

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
24 August 2006 (24.08.2006)

PCT

(10) International Publication Number
WO 2006/087090 A1

(51) International Patent Classification:

A23D 9/05 (2006.01) A23L 1/40 (2006.01)
A23L 1/00 (2006.01)

(21) International Application Number:

PCT/EP2006/000798

(22) International Filing Date: 25 January 2006 (25.01.2006)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

05075391.2	17 February 2005 (17.02.2005)	EP
05075393.8	17 February 2005 (17.02.2005)	EP
05075384.7	17 February 2005 (17.02.2005)	EP
05075392.0	17 February 2005 (17.02.2005)	EP

(71) Applicant (for AL, AM, AT, AZ, BA, BE, BF, BG, BJ, BR, BY, CF, CG, CH, CI, CM, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, FR, GA, GE, GN, GQ, GR, GW, HR, HU, ID, IS, IT, JP, KG, KM, KP, KR, LR, LT, LU, LV, MA, MC, MD, MG, MK, ML, MR, MX, MZ, NE, NI, NL, NO, PH, PL, PT, RO, RU, SE, SI, SK, SM, SN, SY, TD, TG, TJ, TM, TN, TR, UA, UZ, VN, YU only): **UNILEVER N.V.** [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).

(71) Applicant (for AE, AG, AU, BB, BW, BZ, CA, CY, EG, GB, GD, GH, GM, IE, IL, KE, KN, LC, LK, LS, LY, MN, MW, NA, NG, NZ, OM, PG, SC, SD, SG, SL, SZ, TT, TZ, UG, VC, ZA, ZM, ZW only): **UNILEVER PLC** [GB/GB]; Unilever House, Blackfriars, London, Greater London EC4P 4BQ (GB).

(71) Applicant (for IN only): **HINDUSTAN LEVER LIMITED** [IN/IN]; Hindustan Lever ouseE, 165/166 Backbay Reclamation, Maharashtra, Mumbai 400 020 (IN).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **GARBOLINO, Chiara** [IT/NL]; UNILEVER R & D VLAARDINGEN, Olivier Van Noortlaan 120, NL-3133 AT Vlaardingen (NL). **HUIZINGA, Hindrik** [NL/NL]; UNILEVER R & D VLAARDINGEN, Olivier Van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(74) Agent: **JOPPE, Hermina, Laura, Petron**; UNILEVER PATENT GROUP, Olivier Van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: GRANULES COMPRISING STEROL

(57) Abstract: Granule comprising lipid powder particles having a microporous structure and comprising sterol.



WO 2006/087090 A1

Granules comprising sterol**Field of the invention**

The invention relates to granules comprising lipid powder. The invention further relates to use of these granules in the preparation of edible dispersions and/or tablets.

Background to the invention

Powdered lipids, lipid granules, lipid cubes and lipid tablets are known. However these known products cannot be used to structure an oil phase in a food product comprising an oil phase, in the same way as such a product is structured by lipid crystallized from the melt, such as in a votator process for the preparation of margarine.

There is also a need for particular formulations (e.g. concentrates) for soups, sauces, boullions, seasonings which particulate formulations can disperse quickly in an aqueous liquid and/or in a food composition. There is also a need for a process for manufacturing seasonings and particular formulations for preparing boullions, broth, soups, which are in the form of cubes, tablets, etcetera. Usually such cubes or tablets are prepared using a process involving a compression step. Prior to such compression step, the ingredients are usually mixed. The ingredients are usually a combination of dry particulate ingredients (salt, MSG, herbs, spices, starch and starch derivatives) which typically 1-30 wt.% fat. Mixing such dry ingredients with fat can be cumbersome, e.g. involving melting of the fat, maturation times, and other inconveniences. Hence there is a need for an easy way of manufacturing such

seasonings and concentrates for bouillons, broth, soups sauces in cube or tablet form.

Co-pending application PCT/EP2004/006544 describes edible dispersions, including water-in-oil emulsions, that comprise a structuring agent, specifically fat, having a microporous structure of submicron size particles. The water-in-oil emulsion may be a table spread. In the examples pourable emulsions and pourable dispersions were described. The edible dispersions may for instance be prepared by mixing an oil phase with structuring agent particles with a separately prepared water phase.

Summary of the invention

One or more of the above problems are solved according to the invention which provides a granule comprising a liquid and lipid powder particles (herein described as secondary particles) that have a microporous structure and which are agglomerates of primary particles of submicron size.

We have found that micronised fat particles such as used in PCT/EP2004/006544 can become airborne and can be described as dusty. When using the micronised fat powder to produce food products, this dustiness influences the handling properties of the powder negatively. Additionally, we have found that micronised fat powder has a very low bulk density. This creates the problem that a certain mass of powder takes up a huge space if it is transported.

The invention therefore relates to a granule comprising:-

- a) solid micronised lipid powder particles that have a microporous structure; and,
- b) a liquid;

wherein the granule is an agglomeration of said lipid particles herein described as secondary particles; and wherein said secondary particles are agglomerates of primary lipid particles, said primary lipid particles are platelets having an average thickness of 0.01-0.5 μm and wherein the lipid comprises sterol.

Detailed description of the invention

Granule is herein defined as an object prepared by size enlargement of secondary particles as described below.

The following terms are used herein to describe the properties of the granules according to the invention.

Although such particles are herein referred to as granules, and they can be prepared by granulation, such granule-particles can also be prepared by other known techniques for size enlargement of particulate material, as long as the desired particle size of the granules is obtained, and particles with the proper rigidity. Thus, although called "granules" herein, it does refer to all particles having a certain size (being larger than most components of the granules such as powdery material and/or crystalline material), and such granules can be prepared by granulation, but also by other techniques. Thus, for a range of conventional ingredients for e.g. bouillon cubes (salt, MSG, flour, starch, maltodextrin, etcetera) a size enlarging technique is needed, that binds a plurality of

such smaller particles (powdery materials such as flour and/or starch and/or crystalline materials such as salt, sugar, MSG) of the ingredients together to larger particles, herein referred to as granules. Suitable techniques for such size enlargement are known in the art and include granulation, agglomeration, pelletisation, sintering, briquetting or extruding followed by cutting the extrudates and other techniques as known in the art of size enlargement of particulate matter. The granules suitably have a diameter of between 0.5 and 10 mm (some fines may be allowed, at least 80 wt.% should have such size), preferably between 1 and 5 mm, more preferably 2-5 mm.

According to the invention, the granules are agglomerates of solid micronised lipid powder particles, herein described as secondary particles.

According to the invention, the solid structuring agent particles, described herein as secondary particles, should have a microporous structure of submicron size particles, which are described herein as primary particles.

The secondary particles are agglomerates of primary particles which have a microporous structure. The size of the primary particles is submicron (that is have a diameter below 1 μ).

Micronised fat (secondary particles) may be prepared herein as described in PCT/EP2004/006544.

An example of a microporous structure is shown in figures 6 and 7 of PCT/EP2004/006544. The primary particles typically have the shape as shown in figure 7, in which the platelets

with submicron dimensions are the primary particles. The thickness of the platelets should be submicron, preferably the thickness is on average 0.01-0.5 μm , more preferably 0.03-0.2 μm , even more preferably 0.06-0.12 μm .

Equivalent good results were obtained for a secondary particles having a microporous structure of more bubble-like shape, such as shown in figure 10 of PCT/EP2004/006544. In such microporous structure the wall thickness of the bubbles should be submicron, for instance on average 0.01-0.5 μm , more preferably 0.03-0.2 μm , even more preferably 0.06-0.12 μm .

The secondary particles, may, in the course of the preparation of the dispersion, for instance through the force of a mixer, be broken into submicron particles. The resulting submicron particles will form the structuring network of the dispersion.

Preferably, the structuring agent is edible lipid, more preferably it comprises edible fat.

In the context of the invention, the granule comprises a liquid. Preferably the liquid content of the granule is 40-60 wt.%. Preferably the liquid is edible oil or w/o-emulsions of edible oil.

In the context of this invention the lipid comprises sterol.

In a preferred embodiment, the solid micronised lipid particles comprise from 25 to 100 wt.%, preferably 80 to 100 wt.% sterol on total weight of the lipid particles.

In the context of the invention, the term sterol is used to indicate sterol, stanol, the fatty acid esters of sterol/stanol and mixtures of any of these.

The products according to the invention comprise a sterol and/or stanol or a fatty acid ester thereof, or mixtures thereof.

In this application where reference is made to sterolester, this also includes their saturated derivatives, the stanol esters, and combinations of sterol- and stanol esters.

Sterols or phytosterols, also known as plant sterols or vegetable sterols can be classified in three groups, 4-desmethylsterols, 4-monomethylsterols and 4,4'-dimethylsterols. In oils they mainly exist as free sterols and sterol esters of fatty acids although sterol glucosides and acylated sterol glucosides are also present. There are three major phytosterols namely beta-sitosterol, stigmasterol and campesterol. Schematic drawings of the components meant are as given in "Influence of Processing on Sterols of Edible Vegetable Oils", S.P. Kochhar; Prog. Lipid Res. 22: pp. 161-188.

The respective 5 alpha- saturated derivatives such as sitostanol, campestanol and ergostanol and their derivatives are in this specification referred to as stanols.

Preferably the (optionally esterified) sterol or stanol is selected from the group comprising fatty acid ester of β -sitosterol, β -sitostanol, campesterol, campestanol, stigmasterol, brassicasterol, brassicastanol or a mixture thereof.

The sterols or stanols are optionally at least partly esterified with a fatty acid. Preferably the sterols or stanols are esterified with one or more C_{2-22} fatty acids. For the purpose of the invention the term C_{2-22} fatty acid refers to any molecule comprising a C_{2-22} main chain and at least one acid group. Although not preferred within the present context the C_{2-22} main chain may be partially substituted or side chains may be present. Preferably, however the C_{2-22} fatty acids are linear molecules comprising one or two acid group(s) as end group(s). Most preferred are linear C_{8-22} fatty acids as these occur in natural oils.

Suitable examples of any such fatty acids are acetic acid, propionic acid, butyric acid, caproic acid, caprylic acid, capric acid. Other suitable acids are for example citric acid, lactic acid, oxalic acid and maleic acid. Most preferred are myristic acid, lauric acid, palmitic acid, stearic acid, arachidic acid, behenic acid, oleic acid, cetoleic acid, erucic acid, elaidic acid, linoleic acid and linolenic acid.

When desired a mixture of fatty acids may be used for esterification of the sterols or stanols. For example, it is possible to use a naturally occurring fat or oil as a source of the fatty acid and to carry out the esterification via an interesterification reaction.

The lipid further optionally comprises edible fats. Edible fats consist predominantly of triglycerides. Typically such edible fats suitable as structuring agent are mixtures of triglycerides, some of which have a melting point higher than room or ambient temperature and therefore contain solids in the form of crystals.

The solid structuring agent, also denoted as hardstock, may serve to structure the fat phase and may help to stabilise the dispersion.

For imparting to common margarine a semi-solid, plastic, spreadable consistency this stabilising and structuring functionality plays an important role. The crystals of the solid fat form a network throughout the liquid oil resulting into a structured fat phase. The aqueous phase droplets are fixed within the spaces of the lattice of solid fat crystals. In this way coalescence of the droplets and separation of the heavier aqueous phase from the fat phase is prevented.

According to the invention micronised lipid powder is subjected to agglomeration. This is done by spraying a sticky liquid on the micronised fat powder and gluing these secondary fat powder particles together to form granules. This sticky liquid should be a liquid that is able to wet the powder, but is strong enough to form a bridge between the secondary powder particles. Preferably the sticky liquid is edible oil or w/o-emulsions of edible oil.

The granulation technique is not critical, for instance the primary micronised fat powder may be granulated in

conventional granulation machines. Preferably the granulation is conducted as described above.

The granules according to the invention may be used as intermediate for making several fat containing products, such as cooking fats, spreads and liquid margarines. Also it is possible to use the powders as such in an end product or as powder to press tablets.

Surprisingly the granules may be used to stabilise an oil phase in a food product when used as an ingredient in conventional processes for preparing the food products. Though not wishing to be bound to theory and without limiting the scope of the invention an explanation could be that the granules, during the processing of the food product are broken-up by mechanical forces, and the fragments are able to stabilize an oil phase.

Therefore, the invention also relates to a process for the preparation of an edible dispersion comprising;

- a) an oil phase,
- b) a structuring agent; and,
- c) an aqueous phase and optionally a solid phase, in which the dispersion is formed by mixing oil, structuring agent and the aqueous phase and optionally additionally a solid phase;

wherein the structuring agent is a granule according to the present invention.

Further the granules may be used in a process for preparing bouillon or seasoning cubes, the process comprising the steps of:

- a) mixing 2-99% of particulate ingredients, comprising one or more of salt, flavour enhancers, spices, herbs, flavourants, sugar, yeast extract, or hydrolysed vegetable protein, with 1-98% of fat solid at 20°C in particulate form,
- b) pressing to cubes;
wherein the fat in particulate form comprises granules according to the present invention.

In the process as given above wide ranges of the amount of ingredients are given. The actual amount depends e.g. on the intended use. For bouillon (incl. broth and stock and the like) cubes the amount of fat is preferably 1-30%, more preferably 2-25%. For seasonings (incl. melting) cubes the amount of fat is preferably 10-99%, more preferably 20-70%. Cubes e.g. for soups and sauces can have fat contents of 5-60%, preferably 10-50%. All percentages are percentages by weight of the total cube. For very low amounts of fat (e.g. 1-5%) it may be needed to include ingredients of which it is known that they aid compressing into a tablet, like maltodextrin and/or moist ingredients.

The invention further relates to a particulate seasoning or concentrate for preparing a bouillon, broth, soup or sauce comprising 0.5-60 wt.% of fat and 40-99.5 wt.% of one or more of salt, flavour enhancers, starch, starch derivatives, sugar, herbs, spices, vegetable particulates, flavours, colourants, wherein the fat comprises granules according to the present invention.

Benefits of this invention can be separated in what we can define as customer benefits and consumer benefits. Customer

benefits are mainly present in intermediate products. These benefits reveal during storage and production of final products. Consumer benefits reveal in the final products. The benefits described here neglect the benefits of the use of micronised powders, which are numerous already.

Customer benefits:

- reduced dustiness of the powder,
- reduced volume of the powder, that is a higher bulk density,
- improved flow properties of the powder,
- possibility to store ingredients which enable instant production,
- improved dispersion properties if the powder is dispersed in oil.

Consumer benefits:

- lower SAFA-content of the powder product and the tablet if tablets are pressed from the powder,
- ability to build in water components, if w/o-emulsions are used to granule the powder,
- ability to build in components that are water or oil soluble, such as anti-spattering emulsifiers, etc.,
- ability to co-granule components that are solids, like olive-scrap, etc.,

Examples

General/methods

Bulk Density

The bulk density is measured by determining the volume of 100 g of material in a measuring cylinder and is expressed as kg/l and the gluten content is measured by AACC method 38-10 (9th Ed, 1995).

Dynamic flow rate

For the purposes of this invention, the flow properties of the granular product are defined in terms of the dynamic flow rate (DFR), in ml/s, measured by means of the following procedure. A cylindrical glass tube of internal diameter of 35 mm and length of 600 mm is securely clamped with its longitudinal axis in the vertical position. Its lower end is terminated by a cone of polyvinyl chloride having an internal angle of 15' and a lower outlet orifice of diameter 22.5 mm. A first beam sensor is positioned 150 mm above the outlet, and a second beam sensor is positioned 250 mm above the first sensor.

To determine the dynamic flow rate the outlet orifice is temporarily closed and the cylinder filled with the granular product to a point about 10 cm above the upper sensor. The outlet is opened and the flow time t (seconds) taken for the powder level to fall from the upper sensor to the lower sensor measured electronically. This is repeated 2 or 3 times and an average time taken. If V is the volume (ml) of the tube between the upper and lower sensors, the DFR is given by V/t .

Unconfined compressibility

The unconfined compressibility test (UCT) provides a measure of the cohesiveness or "stickiness" of a product and can provide a guide to its storage properties in, for

example, silos. The principle of the test is to compress the granular product into a compact and then measure the force required to break the compact. This is carried out using an apparatus comprising a cylinder of diameter 89 mm and height 114 mm (3.5 x 4.5 inches), a plunger and plastic discs and weights of predetermined weight as follows.

The cylinder, positioned around a fixed locating disc and secured with a clamp, is filled with granular product and the surface leveled by drawing a straight edge across it. A 50 g plastic disc is placed on top of the granular product, the plunger lowered and a 10 kg weight placed slowly on top of the upper plunger disc. The weight is left in position for 2 minutes after which time the 10 kg weight is removed and plunger raised. The clamp is removed from the cylinder and the two halves of the cylinder carefully removed to leave a compact of granular product. If the compact is unbroken, a second 50 g plastic disc is placed on top of the first and left for approximately ten seconds. If the compact is still unbroken, a 100 g disc is placed on top to the plastic discs and left for ten seconds. If the compact is still unbroken, the plunger is lowered very gently onto the discs and 250 g weights added at ten second intervals until the compact collapses. The total weight of plunger, plastic discs and weights at collapse is recorded.

The cohesiveness of the powder is classified by the weight required to break the compact as follows. The greater the weight required, the higher the UCT value and the more cohesive ("sticky") the powder.

As used herein, unless stated explicitly to the contrary, the term "fines", refers to particles with a diameter of less than 180 microns. Further, reference to "coarse" material, means particles with a diameter greater than 1400

microns. Levels of fine and coarse particles can be measured using sieve analysis.

Stevens value

Stevens values give an indication about the firmness of a product. The firmness of all products stored at 5°C for 24 hours was measured at room temperature using a Stevens Texture Analyser (1 mm/sec, 25 mm depth, 4.4. mm probe) and is quoted herein as the Stevens value (in g).

Example 1

To find the optimal liquids/solids ratio a set of experiments was done with micronised high erucic rapeseed hardened to 70°C (RPh070) and sunflower oil (SF). The set of experiments was done with varying liquids/solids ratios as can be seen in the table. The observations during and just after granulation are described in the same table.

Table 1: Result of tests for granulation conditions

<i>SF [g]</i>	<i>RPh070 [g]</i>	<i>Vol. fraction solids [%]</i>	<i>Observations</i>
20	30	40	Still very dusty powder
22.5	27.5	45	Dusty powder
25	25	50	Nice granule, but still some dustiness
27.5	22.5	55	Coarse granule, but still flowing
30	20	60	One fatty lump instead of powder

From these granulation experiments it became clear that the volume fraction of solids of 50% was optimal for further

experiments. However, after storage still some dustiness occurred.

Examples 2-4

Instant spreads (i.e. spreads that can be made using conventional home kitchen mixer) were prepared as follows.

Spreads were produced with a composition as in table 2. Stable spreads resulted. The water phase was made by mixing the salt in distilled water and holding the mixture at room temperature. The fat phase was produced by adding the granulated fat powder to liquid oil using a spatula. The water phase and the oil phase were mixed using a home kitchen mixer.

It was possible to prepare a stable spread without emulsifier and thickener. The long term stability and/or consistency of the spread without emulsifier may be improved by adding a thickener to the water phase, e.g. 1 wt.% starch, a suitable starch type is Resistamyl 310.

The solid ingredients (i.e. all ingredients except water and oil) may be packed together. At home these ingredients may be mixed with oil and water.

Table 2: Composition of instant spreads of examples 2-4

		Ex. 2	Ex. 3	Ex. 4
	Ingredient	Amount (wt.%)	Amount (wt.%)	Amount (wt.%)
Oil Phase		39.85	34.85	34.85
	Sunflower oil	29.49	25.79	25.79
	Granulated fat powder ¹	10.36	9.06	9.06
	Beta-carotene (0.4 wt.% sol in SF)	0.15	0.15	0.15
Water phase				
	Water	59.5	64.5	44.5
	Salt	0.5	0.5	0.5
	Starch solution (5% Resistamyl 310)			20
	Total	100	100	100

¹ Hardstock fat as prepared in example 3 of WO96/19115 mixed with 50 wt% beta sitosterol was micronised as in example 1 of PCT/EP2004/006544, and granulated as in example 1 with sunflower oil in ratio 1:1 (w/w).

Claims

1. Granule comprising:-
 - a) solid micronised lipid powder particles that have a microporous structure; and,
 - b) a liquid;wherein the granule is an agglomeration of said lipid particles herein described as secondary particles; and wherein said secondary particles are agglomerates of primary lipid particles, said primary lipid particles are platelets having an average thickness of 0.01-0.5 μm and wherein the lipid comprises sterol.
2. Granule according to claim 1 wherein the solid lipid particles comprise from 25 to 100 wt.% sterol.
3. Granule according to claims 1 or 2, having a liquid content of 40-60 wt.%.
4. Granule according to any of claims 1-3, wherein the liquid is vegetable oil or a water-in oil emulsion comprising vegetable oil.
5. Granule according to any of claims 1-4, wherein the lipid powder particles comprise micronised fat particles.
6. Process for the preparation of an edible dispersion comprising;
 - a) an oil phase,
 - b) a structuring agent; and,

- c) an aqueous phase, in which the dispersion is formed by mixing oil, structuring agent and the aqueous phase,
wherein the structuring agent is a granule according to any of claims 1-5.
7. Cube or tablet comprising made by compression of granules according to any of claims 1-5.
8. Process for preparing bouillon or seasoning cubes or tablets, the process comprising the steps of:
- a) mixing 2-99% of particulate ingredients comprising one or more of salt, flavour enhancers, spices, herbs, flavourants, sugar, yeast extract or hydrolysed vegetable protein, with 1-98% of fat solid at 20°C in particulate form,
- b) pressing to cubes,
wherein the fat in particulate form comprises granules according to claims 1-5.
9. Particulate seasoning or concentrate for preparing a bouillon, broth, soup or sauce comprising 0.5-60 wt.% of fat and 40-99.5 wt.% of one or more of salt, flavour enhancers, starch, starch derivatives, sugar, herbs, spices, vegetable particulates, flavours, colourants, wherein the fat comprises granules according to claims 1-5.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2006/000798

A. CLASSIFICATION OF SUBJECT MATTER
INV. A23D9/05 A23L1/00 A23L1/40

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)
A23D A23L

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, PAJ, WPI Data, FSTA, BIOSIS, EMBASE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	WO 2005/014158 A (UNILEVER N.V; UNILEVER PLC; HINDUSTAN LEVER LIMITED; VAN DEN BERG, COR) 17 February 2005 (2005-02-17) example 1; table 1	1-6,9
X	DE 102 53 111 A1 (BASF AG) 27 May 2004 (2004-05-27) examples 1-3	1-6
X	EP 1 419 698 A (UNILEVER N.V; UNILEVER PLC) 19 May 2004 (2004-05-19) paragraphs [0002], [0013] - [0015], [0024]; examples 1-6	1-9
X	US 6 248 389 B1 (BILLER FLORIAN ET AL) 19 June 2001 (2001-06-19) examples 1-4	1-9
	----- -/--	

☒ 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
- *E* earlier document but published on or after the international filing date
- *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)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *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
- *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
- *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.
- * & * document member of the same patent family

Date of the actual completion of the international search

26 April 2006

Date of mailing of the international search report

08/05/2006

Name and mailing address of the ISA/
European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Graham, J

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2006/000798

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2002/168450 A1 (DRUDIS JAIME ET AL) 14 November 2002 (2002-11-14) examples 1,2 -----	1-9
X	WO 01/00046 A (COGNIS DEUTSCHLAND GMBH; KROPF, CHRISTIAN; FABRY, BERND; BIERMANN, MAN) 4 January 2001 (2001-01-04) table 1 -----	1,2,5
X	EP 0 897 671 A (UNILEVER N.V; UNILEVER PLC) 24 February 1999 (1999-02-24) paragraphs [0028], [0045] -----	1-9
A	GERBER P ET AL: "EFFECT OF PROCESS-PARAMETERS ON PARTICLES OBTAINED BY THE RAPID EXPANSION OF SUPERCRITICAL SOLUTIONS" PROCEEDINGS WORLD CONGRESS ON PARTICLE TECHNOLOGY, 6 July 1998 (1998-07-06), pages 1-11, XP001080632 figure 5 -----	1-9

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2006/000798

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 2005014158	A	17-02-2005	AU 2004262853 A1	17-02-2005
DE 10253111	A1	27-05-2004	AU 2003304369 A1	14-02-2005
			CN 1741748 A	01-03-2006
			WO 2005009144 A1	03-02-2005
			EP 1575378 A1	21-09-2005
			US 2006035871 A1	16-02-2006
EP 1419698	A	19-05-2004	AU 2003302004 A1	15-06-2004
			BR 0315062 A	16-08-2005
			CN 1711031 A	21-12-2005
			DE 20317533 U1	12-02-2004
			WO 2004045298 A1	03-06-2004
			MX PA05005276 A	25-07-2005
			NL 1024763 C2	26-05-2004
			US 2005281935 A1	22-12-2005
US 6248389	B1	19-06-2001	NONE	
US 2002168450	A1	14-11-2002	NONE	
WO 0100046	A	04-01-2001	AT 304799 T	15-10-2005
			AU 5405200 A	31-01-2001
			CA 2376818 A1	04-01-2001
			DE 50011218 D1	27-10-2005
			EP 1189522 A1	27-03-2002
			ES 2248082 T3	16-03-2006
			JP 2003516115 T	13-05-2003
			NO 20016342 A	21-12-2001
EP 0897671	A	24-02-1999	BR 9803191 A	11-01-2000
			CA 2245467 A1	22-02-1999
			DE 69808207 D1	31-10-2002
			DE 69808207 T2	07-08-2003
			DE 69829119 D1	31-03-2005
			DE 69829119 T2	09-02-2006
			DK 897671 T3	27-01-2003
			ES 2236436 T3	16-07-2005
			ES 2184196 T3	01-04-2003
			JP 11146757 A	02-06-1999
			US 6423363 B1	23-07-2002
			ZA 9807213 A	14-02-2000