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(54) Title: LAUNDRY DETERGENT COMPOSITION COMPRISING A GLYCOSYL HYDROLASE AND A BENEFIT AGENT CONTAINING DELIVERY PARTICLE

(57) Abstract: The present invention relates to a laundry detergent composition comprising a glycosyl hydrolase and a benefit agent containing delivery particles, compositions comprising said particles, and processes for making and using the aforementioned particles and compositions.

LAUNDRY DETERGENT COMPOSITION COMPRISING A GLYCOSYL HYDROLASE
AND A BENEFIT AGENT CONTAINING DELIVERY PARTICLE

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FIELD OF INVENTION

The present application relates to a laundry detergent composition comprising a glycosyl hydrolase and a benefit agent containing delivery particle.

BACKGROUND OF THE INVENTION

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Benefit agents, such as perfumes, silicones, waxes, vitamins and fabric softening agents, are expensive and generally less effective when employed at high levels in fabric care compositions. As a result, there is a desire to maximize the effectiveness of such benefit agents. One method of achieving such objective is to improve the delivery efficiencies of such benefit agents. Unfortunately, it is difficult to improve the delivery efficiencies of benefit agents as such agents may be lost do to the agents' physical or chemical characteristics, or such agents may be incompatible with other compositional components or the situs that is treated.

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Accordingly, there is a need for a composition that provides improved benefit agent delivery efficiency.

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SUMMARY OF THE INVENTION

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The present invention relates to a laundry detergent composition comprising a glycosyl hydrolase and a benefit agent containing delivery particles comprising a core material and a wall material that at least partially surrounds the core material. Without wishing to be bound by theory the Inventors believe that the action of certain glycosyl hydrolase on the fabric surface opens up the pore structure of the cotton fibres so as to increase the entrapment of the benefit agent containing particles in the fabric. In addition, the action of these certain glycosyl hydrolases increases the surface area of the fabric, further improving the performance of the benefit agent during the laundering process.

DETAILED DESCRIPTION OF THE INVENTION

Glycosyl hydrolase

The glycosyl hydrolase has enzymatic activity towards both xyloglucan and amorphous
5 cellulose substrates, wherein the glycosyl hydrolase is selected from GH families 5, 12, 44 or 74.

The enzymatic activity towards xyloglucan substrates is described in more detail below.
The enzymatic activity towards amorphous cellulose substrates is described in more detail below.

The glycosyl hydrolase enzyme preferably belongs to glycosyl hydrolase family 44. The
glycosyl hydrolase (GH) family definition is described in more detail in Biochem J. 1991, v280,
10 309-316.

The glycosyl hydrolase enzyme preferably has a sequence at least 70%, or at least 75% or
at least 80%, or at least 85%, or at least 90%, or at least 95% identical to sequence ID No. 1.

For purposes of the present invention, the degree of identity between two amino acid
sequences is determined using the Needleman-Wunsch algorithm (Needleman and Wunsch,
15 1970, *J. Mol. Biol.* 48: 443-453) as implemented in the Needle program of the EMBOSS package
(EMBOSS: The European Molecular Biology Open Software Suite, Rice *et al.*, 2000, *Trends in
Genetics* 16: 276-277), preferably version 3.0.0 or later. The optional parameters used are gap
open penalty of 10, gap extension penalty of 0.5, and the EBLOSUM62 (EMBOSS version of
BLOSUM62) substitution matrix. The output of Needle labeled "longest identity" (obtained
20 using the -nobrief option) is used as the percent identity and is calculated as follows: (Identical
Residues x 100)/(Length of Alignment – Total Number of Gaps in Alignment).

Suitable glycosyl hydrolases are selected from the group consisting of: GH family 44
glycosyl hydrolases from *Paenibacillus polyxyma* (wild-type) such as XYG1006 described in
WO 01/062903 or are variants thereof; GH family 12 glycosyl hydrolases from *Bacillus*
25 *licheniformis* (wild-type) such as Seq. No. ID: 1 described in WO 99/02663 or are variants
thereof; GH family 5 glycosyl hydrolases from *Bacillus agaradhaerens* (wild type) or variants
thereof; GH family 5 glycosyl hydrolases from *Paenibacillus* (wild type) such as XYG1034 and
XYG 1022 described in WO 01/064853 or variants thereof; GH family 74 glycosyl hydrolases
from *Jonesia* sp. (wild type) such as XYG1020 described in WO 2002/077242 or variants thereof;
30 and GH family 74 glycosyl hydrolases from *Trichoderma Reesei* (wild type), such as the enzyme
described in more detail in Sequence ID no. 2 of WO03/089598, or variants thereof.

Preferred glycosyl hydrolases are selected from the group consisting of: GH family 44 glycosyl hydrolases from *Paenibacillus polyxyma* (wild-type) such as XYG1006 or are variants thereof.

5 Enzymatic activity towards xyloglucan substrates

An enzyme is deemed to have activity towards xyloglucan if the pure enzyme has a specific activity of greater than 50000 XyloU/g according to the following assay at pH 7.5.

The xyloglucanase activity is measured using AZCL-xyloglucan from Megazyme, Ireland as substrate (blue substrate).

10 A solution of 0.2% of the blue substrate is suspended in a 0.1M phosphate buffer pH 7.5, 20°C under stirring in a 1.5ml Eppendorf tubes (0.75ml to each), 50 microlitres enzyme solution is added and they are incubated in an Eppendorf Thermomixer for 20 minutes at 40°C, with a mixing of 1200 rpm. After incubation the coloured solution is separated from the solid by 4 minutes centrifugation at 14,000 rpm and the absorbance of the supernatant is measured at 600nm
15 in a 1cm cuvette using a spectrophotometer. One XyloU unit is defined as the amount of enzyme resulting in an absorbance of 0.24 in a 1cm cuvette at 600nm.

Only absorbance values between 0.1 and 0.8 are used to calculate the XyloU activity. If an absorbance value is measured outside this range, optimization of the starting enzyme concentration should be carried out accordingly.

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Enzymatic activity towards amorphous cellulose substrates

An enzyme is deemed to have activity towards amorphous cellulose if the pure enzyme has a specific activity of greater than 20000 EBG/g according to the following assay at pH 7.5. Chemicals used as buffers and substrates were commercial products of at least reagent grade.

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Endoglucanase Activity Assay Materials:

0.1M phosphate buffer pH 7.5

Cellazyme C tablets, supplied by Megazyme International, Ireland.

Glass microfiber filters, GF/C, 9cm diameter, supplied by Whatman.

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Method:

In test tubes, mix 1ml pH 7,5 buffer and 5ml deionised water.

Add 100 microliter of the enzyme sample (or of dilutions of the enzyme sample with known weight:weight dilution factor). Add 1 Cellzyme C tablet into each tube, cap the tubes and mix on a vortex mixer for 10 seconds. Place the tubes in a thermostated water bath, temperature 40°C. After 15, 30 and 45 minutes, mix the contents of the tubes by inverting the tubes, and replace in the water bath. After 60 minutes, mix the contents of the tubes by inversion and then filter through a GF/C filter. Collect the filtrate in a clean tube.

Measure Absorbance (A_{enz}) at 590nm, with a spectrophotometer. A blank value, A_{water} , is determined by adding 100 μ l water instead of 100 microliter enzyme dilution.

Calculate $\Delta A = A_{enz} - A_{water}$.

10 ΔA must be < 0.5 . If higher results are obtained, repeat with a different enzyme dilution factor. Determine DFO.1, where DFO.1 is the dilution factor needed to give $\Delta A = 0.1$.

Unit Definition: 1 Endo-Beta-Glucanase activity unit (1 EBG) is the amount of enzyme that gives $\Delta A = 0.10$, under the assay conditions specified above. Thus, for example, if a given enzyme sample, after dilution by a dilution factor of 100, gives $\Delta A = 0.10$, then the enzyme sample has an activity of 100 EBG/g.

Benefit Agent Containing Delivery Particle

The Inventors discovered that the problem of achieving effective and efficient benefit agent delivery can be solved in an economical manner when a benefit agent containing delivery particle having a certain combination of physical and chemical characteristics is incorporated in a laundry detergent composition that additionally comprises a glycosyl hydrolase. Such physical and chemical characteristics are defined by the following parameters: particle size coefficient of variation, fracture strength, benefit agent retention ratio and average particle size. Such parameters may be combined to yield a Delivery Index.

In one aspect, the particle comprises a core material and a wall material that at least partially surrounds the core material, said particle having a Delivery Index of at least about 0.05, at least about 7, or at least about 70.

In one aspect, the particle comprises a core material and a wall material that at least partially surrounds the core material, said particle having:

- a.) a particle size coefficient of variation of from about 1.5 to about 6.0, from about 2.0 to about 3.5, or even from about 2.5 to about 3.2;

b.) a fracture strength of from about 0.1 psia to about 110 psia, from about 1 to about 50 psia, or even from about 4 to about 16 psia;

c.) a benefit agent retention ratio of from about 2 to about 110, from about 30 to about 90, or even from about 40 to about 70; and

5 d.) an average particle size of from about 1 micron to about 100 microns, from about 5 microns to about 80 microns, or even from about 15 microns to about 50 microns.

In one aspect of the present invention, said particle may have and/or comprise any combination of the parameters described in the present specification.

Useful wall materials include materials selected from the group consisting of polyethylenes, polyamides, polystyrenes, polyisoprenes, polycarbonates, polyesters, polyacrylates, polyureas, polyurethanes, polyolefins, polysaccharides, epoxy resins, vinyl polymers, and mixtures thereof. In one aspect, useful wall materials include materials that are sufficiently impervious to the core material and the materials in the environment in which the benefit agent containing delivery particle will be employed, to permit the delivery benefit to be obtained. Suitable impervious wall materials include materials selected from the group consisting of reaction products of one or more amines with one or more aldehydes, such as urea cross-linked with formaldehyde or gluteraldehyde, melamine cross-linked with formaldehyde; gelatin-polyphosphate coacervates optionally cross-linked with gluteraldehyde; gelatin-gum Arabic coacervates; cross-linked silicone fluids; polyamine reacted with polyisocyanates and mixtures thereof. In one aspect, the wall material comprises melamine cross-linked with formaldehyde.

Useful core materials include perfume raw materials, silicone oils, waxes, hydrocarbons, higher fatty acids, essential oils, lipids, skin coolants, vitamins, sunscreens, antioxidants, glycerine, catalysts, bleach particles, silicon dioxide particles, malodor reducing agents, dyes, 10 brighteners, antibacterial actives, antiperspirant actives, cationic polymers and mixtures thereof. In one aspect, said perfume raw material is selected from the group consisting of alcohols, ketones, aldehydes, esters, ethers, nitriles alkenes. In one aspect the core material comprises a perfume. In one aspect, said perfume comprises perfume raw materials selected from the group consisting of alcohols, ketones, aldehydes, esters, ethers, nitriles alkenes and mixtures thereof. 15 In one aspect, said perfume may comprise a perfume raw material selected from the group consisting of perfume raw materials having a boiling point (B.P.) lower than about 250°C and a ClogP lower than about 3, perfume raw materials having a B.P. of greater than about 250°C and a ClogP of greater than about 3, perfume raw materials having a B.P. of greater than about 250°C

and a ClogP lower than about 3, perfume raw materials having a B.P. lower than about 250°C and a ClogP greater than about 3 and mixtures thereof. Perfume raw materials having a boiling point B.P. lower than about 250°C and a ClogP lower than about 3 are known as Quadrant I perfume raw materials, perfume raw materials having a B.P. of greater than about 250°C and a ClogP of greater than about 3 are known as Quadrant IV perfume raw materials, perfume raw materials having a B.P. of greater than about 250°C and a ClogP lower than about 3 are known as Quadrant II perfume raw materials, perfume raw materials having a B.P. lower than about 250°C and a ClogP greater than about 3 are known as a Quadrant III perfume raw materials. In one aspect, said perfume comprises a perfume raw material having B.P. of lower than about 250°C. In one aspect, said perfume comprises a perfume raw material selected from the group consisting of Quadrant I, II, III perfume raw materials and mixtures thereof. In one aspect, said perfume comprises a Quadrant III perfume raw material. Suitable Quadrant I, II, III and IV perfume raw materials are disclosed in U.S. patent 6,869,923 B1.

In one aspect, said perfume comprises a Quadrant IV perfume raw material. While not being bound by theory, it is believed that such Quadrant IV perfume raw materials can improve perfume odor "balance". Said perfume may comprise, based on total perfume weight, less than about 30%, less than about 20%, or even less than about 15% of said Quadrant IV perfume raw material.

The perfume raw materials and accords may be obtained from one or more of the following companies Firmenich (Geneva, Switzerland), Givaudan (Argenteuil, France), IFF (Hazlet, NJ), Quest (Mount Olive, NJ), Bedoukian (Danbury, CT), Sigma Aldrich (St. Louis, MO), Millennium Specialty Chemicals (Olympia Fields, IL), Polarone International (Jersey City, NJ), Fragrance Resources (Keyport, NJ), and Aroma & Flavor Specialties (Danbury, CT).

Process of Making Benefit Agent Containing Delivery Particles

The particle disclosed in the present application may be made via the teachings of USP 6,592,990 B2 and/or USP 6,544,926 B1 and the examples disclosed herein.

Laundry detergent composition

The laundry detergent composition comprises: (a) a glycosyl hydrolase having enzymatic activity towards both xyloglucan and amorphous cellulose substrates, wherein the glycosyl hydrolase is selected from GH families 5, 12, 44 or 74; (b) a particle comprising a core material

and a wall material that surrounds the core material, said particle preferably having a Delivery Index of at least about 0.05 said composition being a consumer product; and (c) detergent surfactant.

While the precise level of particle (b) that is employed depends on the type and end use of the composition, a composition may comprise from about 0.01 to about 10, from about 0.1 to about 10, or even from about 0.2 to about 5 weight % of said particle based on total composition weight. In one aspect, a cleaning composition may comprise, from about 0.1 to about 1 weight % of such particle based on total composition weight of such particle. In one aspect, a fabric treatment composition may comprise, based on total fabric treatment composition weight, from about 0.01 to about 10% of such particle.

Aspects of the invention include the use of the particles of the present invention in laundry detergent compositions (e.g., TIDE™). The compositions disclosed herein are typically formulated such that, during use in aqueous cleaning operations, the wash water will have a pH of between about 6.5 and about 12, or between about 7.5 and 10.5.

Laundry detergent compositions disclosed herein typically comprise a fabric softening active ("FSA"). Suitable fabric softening actives, include, but are not limited to, materials selected from the group consisting of quats, amines, fatty esters, sucrose esters, silicones, dispersible polyolefins, clays, polysaccharides, fatty oils, polymer latexes and mixtures thereof.

The composition is preferably in the form of a liquid. The composition typically comprises adjunct materials. The adjunct materials are described in more detail below.

The composition can be in any form. The composition may be in the form of a liquid or solid. The composition is preferably in the form of a liquid. The composition may be at least partially, preferably completely, enclosed by a water-soluble film.

Solid laundry detergent composition

In one embodiment of the present invention, the composition is a solid laundry detergent composition, preferably a solid laundry powder detergent composition.

The composition preferably comprises from 0wt% to 10wt%, or even to 5wt% zeolite builder. The composition also preferably comprises from 0wt% to 10wt%, or even to 5wt% phosphate builder.

The composition typically comprises anionic detergent surfactant, preferably linear alkyl benzene sulphonate, preferably in combination with a co-surfactant. Preferred co-surfactants are alkyl ethoxylated sulphates having an average degree of ethoxylation of from 1 to 10, preferably

from 1 to 3, and/or ethoxylated alcohols having an average degree of ethoxylation of from 1 to 10, preferably from 3 to 7.

The composition preferably comprises chelant, preferably the composition comprises from 0.3wt% to 2.0wt% chelant. A suitable chelant is ethylenediamine-N,N' -disuccinic acid (EDDS).

The composition may comprise cellulose polymers, such as sodium or potassium salts of carboxymethyl cellulose, carboxyethyl cellulose, sulfoethyl cellulose, sulfopropyl cellulose, cellulose sulfate, phosphorylated cellulose, carboxymethyl hydroxyethyl cellulose, carboxymethyl hydroxypropyl cellulose, sulfoethyl hydroxyethyl cellulose, sulfoethyl hydroxypropyl cellulose, carboxymethyl methyl hydroxyethyl cellulose, carboxymethyl methyl cellulose, sulfoethyl methyl hydroxyethyl cellulose, sulfoethyl methyl cellulose, carboxymethyl ethyl hydroxyethyl cellulose, carboxymethyl ethyl cellulose, sulfoethyl ethyl hydroxyethyl cellulose, sulfoethyl ethyl cellulose, carboxymethyl methyl hydroxypropyl cellulose, sulfoethyl methyl hydroxypropyl cellulose, carboxymethyl dodecyl cellulose, carboxymethyl dodecoyl cellulose, carboxymethyl cyanoethyl cellulose, and sulfoethyl cyanoethyl cellulose. The cellulose may be a substituted cellulose substituted by two or more different substituents, such as methyl and hydroxyethyl cellulose.

The composition may comprise soil release polymers, such as Repel-o-Text™. Other suitable soil release polymers are anionic soil release polymers. Suitable soil release polymers are described in more detail in WO05123835A1, WO07079850A1 and WO08110318A2.

The composition may comprise a spray-dried powder. The spray-dried powder may comprise a silicate salt, such as sodium silicate.

Adjunct Materials

Suitable adjunct materials include, but are not limited to, surfactants, builders, chelating agents, dye transfer inhibiting agents, dispersants, enzymes, and enzyme stabilizers, catalytic materials, bleach activators, polymeric dispersing agents, clay soil removal/anti-redeposition agents, brighteners, suds suppressors, dyes, additional perfume and perfume delivery systems, structure elasticizing agents, fabric softeners, carriers, hydrotropes, processing aids and/or pigments. In addition to the disclosure below, suitable examples of such other adjuncts and levels of use are found in U.S. Patent Nos. 5,576,282, 6,306,812 B1 and 6,326,348 B1 that are incorporated by reference.

As stated, the adjunct ingredients are not essential to Applicants' cleaning and fabric care compositions. Thus, certain embodiments of Applicants' compositions do not contain one or more of the following adjunct materials: bleach activators, surfactants, builders, chelating agents, dye transfer inhibiting agents, dispersants, enzymes, and enzyme stabilizers, catalytic metal complexes, polymeric dispersing agents, clay and soil removal/anti-redeposition agents, brighteners, suds suppressors, dyes, additional perfumes and perfume delivery systems, structure elasticizing agents, fabric softeners, carriers, hydrotropes, processing aids and/or pigments. However, when one or more adjuncts is present, such one or more adjuncts may be present as detailed below:

Surfactants - The compositions according to the present invention can comprise a surfactant or surfactant system wherein the surfactant can be selected from nonionic and/or anionic and/or cationic surfactants and/or ampholytic and/or zwitterionic and/or semi-polar nonionic surfactants. The surfactant is typically present at a level of from about 0.1%, from about 1%, or even from about 5% by weight of the cleaning compositions to about 99.9%, to about 80%, to about 35%, or even to about 30% by weight of the cleaning compositions.

Builders - The compositions of the present invention can comprise one or more detergent builders or builder systems. When present, the compositions will typically comprise at least about 1% builder, or from about 5% or 10% to about 80%, 50%, or even 30% by weight, of said builder. Builders include, but are not limited to, the alkali metal, ammonium and alkanolammonium salts of polyphosphates, alkali metal silicates, alkaline earth and alkali metal carbonates, aluminosilicate builders polycarboxylate compounds, ether hydroxypolycarboxylates, copolymers of maleic anhydride with ethylene or vinyl methyl ether, 1,3,5-trihydroxybenzene-2,4,6-trisulphonic acid, and carboxymethyl-oxysuccinic acid, the various alkali metal, ammonium and substituted ammonium salts of polyacetic acids such as ethylenediamine tetraacetic acid and nitrilotriacetic acid, as well as polycarboxylates such as mellitic acid, succinic acid, oxydisuccinic acid, polymaleic acid, benzene 1,3,5-tricarboxylic acid, carboxymethyloxysuccinic acid, and soluble salts thereof.

Chelating Agents - The compositions herein may also optionally contain one or more copper, iron and/or manganese chelating agents. If utilized, chelating agents will generally comprise from about 0.1% by weight of the compositions herein to about 15%, or even from about 3.0% to about 15% by weight of the compositions herein.

Dye Transfer Inhibiting Agents - The compositions of the present invention may also include one or more dye transfer inhibiting agents. Suitable polymeric dye transfer inhibiting agents include, but are not limited to, polyvinylpyrrolidone polymers, polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylloxazolidones and polyvinylimidazoles or mixtures thereof. When present in the compositions herein, the dye transfer inhibiting agents are present at levels from about 0.0001%, from about 0.01%, from about 0.05% by weight of the cleaning compositions to about 10%, about 2%, or even about 1% by weight of the cleaning compositions.

Dispersants - The compositions of the present invention can also contain dispersants. Suitable water-soluble organic materials are the homo- or co-polymeric acids or their salts, in which the polycarboxylic acid may comprise at least two carboxyl radicals separated from each other by not more than two carbon atoms.

Enzymes - The compositions can comprise one or more detergent enzymes which provide cleaning performance and/or fabric care benefits. Examples of suitable enzymes include, but are not limited to, hemicellulases, peroxidases, proteases, other cellulases, other xylanases, lipases, phospholipases, esterases, cutinases, pectinases, keratanases, reductases, oxidases, phenoloxidases, lipoxygenases, ligninases, pullulanases, tannases, pentosanases, malanases, β -glucanases, arabinosidases, hyaluronidase, chondroitinase, laccase, and amylases, or mixtures thereof. A typical combination is a cocktail of conventional applicable enzymes like protease, lipase, cutinase and/or cellulase in conjunction with amylase.

Enzyme Stabilizers - Enzymes for use in compositions, for example, detergents can be stabilized by various techniques. The enzymes employed herein can be stabilized by the presence of water-soluble sources of calcium and/or magnesium ions in the finished compositions that provide such ions to the enzymes.

Catalytic Metal Complexes - Applicants' compositions may include catalytic metal complexes. One type of metal-containing bleach catalyst is a catalyst system comprising a transition metal cation of defined bleach catalytic activity, such as copper, iron, titanium, ruthenium, tungsten, molybdenum, or manganese cations, an auxiliary metal cation having little or no bleach catalytic activity, such as zinc or aluminum cations, and a sequesterant having defined stability constants for the catalytic and auxiliary metal cations, particularly ethylenediaminetetraacetic acid, ethylenediaminetetra (methyl-enephosphonic acid) and water-soluble salts thereof. Such catalysts are disclosed in U.S. patent 4,430,243.

If desired, the compositions herein can be catalyzed by means of a manganese compound. Such compounds and levels of use are well known in the art and include, for example, the manganese-based catalysts disclosed in U.S. patent 5,576,282.

Cobalt bleach catalysts useful herein are known, and are described, for example, in U.S. patents 5,597,936 and 5,595,967. Such cobalt catalysts are readily prepared by known procedures, such as taught for example in U.S. patents 5,597,936, and 5,595,967.

Compositions herein may also suitably include a transition metal complex of a macropolycyclic rigid ligand - abbreviated as "MRL". As a practical matter, and not by way of limitation, the compositions and cleaning processes herein can be adjusted to provide on the order of at least one part per hundred million of the benefit agent MRL species in the aqueous washing medium, and may provide from about 0.005 ppm to about 25 ppm, from about 0.05 ppm to about 10 ppm, or even from about 0.1 ppm to about 5 ppm, of the MRL in the wash liquor.

Preferred transition-metals in the instant transition-metal bleach catalyst include manganese, iron and chromium. Preferred MRL's herein are a special type of ultra-rigid ligand that is cross-bridged such as 5,12-diethyl-1,5,8,12-tetraazabicyclo[6.6.2]hexa-decane.

Suitable transition metal MRLs are readily prepared by known procedures, such as taught for example in WO 00/32601, and U.S. patent 6,225,464.

Processes of Making and Using Compositions

The compositions of the present invention can be formulated into any suitable form and prepared by any process chosen by the formulator, non-limiting examples of which are described
5 in U.S. 5,879,584; U.S. 5,691,297; U.S. 5,574,005; U.S. 5,569,645; U.S. 5,565,422; U.S. 5,516,448; U.S. 5,489,392; U.S. 5,486,303.

TEST METHODS

It is understood that the test methods that are disclosed in the Test Methods Section of the
10 present application must be used to determine the respective values of the parameters of Applicants' invention as such invention is described and claimed herein.

(1) Particle size distribution

a.) Place 1 gram of particles in 1 liter of distilled deionized (DI) water.

- b.) Permit the particles to remain in the DI water for 10 minutes and then recover the particles by filtration.
- c.) Determine the particle size distribution of the particle sample by measuring the particle size of 50 individual particles using the experimental apparatus and method of Zhang, Z.; Sun, G; "Mechanical Properties of Melamine-Formaldehyde microcapsules," J. Microencapsulation, vol 18, no. 5, pages 593-602, 2001.
- d.) Average the 50 independent particle diameter measurements to obtain an average particle diameter.
- e.) Use the 50 independent measurements to calculate a standard deviation of particle size using the following equation:

$$\mu = \sqrt{\frac{\sum (d - s)^2}{n - 1}}$$

where

- μ is the standard deviation
- s is the average particle diameter
- d is the independent particle diameter
- n is the total number of particles whose diameter is measured.

(2) Benefit Agent Retention Ratio

- a.) Add 1 gram of particle to 99 grams of composition that the particle will be employed in.
- b.) Age the particle containing composition of a.) above for 2 weeks at 40°C in a sealed, glass jar.
- c.) Recover the particles from b.) above by filtration.
- d.) Treat the particles of c.) above with a solvent that will extract all the benefit agent from the particles.
- e.) Inject the benefit agent containing solvent from d.) above into a Gas Chromatograph and integrate the peak areas to determine the total quantity of benefit agent extracted from the particle sample.

f.) This quantity is then divided by the quantity that would be present if nothing had leaked out of the microcapsule (e.g. the total quantity of core material that is dosed into the composition via the microcapsules). This value is then multiplied by the ratio of average particle diameter to average particle thickness to obtain a Benefit Agent Retention Ratio.

A detailed analytical procedure to measure the Benefit Agent Retention Ratio is:

ISTD Solution

1. Weigh out 25mg dodecane into a weigh boat.
2. Rinse the dodecane into a 1000mL volumetric flask using ethanol.
3. Add ethanol to volume mark.
4. Stir solution until mixed. This solution is stable for 2 months.

Calibration Standard

1. Weigh out 75mg of core material into a 100 mL volumetric flask.
2. Dilute to volume with ISTD solution to from above. This standard solution is stable for 2 months.
3. Mix well.
4. Analyze via GC/FID.

Basic Sample Prep

(Prepare samples in triplicate)

1. Weigh 1.000 gram sample of aged composition containing particles into a 100 mL tri-pour beaker. Record weight. .
2. Add 4 drops (approximately 0.1 gram) 2-ethyl-1,3-Hexanediol into the tri-pour beaker.
3. Add 50 mL Deionized water to the beaker. Stir for 1 minute.
4. Using a 60cc syringe, filter through a Millipore Nitrocellulose Filter Membrane (1.2 micron, 25 mm diameter).
5. Rinse through the filter with 10 mL of Hexane
6. Carefully remove the filter membrane and transfer to a 20 mL scintillation vial (using tweezers).
7. Add 10mL ISTD solution (as prepared above) to the scintillation vial containing the filter.

8. Cap tightly, mix, and heat vial at 60°C for 30min.
9. Cool to room temperature.
10. Remove 1mL and filter through a 0.45-micron PTFE syringe filter into GC vial. Several PTFE filters may be required to filter a 1mL sample aliquot.
- 5 11. Analyze via GC/FID.

GC/FID Analysis Method:

Column – 30m X 0.25mm id, 1-um DB-1 phase

GC – 6890 GC equipped with EPC control and constant flow capability

10 Method – 50°C, 1min. hold, temperature ramp of 4°C/min. to 300°C, and hold for 10min.

Injector – 1uL splitless injection at 240°C

GC/FID Analysis Method - Microbore Column Method:

Column – 20m X 0.1mm id, 0.1µm DB-5

15 GC – 6890 GC equipped with EPC control and constant flow capability (constant flow 0.4mL/min)

Method - 50°C, no hold, temperature ramp of 16°C/min to 275°C, and hold for 3min.

Injector - 1µL split injection (80:1 split) at 250°C

20 Calculations:

$$\% \text{ Total Perfume} = \frac{A_{IS} \times W_{per-std} \times A_{per-sam}}{A_{per-std} \times A_{IS-sam} \times W_{sam}} \times 100\%$$

where

25 A_{is} = Area of internal standard in the core material calibration standard;

$W_{per-std}$ = weight of core material in the calibration sample

$A_{per-sam}$ = Area of core material peaks in the composition containing particle sample;

$A_{per-std}$ = Area of core material peaks in the calibration sample.

30 A_{is-sam} = Area of internal standard in composition containing particle sample;

W_{sam} = Weight of the composition containing particle sample

$$\text{Retention_Ratio} = \left(\frac{\text{Total_Perfume}}{\text{Perfume_Dosed_Into_Product_Via_Microcapsules}} \right) \left(\frac{\mu}{T} \right)$$

35 where

μ is the average particle diameter, from Test Method 1
 T is the average particle thickness as calculated from Test Method 3

(3) **Fracture Strength**

- 5 a.) Place 1 gram of particles in 1 liter of distilled deionized (DI) water.
- b.) Permit the particles to remain in the DI water for 10 minutes and then recover the particles by filtration.
- c.) Determine the average rupture force of the particles by averaging the rupture force of 50 individual particles. The rupture force of a particle is determined using the procedure
 10 given in Zhang, Z.; Sun, G; "Mechanical Properties of Melamine-Formaldehyde microcapsules," J. Microencapsulation, vol 18, no. 5, pages 593-602, 2001. Then calculate the average fracture pressure by dividing the average rupture force (in Newtons) by the average cross-sectional area (as determined by Test Method 1 above) of the spherical particle (πr^2 , where r is the radius of the particle before compression).
- 15 d.) Calculate the average fracture strength by using the following equation:

$$\sigma_{fracture_stress} = \frac{P}{4(d/T)}$$

where

- 20 P is the average fracture pressure from a.) above
- d is the average diameter of the particle (as determined by Test Method 1 above)
- T is the average shell thickness of the particle shell as determined by the following equation:

$$25 \quad T = \frac{r_{capsule}(1-c)\rho_{perfume}}{3[c\rho_{wall} + (1-c)\rho_{perfume}]}$$

where

- c is the average perfume content in the particle
- r is the average particle radius
- 30 ρ_{wall} is the average density of the shell as determined by ASTM method B923-02, "Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry", ASTM International.

$\rho_{perfume}$ is the average density of the perfume as determined by ASTM method D1480-93(1997) "Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer", ASTM International.

5

(4) **ClogP**

The "calculated logP" (ClogP) is determined by the fragment approach of Hansch and Leo (cf., A. Leo, in Comprehensive Medicinal Chemistry, Vol. 4, C. Hansch, P.G. Sammens, J.B. Taylor, and C.A. Ramsden, Eds. P. 295, Pergamon Press, 1990, incorporated herein by reference). ClogP values may be calculated by using the "CLOGP" program available from Daylight Chemical Information Systems Inc. of Irvine, California U.S.A..

10

(5) **Boiling Point**

Boiling point is measured by ASTM method D2887-04a, "Standard Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography," ASTM International.

15

(6) **Delivery Index Calculation**

The Delivery Index for a particle is calculated using the following equation:

20

$$Delivery_Index = \frac{\left[\left(\frac{\mu}{\sigma} \right)_{Particle_Size} \left(\frac{f_0}{f} \right)_{Fracture_Stress} \left(\frac{L/L_0}{t/\mu} \right) \right]}{100}$$

25

Where

μ is the average particle diameter

σ is the standard deviation of the average particle diameter

f_0 is the minimum in-use fracture strength required to break the microcapsule

30

f is the measured Fracture Strength

$(L/L_0)/(t/\mu)$ is the Benefit Agent Retention Ratio

t is the shell thickness of the particle

EXAMPLESExamples 1-8

Liquid laundry detergent compositions suitable for front-loading automatic washing machines.

Ingredient	Composition (wt% of composition)							
	1	2	3	4	5	6	7	8
Alkylbenzene sulfonic acid	7	11	4.5	1.2	1.5	12.5	5.2	4
Sodium C ₁₂₋₁₄ alkyl ethoxy 3 sulfate	2.3	3.5	4.5	4.5	7	18	1.8	2
C ₁₄₋₁₅ alkyl 8-ethoxylate	5	8	2.5	2.6	4.5	4	3.7	2
C ₁₂ alkyl dimethyl amine oxide	-	-	0.2	-	-	-	-	-
C ₁₂₋₁₄ alkyl hydroxyethyl dimethyl ammonium chloride	-	-	-	0.5	-	-	-	-
C ₁₂₋₁₈ Fatty acid	2.6	4	4	2.6	2.8	11	2.6	1.5
Citric acid	2.6	3	1.5	2	2.5	3.5	2.6	2
Protease (Purafect® Prime)	0.5	0.7	0.6	0.3	0.5	2	0.5	0.6
Amylase (Natalase®)	0.1	0.2	0.15	-	0.05	0.5	0.1	0.2
Mannanase (Mannaway®)	0.05	0.1	0.05	-	-	0.1	0.04	-
Xyloglucanase XYG1006* (mg aep/100g detergent)	1	4	3	3	2	8	2.5	4
Random graft co-polymer ¹	1	0.2	1	0.4	0.5	2.7	0.3	1
A compound having the following general structure: bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n)(CH ₃)-N ⁺ -C _x H _{2x} -N ⁺ -(CH ₃)-bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n), wherein n = from 20 to 30, and x = from 3 to 8, or sulphated or sulphonated variants thereof	0.4	2	0.4	0.6	1.5	1.8	0.7	0.3
Ethoxylated Polyethylenimine ²	-	-	-	-	-	0.5	-	-
Amphiphilic alkoxyated grease cleaning polymer ³	0.1	0.2	0.1	0.2	0.3	0.3	0.2	0.3
Diethoxylated poly (1,2 propylene terephthalate short block soil release polymer.	-	-	-	-	-	-	0.3	-
Diethylenetriaminepenta(methylene phosphonic) acid	0.2	0.3	-	-	0.2	-	0.2	0.3
Hydroxyethane diphosphonic acid	-	-	0.45	-	-	1.5	-	0.1
FWA	0.1	0.2	0.1	-	-	0.2	0.05	0.1
Solvents (1,2 propanediol, ethanol), stabilizers	3	4	1.5	1.5	2	4.3	2	1.5
Hydrogenated castor oil derivative structurant	0.4	0.4	0.3	0.1	0.3	-	0.4	0.5
Boric acid	1.5	2.5	2	1.5	1.5	0.5	1.5	1.5
Na formate	-	-	-	1	-	-	-	-
Reversible protease inhibitor ⁴	-	-	0.002	-	-	-	-	-
Perfume	0.5	0.7	0.5	0.5	0.8	1.5	0.5	0.8
Perfume MicroCapsules slurry (30%am)	0.2	0.3	0.7	0.2	0.05	0.4	0.9	0.7
Ethoxylated thiophene Hueing Dye							0.007	0.008
Buffers (sodium hydroxide, Monoethanolamine)	To pH 8.2							
Water and minors (antifoam, aesthetics)	To 100%							

Examples 9-16

Liquid laundry detergent compositions suitable for top-loading automatic washing machines.

Ingredient	Composition (wt% of composition)							
	9	10	11	12	13	14	15	16
C ₁₂₋₁₅ Alkylethoxy(1.8)sulfate	20.1	15.1	20.0	15.1	13.7	16.7	10.0	9.9
C _{11.8} Alkylbenzene sulfonate	2.7	2.0	1.0	2.0	5.5	5.6	3.0	3.9
C ₁₆₋₁₇ Branched alkyl sulfate	6.5	4.9		4.9	3.0	9.0	2.0	
C ₁₂₋₁₄ Alkyl -9-ethoxylate	0.8	0.8	0.8	0.8	8.0	1.5	0.3	11.5
C ₁₂ dimethylamine oxide			0.9					
Citric acid	3.8	3.8	3.8	3.8	3.5	3.5	2.0	2.1
C ₁₂₋₁₈ fatty acid	2.0	1.5	2.0	1.5	4.5	2.3		0.9
Protease (Purafect® Prime)	1.5	1.5	0.5	1.5	1.0	1.8	0.5	0.5
Amylase (Natalase®)	0.3	0.3	0.3	0.3	0.2	0.4		
Amylase (Stainzyme®)								1.1
Mannanase (Mannaway®)	0.1					0.1		
Pectate Lyase (Pectawash®)	0.1					0.2		
Xyloglucanase XYGI006* (mg aep/100g detergent)	5	13	2	5	20	1	2	3
Borax	3.0	3.0			2.0	3.0	3.0	3.3
Na & Ca formate	0.2	0.2		0.2	0.2		0.7	
A compound having the following general structure: bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n)(CH ₃)-N ⁺ -C _x H _{2x} -N ⁺ -(CH ₃)-bis((C ₂ H ₅ O)(C ₂ H ₄ O) _n), wherein n = from 20 to 30, and x = from 3 to 8, or sulphated or sulphonated variants thereof	1.6	1.6	3.0	1.6	2.0	1.6	1.3	1.2
Random graft co-polymer ¹	0.4	0.2	1.0	0.5	0.6	1.0	0.8	1.0
Diethylene triamine pentaacetic acid	0.4	0.4	0.4	0.4	0.2	0.3	0.8	
Tinopal AMS-GX	0.2	0.2	0.2	0.2	0.2	0.3	0.1	
Tinopal CBS-X						0.1		0.2
Amphiphilic alkoxyated grease cleaning polymer ³	1.0	1.3	1.3	1.4	1.0	1.1	1.0	1.0
Texcare 240N (Clariant)				1.0				
Ethanol	2.6	2.6	2.6	2.6	1.8	3.0	1.3	
Propylene Glycol	4.6	4.6	4.6	4.6	3.0	4.0	2.5	
Diethylene glycol	3.0	3.0	3.0	3.0	3.0	2.7	3.6	
Polyethylene glycol	0.2	0.2	0.2	0.2	0.1	0.3	0.1	1.4
Monoethanolamine	2.7	2.7	2.7	2.7	4.7	3.3	1.7	0.4
Triethanolamine								0.9
NaOH	to pH 8.3	to pH 8.3	to pH 8.3	to pH 8.3	to pH 8.3	to pH 8.3	to pH 8.3	to pH 8.5
Suds suppressor								
Dye	0.01	0.01	0.01		0.01	0.01	0.01	0.0
Perfume	0.5	0.5	0.5	0.5	0.7	0.7	0.8	0.6
Perfume MicroCapsules slurry (30%am)	0.2	0.5	0.2	0.3	0.1	0.3	0.9	1.0

Ethoxylated thiophene Hueing Dye					0.002	0.004		
Water	balance	balance	balance	balance	balance	balance	balance	Balance

Examples 17-22

- The following are granular detergent compositions produced in accordance with the invention suitable for laundering fabrics.

	17	18	19	20	21	22
Linear alkylbenzenesulfonate with aliphatic carbon chain length C ₁₁ -C ₁₂	15	12	20	10	12	13
Other surfactants	1.6	1.2	1.9	3.2	0.5	1.2
Phosphate builder(s)	2	25	4	3	2	
Zeolite		1		1	4	1
Silicate	4	5	2	3	3	5
Sodium Carbonate	9	20	10	17	5	23
Polyacrylate (MW 4500)	1	0.6	1	1	1.5	1
Carboxymethyl cellulose (Finnfix BDA ex CPKelco)	1	-	0.3	-	1.1	-
Xyloglucanase XYG1006* (mg aep/100g detergent)	1.5	2.4	1.7	0.9	5.3	2.3
Other enzymes powders	0.23	0.17	0.5	0.2	0.2	0.6
Fluorescent Brightener(s)	0.16	0.06	0.16	0.18	0.16	0.16
Diethylenetriamine pentaacetic acid or Ethylene diamine tetraacetic acid	0.6		0.6	0.25	0.6	0.6
MgSO ₄	1	1	1	0.5	1	1
Bleach(es) and Bleach activator(s)	6.88		6.12	2.09	1.17	4.66
Perfume MicroCapsules	0.2	0.5	0.2	0.3	0.2	0.1
Sulfate/Moisture/perfume	Balance to 100%					

Examples 23-28

- The following are granular detergent compositions produced in accordance with the invention suitable for laundering fabrics.

	23	24	25	26	27	28
Linear alkylbenzenesulfonate with aliphatic carbon chain length C ₁₁ -C ₁₂	8	7.1	7	6.5	7.5	7.5
Other surfactants	2.95	5.74	4.18	6.18	4	4
Layered silicate	2.0	-	2.0	-	-	-
Zeolite	7	-	2	-	2	2
Citric Acid	3	5	3	4	2.5	3
Sodium Carbonate	15	20	14	20	23	23
Silicate	0.08	-	0.11	-	-	-

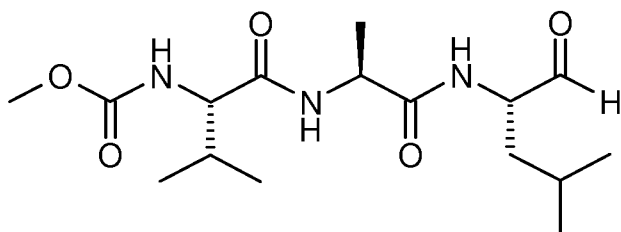
Soil release agent	0.75	0.72	0.71	0.72	-	-
Acrylic Acid/Maleic Acid Copolymer	1.1	3.7	1.0	3.7	2.6	3.8
Carboxymethyl cellulose (Finnfix BDA ex CPKelco)	0.15	-	0.2	-	1	-
Xyloglucanase XYG1006* (mg aep/100g detergent)	3.1	2.34	3.12	4.68	3.52	7.52
Other enzyme powders	0.65	0.75	0.7	0.27	0.47	0.48
Bleach(es) and bleach activator(s)	16.6	17.2	16.6	17.2	18.2	15.4
Perfume MicroCapsules	0.05	0.1	0.21	0.06	0.22	0.3
Sulfate/ Water & Miscellaneous	Balance to 100%					

¹ Random graft copolymer is a polyvinyl acetate grafted polyethylene oxide copolymer having a polyethylene oxide backbone and multiple polyvinyl acetate side chains. The molecular weight of the polyethylene oxide backbone is about 6000 and the weight ratio of the polyethylene oxide to polyvinyl acetate is about 40 to 60 and no more than 1 grafting point per 50 ethylene oxide units.

² Polyethylenimine (MW = 600) with 20 ethoxylate groups per -NH.

³ Amphiphilic alkoxyated grease cleaning polymer is a polyethylenimine (MW = 600) with 24 ethoxylate groups per -NH and 16 propoxylate groups per -NH

⁴ Reversible Protease inhibitor of structure:



* Remark: all enzyme levels expressed as % enzyme raw material, except for xyloglucanase where the level is given in mg active enzyme protein per 100g of detergent. XYG1006 enzyme is according to SEQ ID: 1.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as “40 mm” is intended to mean “about 40 mm”.

CLAIMS

What is claimed is:

1. A laundry detergent composition comprising:
 - (a) a glycosyl hydrolase having enzymatic activity towards both xyloglucan and amorphous cellulose substrates, wherein the glycosyl hydrolase is selected from GH families 5, 12, 44 or 74
 - (b) a particle comprising a core material and a wall material that surrounds the core material, said particle having a Delivery Index of at least about 0.05 said composition being a consumer product; and
 - (c) deterative surfactant.
2. A composition according to claim 1, wherein the glycosyl hydrolase enzyme belongs to glycosyl hydrolase family 44.
3. A composition according to any preceding claim, wherein the glycosyl hydrolase enzyme
5 has a sequence at least 80% homologous to sequence ID No. 1.
4. A composition according to any preceding claim, wherein the composition is in the form of a liquid.
5. A composition according to any preceding claim, wherein said composition has a Delivery Index of at least 7.
6. A composition according any preceding claim, wherein said particle's core material comprises a material selected from the group consisting of perfume, silicone oils, waxes, hydrocarbons, higher fatty acids, essential oils, lipids, skin coolants, vitamins, sunscreens, antioxidants, glycerine, catalysts, bleach particles, silicon dioxide particles, malodor reducing agents, dyes, brighteners, antibacterial actives, antiperspirant actives, cationic polymers and mixtures thereof.

7. A composition according any preceding claim, wherein said particle's wall material comprises a material selected from the group consisting of polyamine, polyurea, polyurethane polysaccharides and modified polysaccharides, gel forming proteins, modified celluloses, carboxylic acid containing acrylic polymers, gelatin, gum arabic, urea crosslinked with formaldehyde, urea crosslinked with gluteraldehyde, melamine crosslinked with formaldehyde, chitin and chitosan and modified chitin and modified chitosan, sodium alginate, latexes, silicon dioxide, sodium silicates and mixtures thereof.
8. A composition according to any preceding claim, wherein said particle comprises at least 1 weight % of a benefit agent.
9. A composition according to any preceding claim, wherein said particle comprises from about 20 to about 95 weight% of a benefit agent.
10. A composition according to any preceding claim, wherein said particle's core material comprises, based on total core material weight, at least about 20 wt% benefit agent.
11. A composition according to any preceding claim, wherein said benefit agent comprises a perfume composition, said particle comprising, based on total particle weight, from about 20 weight % to about 95 weight % of said perfume composition.
12. A composition according to any preceding claim, wherein said perfume composition comprises a Quadrant III perfume raw material.
13. A composition according to any preceding claim, wherein said composition comprises, based on total composition weight, from about 0.2 to about 10 weight % of said particle.
14. A composition according to any preceding claim, wherein the composition comprises a material selected from the group consisting of calcium formate, formic acid, polyamines and mixtures thereof.

15. A method of treating and/or cleaning a situs, said method comprising
 - a.) optionally washing and/or rinsing said situs;
 - b.) contacting said situs with a composition according to any preceding Claim; and
 - c.) optionally washing and/or rinsing said situs.

16. A situs treated with a composition according to any preceding claim.

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2008/055470

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D3/386 C11D3/50 C11D17/00

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.
A	WO 02/12462 A (GENENCOR INT) 14 February 2002 (2002-02-14) page 14, paragraph 2; claims -----	1-16
A	WO 2007/057418 A (NOVOZYMES AS) 24 May 2007 (2007-05-24) claims -----	1-16
A	US 2003/032162 A1 (SCHNORR KIRK ET AL) 13 February 2003 (2003-02-13) claims -----	1-16
A	WO 2007/118935 A (ENZYMES OY AB) 25 October 2007 (2007-10-25) claims -----	1-16
	-/--	

Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents :

A document defining the general state of the art which is not considered to be of particular relevance	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
E earlier document but published on or after the international filing date	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
O document referring to an oral disclosure, use, exhibition or other means	*G* document member of the same patent family
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 13 March 2009	Date of mailing of the international search report 02/04/2009
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Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Hillebrecht, Dieter
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INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2008/055470

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2007/135320 A1 (MELI FABRIZIO ET AL) 14 June 2007 (2007-06-14) claims	1-16
A	WO 01/62903 A (NOVOZYMES AS) 30 August 2001 (2001-08-30) page 47, line 10; claim 1	1-16

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/IB2008/055470

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			EP 1259594	A1	27-11-2002