and synthetic origins. They include single compounds and mixtures. Specific examples of such components may be found in the current literature, e.g. in Perfume and Flavor Chemicals by S. Arctander, Montclair, N.J. (USA), and other similar text books;
30 and are well-known to the person skilled in the art of perfuming consumer products, i.e. of imparting an odor to a consumer product.

We have now established that the fragrance can act as a solvent for the antifoam compound that is first emulsified and then encapsulated by spray-drying of the emulsion. Furthermore, the antifoam reduces the explosivity of the spray-dried powder so that no further flame retardant additives need to be added in order to allow a safe spray-drying process. Of course, if desired, such flame retardants can also be added.

The polymeric matrices which are convenient for the objectives of the invention retaining the emulsified fragrance during the spray-drying process, are generally water soluble or dispersible polymers that form a dry powder after the spray-drying process.

Non-limiting examples of such polymers are modified cellulose derivatives, like ethyl cellulose or hydroxypropyl cellulose (available under the trade name Tylose[®] from Clariant), modified starch derivatives, like dioctenyl succinated starch (Capsul[®] from National Starch), sugars or dextroses or their modified derivatives, like refined polydextrose (Litesse[®] from Danisco), polyvinyl alcohols (Mowiol[®] from Clariant) and combinations thereof.

We have ascertained that the specific combination of sugar, polydextrose and polyvinyl alcohol provides excellent storage stability for the encapsulated antifoam in powder detergents, also under excessive humidity conditions. Furthermore, the matrix allows the encapsulated fragrance to diffuse out off the matrix into the powder detergent and to thus participate in the perfume odor of this powder detergent, without significant loss of fragrance, or degradation thereof, during the storage period.

The present invention also provides in another of its aspects a detergent composition in powder form, comprising a detergent component and a fragranced foam control agent or composition according to the invention. The fragranced foam control agent according to the invention may be added to the detergent component in a proportion of from 0.1 to 25% by weight based on the total detergent composition. Preferably fragranced foam control agents are added in a proportion of from 0.25 to 5% by weight based on the total detergent composition.

Suitable detergent components comprise an active detergent, organic and inorganic builder salts and other additives and diluents. The active detergent may comprise organic detergent surfactants of the anionic, cationic, non-ionic or amphoteric type, or mixtures thereof. Suitable anionic organic detergent surfactants include alkali metal soaps of higher fatty acids, alkyl aryl sulphonates, for example sodium dodecyl

benzene sulphonate, long chain (fatty) alcohol sulphates, olefin sulphates and sulphonates, sulphated monoglycerides, sulphated ethers, sulphosuccinates, alkane sulphonates, phosphate esters, alkyl isothionates, sucrose esters and fluoro-surfactants. Suitable cationic organic detergent surfactants include alkyl-amine salts, quaternary ammonium salts, sulphonium salts and phosphonium salts.

Suitable non-ionic organic surfactants include condensates of ethylene oxide with a long chain (fatty) alcohol or fatty acid, for example C14-15 alcohol, condensed with 7 moles of ethylene oxide (Dobanol[®] 45-7), condensates of ethylene oxide with an amine or an amide, condensation products of ethylene and propylene oxides, fatty acid alkylol amides and fatty amine oxides. Suitable amphoteric organic detergent surfactants include imidazoline compounds, alkylaminoacid salts and betaines. Examples of inorganic components are phosphates and polyphosphates, silicates, such as sodium silicates, carbonates, sulphates, and oxygen releasing compounds, such as sodium perborate and other bleaching agents and zeolites. Examples of organic components are anti-re-deposition agents such as carboxy methyl cellulose (CMC), brighteners, chelating agents, such as ethylene diamine tetra-acetic acid (EDTA) and nitrilotriacetic acid (NTA), enzymes and bacteriostats. Materials suitable for the detergent component are well known to the person skilled in the art and are described in many text books, for example Synthetic Detergents, A. Davidsohn and B. M. Milwidsky, 6th edition, George Godwin (1978).

Brief Description of the Figures

- Fig. 1 shows the migration of silicone antifoam onto powder detergent.
- Fig. 2 shows the intensity of volatile perfume ingredients in the gas phase of a powder detergent containing equal amounts of perfume in liquid and encapsulated form.
- Fig. 3 shows the DVS analysis of granulated fragranced antifoam (sample B from Example 1) showing stability of the powder but weight loss at elevated humidity due to evaporation of perfume.
- Fig. 4 shows the intensity of volatile perfume ingredients in the gas phase of dried fabric washed with powder detergent containing equal amounts of encapsulated perfume with antifoam (sample B) and without antifoam (sample A).

Specific Embodiments of The Invention

Example 1

5 Preparation of spray-dried fragranced antifoam microcapsules

A perfuming antifoam emulsion was prepared from the following formula:

Ingredients	sample A grams	sample B grams
TP-331 ¹⁾	0	20.00
Isopropyl Myristate	20.00	0
Lavender perfume ²⁾	19.88	19.88
Tween-20 ³⁾	0.12	0.12
Litesse [®] ⁴⁾	15.00	15.00
Sucrose	10.00	10.00
Mowiol 4-88 G2 ⁵⁾	35.00	35.00
Water	300.00	300.00

- 10 1) antifoam compound; origin: General Electric, Switzerland
 2) origin: Firmenich SA, Geneva, Switzerland
 3) polyoxyethylene monolaurate; origin: ICI Chemicals, Great Britain
 4) refined polydextrose; origin: Danisco, Great Britain
 5) polyvinyl alcohol; origin: Kuraray/Omya, Switzerland

15

First, the antifoam compound was dissolved in the perfume and Tween-20. The remaining ingredients were homogenized by means of a Silverson type fast stirrer and the solution of perfume and antifoam was emulsified into this mix at the end. The mixture was then spray-dried in a Sodeva apparatus with an emulsion output of 2 kg/h, drying air:

20 320 m³/h at 350°C and 0.45 x 10⁵Pa.

The resulting powder had an average particle size of 27 microns, evaluated by laser diffraction, and the content of encapsulated liquids being 38.5% by weight, analyzed by hydrodistillation. The dust explosion violence was determined to be K_{st} = 93 bar • m • s⁻¹

which leads to a dust explosion hazard class of ST-1, according to VDI-3673. This means that the powder has weak to moderate explosion characteristics. The dust explosion violence was measured in a 20 liter Sphere Apparatus. The apparatus monitors the evolution of the explosion pressure of fuel/air mixtures in a closed vessel (under vacuum
5 and by a chemical ignition source of 10 kJ) with time and over a range of product concentrations. The international standards (VDI Guideline 2263 part 1: Dust Fires and Dust Explosions, Hazard Assessment – Protective Measures, Test Methods for the Determination of Safety Characteristics of Dusts, Beuth, Berlin, May 1990) describe the test equipments and methods.

10

Fragranced foam control agents according to the invention do not appear to give rise to deposits of the organic material upon textiles laundered with detergent compositions containing these fragranced foam control agents in amounts sufficient to control the foam level during laundering operations. At the same concentration that is required to obtain
15 good foam control, the fragranced foam control agents are sufficient to fragrance the dry powder so that no additional liquid fragrance needs to be spray-applied onto the powder detergent.

Example 2

20

Preparation of a detergent and storage stability testing thereof

A detergent composition was prepared by mixing 7.8 parts sodium dodecyl benzene sulphonate, 4 parts of Dobanol[®] 45-7 (linear primary alcohol ethoxylate C14-15 7EO),
25 21 parts sodium carbonate, 30.2 parts zeolite A, 12 parts sodium sulphate and 25 parts sodium perborate. This composition is a base for a detergent powder composition.

To 110 g of the detergent composition above, there was added 2.4 weight % of the granular fragranced foam control agent of Example 1 to prepare a detergent according to
30 the invention.

To a second sample of 110 g of the detergent composition 0.96% of a liquid blend of antifoam and fragrance were added to give, based on the weight of the detergent test composition, 0.48% of silicone antifoam and 0.48% of fragrance in a comparative detergent.

5

A conventional automatic washing machine (Miele Professional WS 5425) of the front loading type having a transparent door, through which clothes may be loaded to a rotation drum of the machine, was loaded with 8 liters of tap water. A main wash cycle at 95°C was carried out using 110 g of one or the other detergent composition. The door of the washing machine was divided in its height by a scale from 0 to 14. The level of the top of the foam during the wash cycle was compared with the scale every five minutes of the main wash, when the rotation drum of the washing machine was stationary and the scale values were recorded. Scale of 15 meant overflow. By overflow we mean that the foam came out of the washing machine through a vent at the top.

15 The results of the tests are recorded in Table 1.

Table 1: Foam height (scale 0-15) of fragranced antifoam compounds in 110 g test detergent, 95°C wash cycle, no laundry

minutes of wash cycle	Foam height (0 – 15 scale; 15 = foam runs out off the machine)				
	no additive	0.96% of liquid blend of TP-331: perfume (1:1)	0.96% of liquid blend of TP-331: perfume (1:1)	2.4% of encapsulated fragranced antifoam from Example 1	2.4% of encapsulated fragranced antifoam from Example 1
		freshly mixed into detergent	1 month storage in detergent, open paper cup, 37°C, 70% RH	freshly mixed into detergent	1 month storage in detergent, open paper cup, 37°C, 70% RH
5 min	5	0	5	0	3
10 min	8	0	8	3	0
12 min	10	0	10	3	0
13 min	15	0	15	3	0
15 min	15	0	15	3	3
17 min	15	4	15	5	4
20 min	15	4	15	5	5
25 min	15	5	15	6	5
29 min	15	6	15	6	5

As can be seen from the results shown in Table 1, the sample detergent compositions containing a fragranced foam control agent according to the invention retain their foam control ability after prolonged storage, whereas the non-encapsulated liquids are not sufficiently effective.

Foam control activity was then tested under prolonged storage for 90 days at 37°C and 70% relative humidity.

The fragranced foam control agent was mixed with the powder detergent and stored in an open paper cup and in a closed plastic cup for comparison. The results are recorded in Table 2.

Table 2: Foam height (scale 0-15) of fragranced antifoam compounds in 110 g test detergent, 95°C wash cycle, no laundry

15

minutes of wash cycle	Foam height (0 – 15 scale; 15 = foam runs out off the machine)			
	2.4% of encapsulated fragranced antifoam from Example 1	2.4% of encapsulated fragranced antifoam from Example 1	2.4% of encapsulated fragranced antifoam from Example 1	2.4% of encapsulated fragranced antifoam from Example 1
	freshly mixed into detergent	1 month storage in detergent, open paper cup, 37°C, 70% RH	3 months storage in detergent, open paper cup, 37°C, 70% RH	3 months storage in detergent, closed plastic cup, 37°C, 70% RH
5 min	0	3	5	0
10 min	3	0	7	0
12 min	3	0	9	0
13 min	3	0	9	0
15 min	3	3	15	0
17 min	5	4	15	0
20 min	5	5	15	3
25 min	6	5	15	4
29 min	6	5	15	4

As can be seen from the results shown in Table 2, the fragranced foam control agent fully protects the antifoam for 1 month in powder detergent when exposed to elevated

humidity (70% relative humidity). After 3 months storage under this elevated humidity, the water soluble encapsulation matrix starts dissolving and cannot protect the antifoam from the detergent anymore. However, if the detergent is stored in sealed containers, the encapsulation matrix remains highly protective for the antifoam, even after 3 months
5 storage at 37°C.

The amount of olfactively perceivable fragrance in the powder detergent samples was determined by gas chromatographic head space analysis. In one test sample 0.96% of a liquid blend of antifoam and perfume (antifoam:perfume = 1:1) was added to the
10 detergent.

In a second test sample, 2.4% of microcapsules from Example 1 containing 40% of the same encapsulated blend were added. The test samples were stored at 37°C in a closed glass jar for 1 month. Afterwards, the samples were heated up to 60°C and the concentration of fragrance in the head space of the samples was analyzed by gas
15 chromatography. The results recorded in Figure 2 indicate that most of the encapsulated fragrance diffused out into the powder detergent and could be detected in the air.

The stability of the granulated fragranced antifoam against humidity was analysed by Dynamic Vapour Sorption (DVS) in an apparatus from Surface Measurement Systems
20 Ltd.

In this test, a sample of the granules are weighed on a balance which is placed in a chamber which is flushed by a humidity controlled air stream. Every 2 hours, the relative humidity is increased by 10% starting from 40% relative humidity up to 90% relative humidity. Temperature is controlled to remain constant at 30°C. The weight change of
25 the exposed sample is observed. Increase in weight indicates an uptake of humidity due to hygroscopicity and decrease in weight indicates loss of volatile materials, in our case perfume. At the same time, photos can be taken of the powder on the balance to follow its physical structure. The DVS analysis of granulated fragranced antifoam (sample B from Example 1) is shown in Figure 3.

30

The analysis shows that the granular powder remains stable even at elevated humidity which allows full protection of the encapsulated antifoam compound.

At the same time, volatile perfume diffuses out of the granular powder and participates to the odor of the powder.

Example 3

5

Perfume retention on fabric testing

A. Olfactive performance

As described in Example 1, spray-dried fragranced antifoam granules were prepared
10 containing 20% of antifoam compound and 19.88% of fragrance (sample B).

The same encapsulation was repeated but the antifoam compound was replaced by isopropyl myristate resulting in a spray-dried fragranced granule without antifoam (sample A).

15 With these two samples of granules, wash tests were carried out.

0.19 g of spray-dried granules were added to 8 g of model powder detergent. The detergent was added to 500 ml of warm water containing a 40 g piece of terry towel fabric. The towel was washed with this mixture in a sealed plastic container for 1 hour by continuous shaking as shown in Figure 5.

20

Afterwards, the fabric piece was rinsed with cold water 3 times and manually wringed after each rinse. The fabric piece was then line dried at ambient temperature for two days.

Table 3: Olfactive evaluation (scale 0 – 10):

25

washed	wet, 1 hour after wash	dry, 2 days after wash
fragrance + antifoam encapsulated (sample B)	1	0 but 4 after rubbing
fragrance + isopropyl myristate encapsulated (sample A)	1	0

Olfactive evaluation was carried out on a blind test with a panel of 5 persons. An intensity scale from 0 (= no smell) to 10 (= very strong smell) was applied. The fabric was smelled wet and after line drying. The dry fabric was smelled before and after gentle rubbing.

- 5 A higher rating after rubbing was given to the fabric that was washed with the granulated fragranced antifoam compared to the antifoam-free granules (Table 3).

This indicates an improved perfume retention on dry fabric by the blending of silicone antifoam with the perfume prior to encapsulation.

10 B. Head Space Analysis

To determine the retention of volatile notes from the fragrance on the dried fabric, the fabric pieces were heated up to 60°C and the volatile substances in the head space were measured by gas chromatography coupled with mass spectrometry. The results are represented in Figure 4 and summarized in the Table herebelow.

15

GC-MS Results

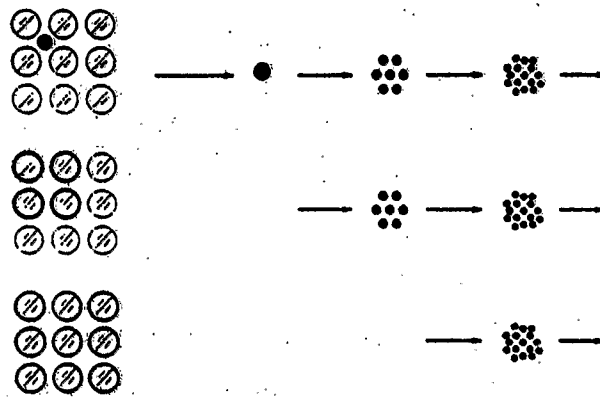
	Sample A		Sample B		
Retention time (min)	Area	Percent	Area	Percent	raw material
4.71	79'378'949	2.84	102'034'403	4.86	Hedione®
6.93	35'989'219	1.29	40'829'763	1.94	Ethylhexyl acetate
9.65	21'554'122	0.77	45'577'180	2.17	n.d.
9.77	138'618'312	4.97	127'325'438	6.06	Verdox
19.35	75'746'027	2.71	94'057'772	4.48	n.d.

- 20 The head space analysis of the washed and dried fabrics indicates that some volatile notes of the fragrance can be better retained if the fragrance is blended with the silicone antifoam compound prior to encapsulation.

Claims

1. A granular fragranced antifoam composition comprising an encapsulated
5 combination of a silicone foam control agent and a fragrance in a protective encapsulating material.
2. A granular fragranced antifoam composition according to claim 1 wherein the
silicone foam control agent comprises polydiorganosiloxane and a solid silica.
- 10 3. A granular fragranced antifoam composition according to claim 1 or 2
wherein the protective encapsulating material comprises combinations of polyvinyl
alcohol and a dextrose derivative.
- 15 4. A granular fragranced antifoam composition according to anyone of claims 1
to 3 containing 1-60%, preferably 10-50% by weight of encapsulate.
5. A granular fragranced antifoam composition according to claims 1 to 4 which
is obtainable by spray drying an oil in water emulsion, wherein the solution of silicone
20 foam control agent and fragrance form the oil phase thereof and the dispersion of the
polymeric matrix constitutes the aqueous phase.
6. A granular fragranced antifoam composition according to anyone of claims 1
to 5 that protects the encapsulated antifoam compound upon storage in powder detergents
25 and that allows the encapsulated fragrance to diffuse into the powder detergent.
7. A powder detergent composition containing 0.1-5%, preferably 1-2% of a
granular fragranced antifoam composition according to anyone of claims 1 to 6.

Figure 1



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(07.08.06)

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Figure 2

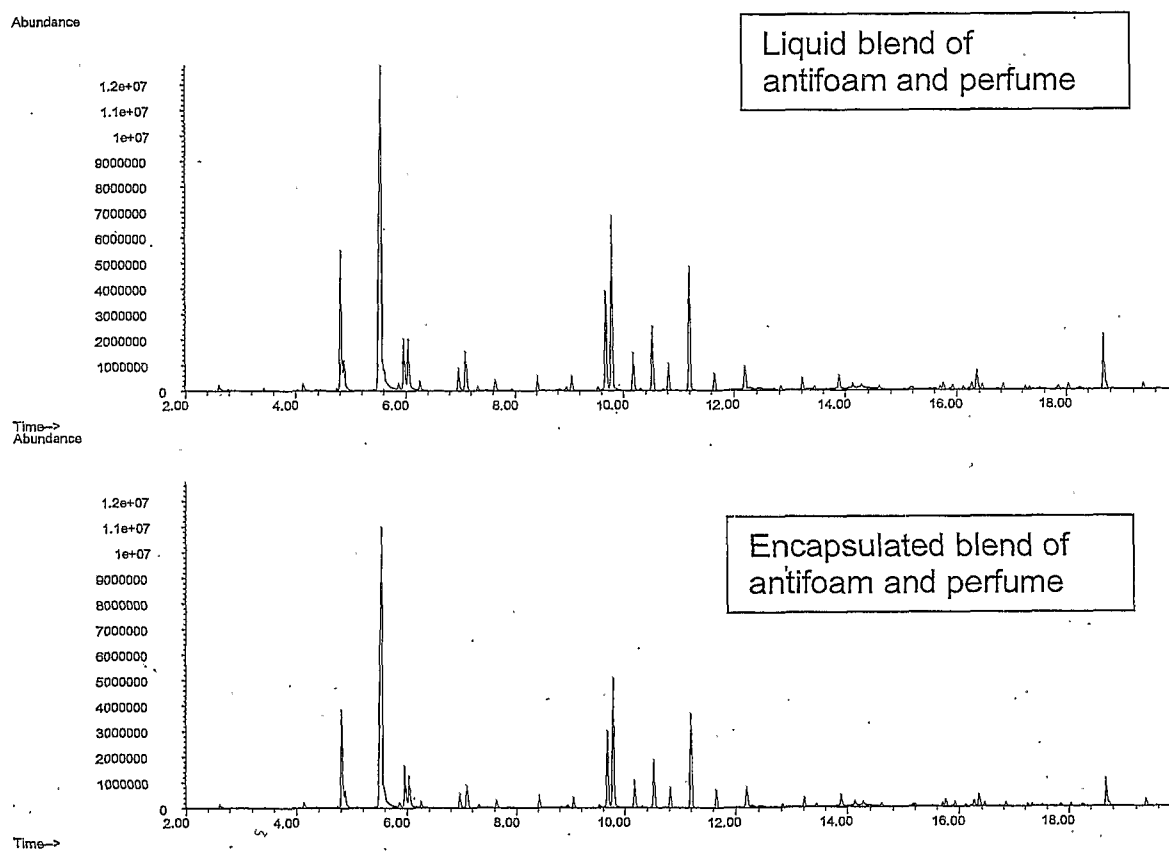


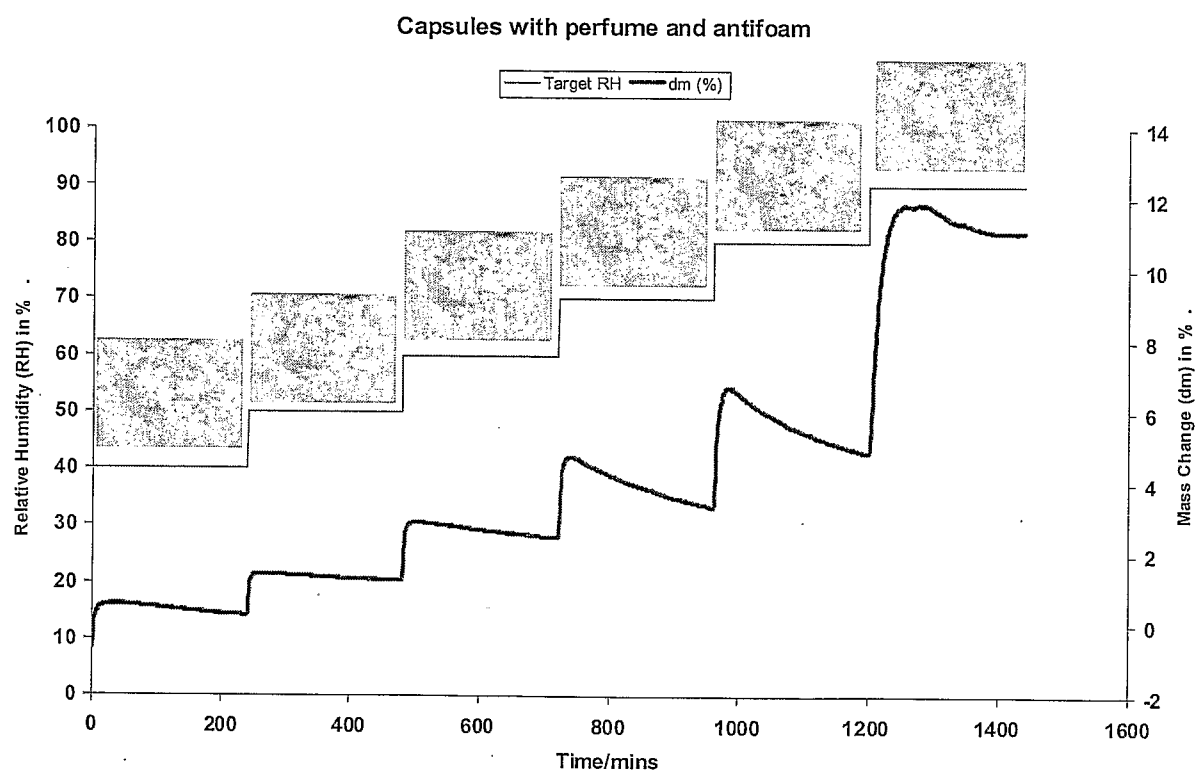
Figure 3

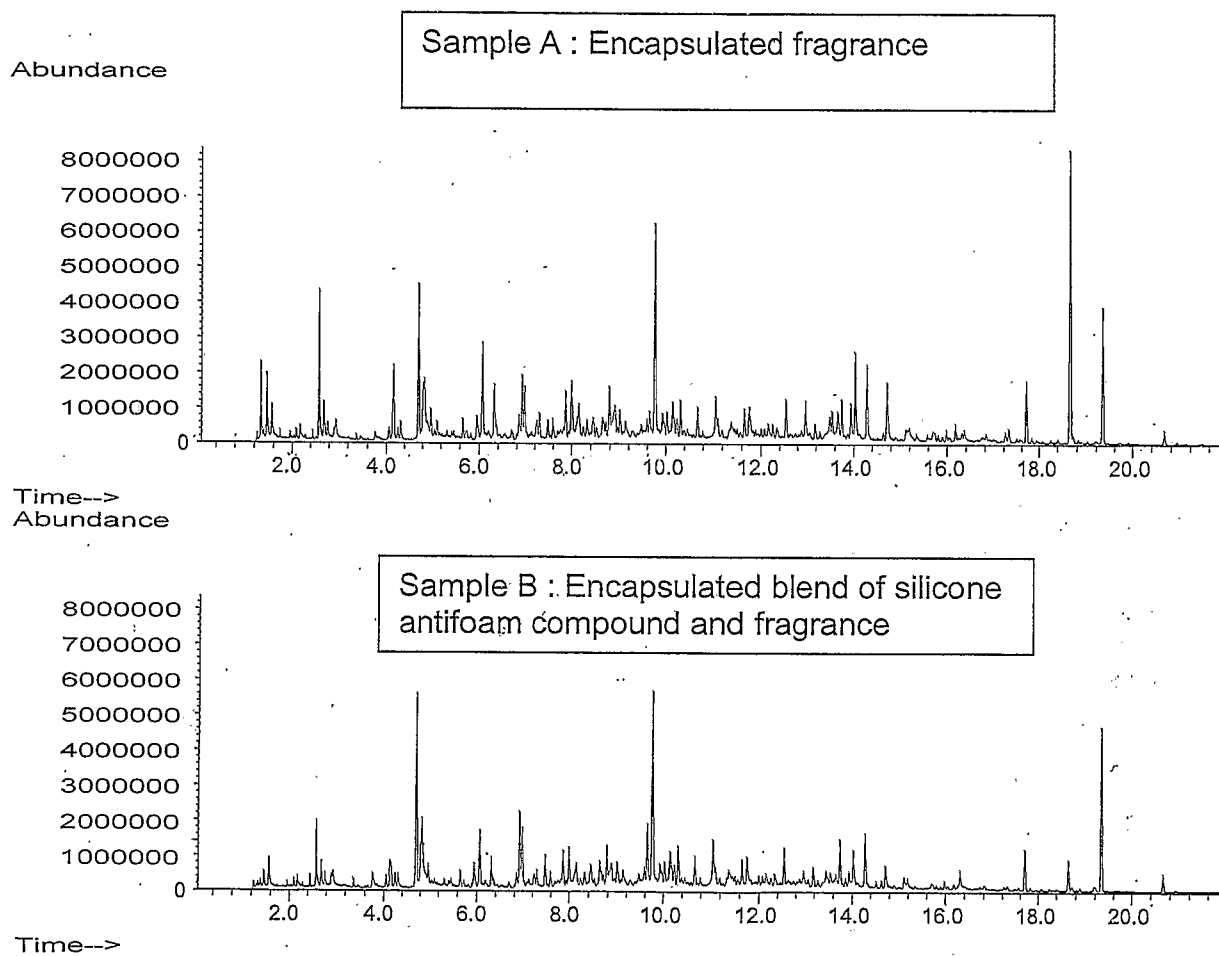
Figure 4

Figure 5



Fabric wash test

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2006/052041

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D3/50 C11D3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 129 906 A (STEVENTON ANTHONY JAMES [GB]) 10 October 2000 (2000-10-10) cited in the application column 2, line 50 - column 3, line 24 column 5, lines 59-65 column 6, lines 63-66 claims 1,2,9,14,17; examples I-III	1-7
X	US 2004/022821 A1 (HOLZNER GUNTER [CH] ET AL) 5 February 2004 (2004-02-05) cited in the application paragraphs [0023], [0024], [0028], [0030], [0031]; claims 1,7,11,12 ----- -/-	1-7

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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Date of the actual completion of the international search

10 November 2006

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 1 533 364 A2 (INTERNATIONAL FLAVORS & FRAGRANCES INC [US]) 25 May 2005 (2005-05-25) paragraphs [0001], [0011], [0016], [0029], [0048]; claims 1,17,18,22,23,32,33,38-46	1-7
A	US 5 804 544 A (POWELL SUZANNE [GB] ET AL) 8 September 1998 (1998-09-08) claim 1; example 1	1-7

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International application No
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US 5804544	A	08-09-1998	NONE