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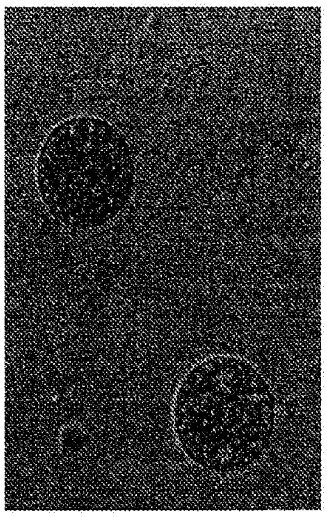
(54) **Double emulsion and method to produce such.**

(57) The invention is directed to a method to produce a water-in-oil-in-water (w/o/w) emulsion, comprising:
a. preparing a water-in-oil (w/o) emulsion;
b. atomizing said w/o emulsion in the presence of a carrier material comprising at least a water soluble matrix material and at least one emulsifier, to form agglomerates;
c. dispersing said agglomerates in an aqueous solution.
Also provided is a new instant powder (obtained after step b) that can be used to prepare the w/o/w emulsion. The emulsion of the invention is advantageously suited for the encapsulation of active components.



NL C 2002046

P86141NL00

Title: Double emulsion and method to produce such

FIELD OF THE INVENTION

The invention relates to the field of preparing emulsions, more specifically for the encapsulation of components. In particular the invention is related to making double emulsions of the water-oil-water (w/o/w) type.

BACKGROUND

In many applications in the food and pharmaceutical field there is a need to encapsulate water soluble components. The reason for this need is, for instance, to mask the taste of the water soluble component or to prevent chemical reactions between the water soluble components and further ingredients.

Double emulsions of the water-in-oil-in-water type (w/o/w emulsions) would be a solution for this problem. A w/o/w emulsion consists of droplets of oil in an aqueous environment in which the oil droplets are filled with water droplets in which hydrophilic components can be solved. Many examples of w/o/w emulsions are known in the literature (see e.g. Garti, N. and Lutz, R., 2004, Interface Sci. Technol. 4:557-605). In general, a double emulsion can be made by first dispersing water in oil, thereby creating a water-in-oil emulsion and subsequently dispersing this emulsion in water (Matsumoto et al., 1976, J. Colloid Interference Sci. 57:353-361). A common problem in preparing these type of emulsions is found in the behaviour of the inner water droplets which tend to 'escape' to the outer water phase because of thermodynamic stability. This behaviour not only occurs at the moment of preparing the emulsion but also during storage. Lifetime of double emulsions therefore is rather low. This could be solved by drying the double emulsion by, for instance, spray drying. However, the forces that are involved with spray drying would cause the

emulsion to disintegrate. Therefore, there is still need for a process to obtain a double emulsion that can be stored in powder form.

SUMMARY OF THE INVENTION

The invention concerns a method to produce a water-in-oil-in-water (w/o/w) emulsion comprising:

a. preparing a water-in-oil (w/o) emulsion;
b. atomizing said w/o emulsion in the presence of a carrier material comprising at least a water soluble matrix material and at least one emulsifier, to form agglomerates;
c. dispersing said agglomerates in an aqueous solution.

Preferably in said method the water soluble matrix is chosen from the group comprising lactose, dextrins such as maltodextrins, low molecular weight poly-ethylene glycols, highly disperse silicon dioxide, starch, modified starch and fatty acid salts. Also preferred for the emulsifier are a protein, preferably selected from the group consisting of casein, whey proteins, lectins, Tween's, esters of fatty acids such as citric, lactic and acetic acid esters, sodium and calcium stearoyl lactylates, and sorbitan esters of fatty acids.

The method of the invention is meant to encapsulate an active component. Thereto, either the w/o emulsion of step a) comprises an active component in the aqueous phase or in the oil phase.

Preferably in said method the atomization and agglomeration is performed in a spray drying tower. Also preferred in said method is the addition of gas to the w/o emulsion before atomization.

In another embodiment, the invention is directed to a w/o/w emulsion obtainable by any of the above methods.

In still a further embodiment, the invention comprises a powder that is obtained after step b) in a method according to the invention. Said powder preferably comprises agglomerates of oily o/w particles and powder

particles, wherein the powder particles comprise at least a water soluble matrix material and at least one emulsifier.

Also part of the invention is said powder, wherein the oily o/w particles and the powder particles have an average size of about 1 to about 20 micron.

A further part of the invention is a method to encapsulate an active ingredient comprising:

d. preparing a water-in-oil (w/o) emulsion in which said active ingredient is present in either the oil or the water phase;
e. spraying said w/o emulsion in the presence of a carrier material comprising at least a water soluble matrix material and at least one emulsifier, to form agglomerates; and
f. optionally dispersing said agglomerates in an aqueous solution.

The invention further relates to the use of a method of the invention for the encapsulation of an active ingredient.

LEGENDS TO THE FIGURES

Fig. 1 is a light microscopic view of the double emulsion of the invention. It shows relatively large oil droplets (diameter about 20 µm) in an aqueous phase filled with smaller water droplets.

Fig. 2 is a confocal scanning laser microscopic image from the powder obtained using the method claimed herein . Visible is an agglomerate of powder particles (black) and oil droplets (red) filled with water droplets.

Fig. 3 is a light microscopic view of the double emulsion obtained in Example 2.

DETAILED DESCRIPTION

Formulating active components in food and pharmaceutical applications is needed to give an optimal utilisation of the component. An 'active component' as used herein is defined as a water-soluble or oil-soluble compound or composition that needs to be formulated for application in food or pharmaceuticals. Such an active component may be a small molecular pharmaceutical compound, a peptide, a nucleic acid, a carbohydrate, a mineral, enzymes, microorganisms, any chemical compound that can be used as an additive in food, such as a flavour, an aroma, an antioxidant, a colorant, a perfume, a bio-active (meaning a substance inducing a health benefit), and combinations thereof

Emulsions, in general, are heterogeneous systems of one immiscible liquid dispersed in another in the form of droplets that usually have a diameter of more than 1 micron (μm). The two liquids are chemically unreactive and form systems characterized by a low thermodynamic stability. In most cases one liquid will be water or another aqueous solution (but in emulsions generally indicated as the water-phase), while the other liquid will be an oil-type liquid, such as an oil or fat or an organic solvent (but in emulsions generally indicated as the oil phase). Simple emulsions are classified according to the nature of their continuous and dispersed phase. It is custom to set forth the droplet (dispersed) phase first followed by the continuous phase separated by a / mark, i.e. either water(droplets)-in-oil(continuous) (w/o) or oil-in-water (o/w) emulsions. Multiple (or double) emulsions are characterized by listing the primary emulsion set first which is dispersed in a continuous phase. For example in a water-in-oil-in-water (w/o/w) multiple emulsion, a w/o emulsion is dispersed in a water continuous phase. During this dispersion step, for thermodynamic reasons, water droplets are likely to escape from the oil phase to the surrounding continuous water phase. Multiple emulsions are more complex systems as the drops of the dispersed

phase themselves contain even smaller dispersed droplets that normally consist of a liquid that is miscible, and in most cases, is identical with the continuous phase. They are, therefore, emulsions of emulsions.

Spray drying, as used herein, is a commonly used technique of drying a liquid feed through a hot gas. Typically, this hot gas is air. The liquid feed can be in the form of solutions, colloids, dispersions or emulsions. This process of drying is a one step rapid process and eliminates additional processing. The liquid feed is pumped through an atomiser device that produces fine droplets into the main drying chamber. Atomisers vary with rotary, single fluid, two-fluid, and ultra-sonic designs. These different styles have different advantages and disadvantages depending on the application of the spray drying required. In many instances a spray nozzle is used in place of an atomiser for a different dispersion rate. The hot drying gas can be passed as a co-current or counter-current flow to the atomiser direction. The co-current flow enables the particles to have a lower residence time within the system and the particle separator (typically a cyclone device) operates more efficiently.

Spray drying often is used as an encapsulation technique by the food and pharmaceutical industries. A substance to be encapsulated (the active component or a solution with the active component) and an amphipathic carrier (usually some sort of modified starch) are homogenized as a suspension. The suspension is then fed into a spray drier, usually a tower heated to temperatures well over the boiling point of water. As the slurry enters the tower, it is atomized. The small size of the drops results in a relatively large surface area which dries quickly. As the water dries, the carrier forms a hardened shell around the load. This spray drying technique has been used in US 5,496,574 to encapsulate an oil-in-water emulsion, whereby the emulsion is spray dried onto a soluble beverage powder, whereupon the continuous aqueous phase of the emulsion desiccated to form capsules. Similar techniques have been described to make a water-in-oil powder (see, e.g. Albertini, B. et al., 2008, Eur. J. Pharm. Biopharm., 69:348-

357) or a related technique, indicated as Particle Generation for Supersaturated Solutions (PGSS, see e.g. EP-A-1 569522). In this latter case supercritical CO_2 is solved into the emulsion before spray drying, which during spraying exacerbates from the emulsion and helps the atomization. Further, by the adiabatic effect automatically a cooling is caused which makes air cooling redundant. However, these techniques show a number of disadvantages. The particles that are produced in general are not or only marginally dispersable in water, the fat that is used as the oil phase should be solid at storage temperature and if small particles are needed it is very difficult to separate these from the air stream. These disadvantages can be solved by a technique called Concentrated Powder Form (CPF, see e.g. US 6,440,336, WO 2004/018070 and www.natex.at). In this technique a liquid feed like a solution or an emulsion is sprayed using supercritical CO_2 under addition of a powdery material, like starch, silica or cellulose. The result of the spraying in this case is the formation of agglomerates that are able to be dispersed in water. However, in this case, the oil droplets tend to attract to form large oil slicks, which float on the product, i.e. no stable o/w or w/o/w emulsion is formed.

In the present invention a powdery carrier material is used comprising at least a water soluble matrix material and at least one emulsifier, to form agglomerates. Agglomerates, as used in the present invention, are defined as any loosely connected particles of different substances, wherein the connection is formed by physical and not by chemical binding forces, such as adhesion, gravity, stickiness, etc. The powdery carrier material is agglomerated with a w/o emulsion in a spraying step. Upon dissolution of the thus obtained agglomerates, the oil droplets (filled with water containing optionally an active component) are dispersed in the water without coalescing with each other and without forming an oil/fat film on top of the water.

The matrix material should be rapidly solvable in an aqueous liquid and would preferably be chosen from the group of lactose, maltodextrin, low molecular weight poly-ethylene glycols, highly disperse silicon dioxide, starch, modified starch and magnesium stearate .

As emulsifier all commonly known emulsifiers which are usable in the food and the pharmaceutical industry can be used. The emulsifier is a surface acting agent, preferably selected from the group consisting of Tween, egg yolk, lecithin, proteins such as caseinate or whey protein, carrageenan, alginates, mannitol, glycerol, sorbitol, Arabic gum, guar gum, xanthan gum, tragacanth, methylcellulose, ethylcellulose, hydroxypropylcellulose, ethylmethylcellulose, carboxymethylcellulose, hydroxypropylmethylcellulose, monoacyl and diacyl glycerols.

The function of the emulsifier is to keep the w/o emulsion in its original form, i.e. preventing the oil droplets from coalescing with each other and from forming an oil film during reconstition of the agglomerates in an aqueous solution. The emulsifier, also indicated as stabiliser, thus allows a stable w/o/w emulsion to be formed upon dispersion/dissolution in water of the agglomerates between the carrier material and the w/o droplets. Preferably surface active compounds like proteins (such as whey protein or caseinate) are used as emulsifier. These compounds will effect their stabilising functions by surrounding the oil phase during dispersion / dissolution of the agglomerates in an aqueous solution.

In this way the invention comprises a method to form a water-in-oil-in-water (w/o/w) emulsion, comprising:

a. preparing a water-in-oil (w/o) emulsion;
b. atomization of said w/o emulsion in the presence of a carrier material comprising at least a water soluble matrix material and at least one further compound, selected from the group consisting of emulsifiers, to form agglomerates; the agglomerates combined forming a free-flowing powder.

c. dispersing said agglomerates in an aqueous solution.

The preparation of the w/o emulsion with which the process is started, will be done according to the methods and processes known to a person of skill in the art. Preferably the water phase of the w/o emulsion comprises one or more active ingredients that needs to be encapsulated. The water phase may consist of water or any other aqueous solution that is suitable for the food and pharmaceutical application. Also the oil phase may comprise any oil, fat or organic solvent that is acceptable in food and/or pharmaceuticals. Examples of oils that can be used are vegetable oils such as sunflower oil, soybean oil, corn oil, cottonseed oil, coconut oil, palpom kernel oil, safflower oil, neobee oil, canola oil, peanut oil, sesame oil, linseed (flax) oil, olive oil, Medium Chain Triglyceride (MCT) oils and the like, and animal fats and oils, such as fish oil and fats, milk fats, and the like. Of course, mixtures of any of these are also usable. When an oil is to be used, it is advantageous to use MCT oil in order to prevent the droplets from being oxidised during storage. The oil phase also contains an emulsifier to stabilise the water droplets in the w/o emulsion produced from the oil. Examples include Span's, Admul WOL and monoglycerides.

Atomization of the w/o emulsion can be performed in any way known to the person skilled in the art. To this end, well-known atomization nozzles such as a whirl chamber type pressure nozzle or a 2-fluid nozzle may be used. The carrier material may be introduced around the atomization nozzle for example by carrying the carrier material in an air stream co-flowing with the spray of atomized droplets. It may be advantageous to inject a gas into the w/o emulsion before atomization. This may help to create small droplets of atomized w/o emulsion and may enhance mixing of the atomized w/o emulsion with the carrier material in order to increase agglomeration. . Any gas would be suitable for this purpose, but preferred are N_2 and CO_2, wherein the last may be supercritical CO_2.

The above process may well be performed in a spray drying tower in which case the atomized emulsion is sprayed along with an air stream that surpasses the spray nozzle, in which air stream the carrier material as described above is supplied. In this way, agglomerates of the carrier material particles and oil droplets of the emulsion are formed. These agglomerates form a free flowing powder that is easily dispersed in an aqueous solution, thereby forming the double emulsion. The air stream helps to mix the atomized emulsion with the carrier material, to increase agglomeration, and transports the agglomerates to the exit of the tower to facilitate collection of the powder. It is not necessary to use a stream of hot air, i.e. to perform a drying action. On the other hand, the stream of air may well be hot e.g. to at least partially dry away the water inside the atomized w/o emulsion.

Another embodiment of the invention relates to the w/o/w emulsions that are obtained by the above mentioned method. The emulsions are characterized in that they are more stable than the double emulsions produced thus far. Double emulsions are thermodynamically stable and hence their shelf-life is limited, e.g. because the inner phase leaks out to the outer water phase. This could be solved by removing the outer water phase by drying as to stop processes of leakage until the moment when the double emulsion is reconstituted for use. However, spray-drying is unsuitable for this because of the high shear involved in the atomization step which will generally destroy the emulsion. The invention described here provides a means of producing a double emulsion in which the outer water phase is absent until the moment of reconstitution. Preferably, the oil droplets have a diameter of about 1 to about 25 µm, but if needed, the spray conditions can be adjusted to have very fine oil particles (< 1µm)

Another embodiment of the invention is formed by the powdery agglomerate that is obtained directly by the spraying/agglomerating. This powder is stable during storage and the double emulsion of the invention can instantly be obtained by dispersing it in an aqueous solution. Thus, it is usable

also as an instant power to be commercialized directly (e.g. for applications in soups or for pharmaceutical suspensions that need to be reconstituted just before use, like in vaccines). This powder thus will comprise fat or oil particles agglomerated with particles comprising a water-soluble matrix material and an emulsifier.

The method of the invention and/or the powder of the invention are usable for encapsulating an active component for use in food and pharmacology. It can be applied to all kinds of foods, for example cereals and derived (optionally muesli, cereals for milk), pastry shop, dairy products, nutritional supplements, sugars and derived (optionally chocolates, sweet, nougat, marzipan), sweet dietary (with low level of calories), in regime foods and for diabetics, oils and derived, milk and derived, eggs, vegetables, fruits, tubers and derived, eatable shafts, snacks, appetizers, eatable roots (optionally licorice), bay and wild products, preserves of fruits, meats, sausages, fish, shellfish and crustaceans and their preserves, alcoholic and non-alcoholic drinks, carbonated or still drinks, juices, syrups, nectars, spices, condiments, pre-cooked foods, pre-processed foods (frozen mass of bread), pizzas, honey, soups, bakery products (including bread, cookies, cakes), desserts, crèmes, etc.

Especially preferred is the encapsulation of minerals (e.g. in drinks) so that they cannot chemically react with other ingredients, and the encapsulation of peptides to mask their taste.

Also preferred is the encapsulation of probiotics in order to have the oil phase protect them from harsh environments such as acidic media.

Although the main and more useful embodiments of the invention relate to food and pharmaceuticals, the encapsulation process can be employed for other purposes, in particular to encapsulate herbicides, pesticides, germicides, viricides, attractants, repellents, sterilizers, (recombinant) DNA, nucleic acid derivatives, peptides, marker compounds, toilet chemicals, cosmetics, etc.

EXAMPLE 1

A w/o emulsion was made from 25% sucrose, 25% water, 1% Admul WOL (Polyglycerol polyricinoleate supplied by Danisco) and 49% sunflower oil. The aqueous emulsion was dispersed finely in the oil phase by homogenization with a high pressure homogenizer (Niro Soavi) at a pressure of 150/50 bar. Thereafter, the emulsion was spray dried through a high pressure nozzle (whirl chamber type) in a filtermat spray dryer. The liquid flow was 20 L/hr, the spraying pressure 100 bar and the temperature of the emulsion 70°C. Before spraying 10 kg CO_2 per hour was injected into the liquid feed. Around the high pressure nozzle an air stream was created in which 40 kg/hr powder was mixed with the emulsion spray. This powder had an average particle size of 10 µm and consisted of 10% caseinate and 90% maltodextrin 33DE. The temperature of the airstream in the tower was about 25°C.

In this way a free flowing powder was created. When 10 grams of this powder were solved into 150 ml water a stable emulsion was formed without any visible oil droplets. Analysis by microscope (Fig. 1) revealed that the emulsion consisted of droplets of about 10 µm filled with small droplets (<1 µm) of water.

Confocal scanning laser microscopy (CSLM) analysis of the powder, in which the oil was stained red, revealed that the powder consisted of an agglomerate of the particles injected through powder injection and oil droplets filled with an aqueous solution (Fig. 2).

EXAMPLE 2

A water-in-oil emulsion was prepared with the following composition: 49% sunflower oil, 1% Admul WOL, 24% water, 3% skimmed milk powder, 3% probiotics (BB-12 Bifidobacterium from Chr. Hanssen).

The emulsion was homogenized with a high pressure homogenizer at 150/50 bar and sprayed with a flat body high pressure nozzle (Spraying

systems 70/17). Around the nozzle cryogenicall milled Hiprotal 35 (obtained from Friesland Foods Domo) was blown through powder injection, in a ratio of powder : emulsion of 2:1, causing agglomeration with the emulsion and giving a free-flowing powder.

Microscopic analysis of the w/o/w emulsion obtained through dispersing 5% of the powder into water showed that the emulsion comprised oil droplets in which smaller water droplets in which the probiotic was solved (Fig. 3) .

To this emulsion HCl was added to pH 2 and the acidified emulsion was kept for 80 minutes at 37°C to simulate passage through the stomach. The same treatment was applied to a control solution of BB12 powder in water. After incubation, viability of the bacteria was scored: the double emulsion showed 13% survival, while in the control only 1.6% of the bacteria survived.

CONCLUSIES

1. Werkwijze voor het produceren van een water-in-olie-in-water (w/o/w) emulsie, omvattende:
   a. bereiden van een water-in-olie (w/o) emulsie;
   b. atomiseren van genoemde w/o emulsie in de aanwezigheid van een dragermateriaal omvattende ten minste een wateroplosbaar matrixmateriaal en ten minste een emulgator, om agglomeraten te vormen;
   c. dispergen van genoemde agglomeraten in een waterige oplossing.

2. Werkwijze volgens conclusie 1, waarin de wateroplosbare matrix gekozen is uit de groep bestaande uit lactose, dextrines zoals maltodextrines, polyethyleenglycolen met laag moleculair gewicht, zeer disperse siliciumdioxide, zetmeel, gemodificeerd zetmeel en vetzure zouten.

3. Werkwijze volgens conclusie 1 of 2, waarin de emulgator gekozen is uit de groep bestaande uit een eiwit, bij voorkeur gekozen uit de groep bestaande uit caseïne, wei-eiwitten en lectines, Tween's, esters van vetzuren zoals citroenzure, melkzure en azijnzure esters, natrium en calcium stearoyllactylaten, een sorbitan esters van vetzuren.

4. Werkwijze volgens een der voorgaande conclusies waarin de w/o emulsie van stap a) een actieve component in de waterige fase omvat.

5. Werkwijze volgens een der voorgaande conclusies waarin de w/o emulsie van stap a) een actieve component in de olie fase omvat.

6. Werkwijze volgens een der voorgaande conclusies, waarin het atomiseren en agglomeratie wordt uitgevoerd in een sproeidroogtoren.

7. Werkwijze volgens conclusie 6, waarin gas wordt toegevoegd aan de w/o emulsie voorafgaand aan het atomiseren.

8. W/o/w emulsie verkrijgbaar door de werkwijze van een der conclusies 1-7.

9. Poeder na stap b) in een werkwijze volgens een der conclusies 1-7.

10. Poeder volgens conclusie 9 omvattende agglomeraten van olieachtige o/w deeltjes en poeder deeltjes, waarin de poeder deeltjes ten minste een wateroplosbaar matrixmateriaal en ten minste een emulgator omvatten.

11. Poeder volgens conclusie 9 of 10, waarin de olieachtige o/w deeltjes en de poeder deeltjes een gemiddelde grootte van ongeveer 1 tot ongeveer 20 micron hebben.

12. Werkwijze voor het inkapselen van een active ingrediënt, omvattende:

    a. bereiden van een water-in-olie (w/o) emulsie waarin genoemde actieve ingrediënt aanwezig is in hetzij de olie hetzij de water fase;

b. atomiseren van genoemde w/o emulsie in de aanwezigheid van een dragermateriaal omvattende ten minste een wateroplosbaar matrixmateriaal en ten minste een emulgator, om agglomeraten te vormen;

c. optioneel dispergen van genoemde agglomeraten in een waterige oplossing.

13. Toepassing van een werkwijze volgens conclusie 4 of 5 voor het inkapselen van een actieve ingrediënt.

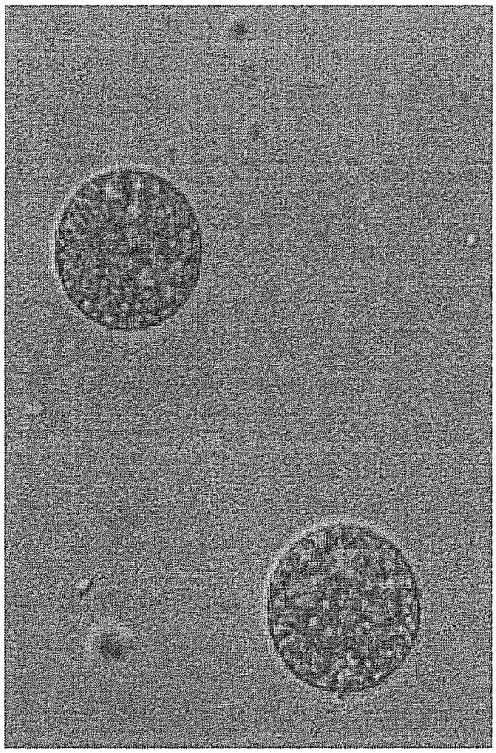


Fig. 1

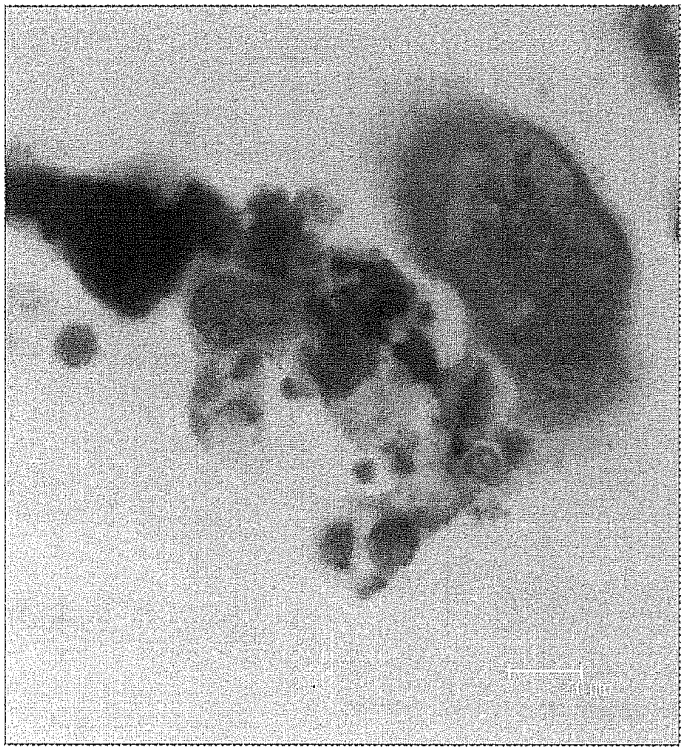


Fig. 2

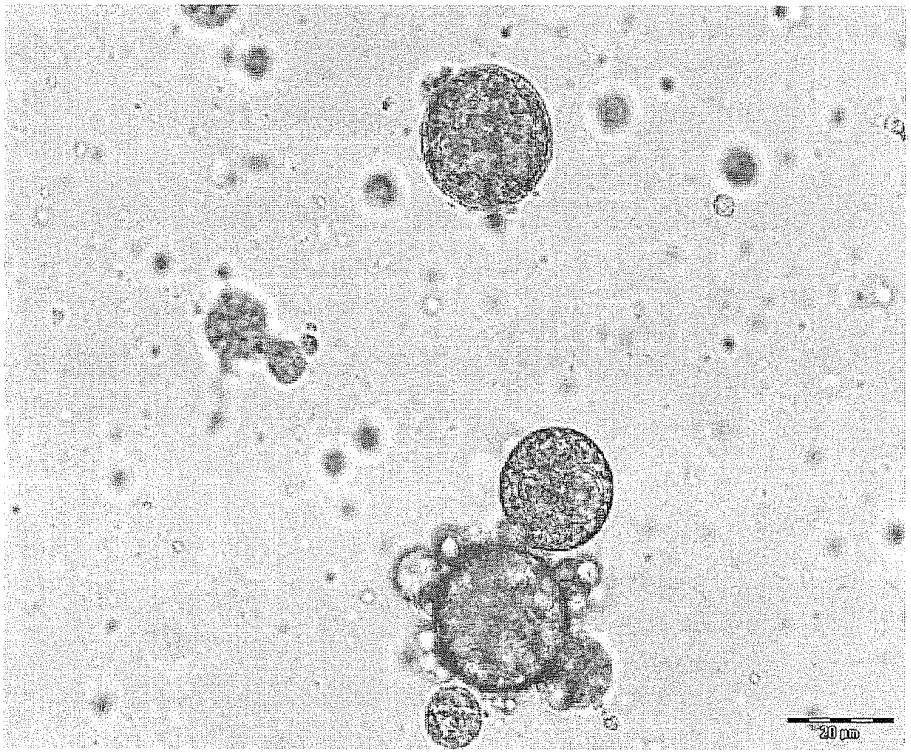


Fig. 3

SAMENWERKINGSVERDRAG (PCT)

RAPPORT BETREFFENDE NIEUWHEIDSONDERZOEK VAN INTERNATIONAAL TYPE

IDENTIFICATIE VAN DE NATIONALE AANVRAGE	KENMERK VAN DE AANVRAGER OF VAN DE GEMACHTIGDE
	P86141NL00
Nederlands aanvraag nr. **2002046**	Indieningsdatum **01-10-2008**
	Ingeroepen voorrangsdatum
Aanvrager (Naam) **Friesland Brands B.V.**	
Datum van het verzoek voor een onderzoek van internationaal type **03-02-2009**	Door de Instantie voor Internationaal Onderzoek aan het verzoek voor een onderzoek van internationaal type toegekend nr. **SN 51640**

I. CLASSIFICATIE VAN HET ONDERWERP (bij toepassing van verschillende classificaties, alle classificatiesymbolen opgeven)

Volgens de internationale classificatie (IPC)

A23L1/22 **A23L1/00** **A23P1/04** **A23P1/06**
A61K9/113

II. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

Onderzochte minimumdocumentatie

Classificatiesysteem	Classificatiesymbolen
IPC8	**A23L** **A23P** **A61K**

Onderzochte andere documentatie dan de minimum documentatie, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

III. ☐ **GEEN ONDERZOEK MOGELIJK VOOR BEPAALDE CONCLUSIES** (opmerkingen op aanvullingsblad)

IV. ☐ **GEBREK AAN EENHEID VAN UITVINDING** (opmerkingen op aanvullingsblad)

ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Nummer van het verzoek om een onderzoek naar de stand van de techniek
NL 2002046

A. CLASSIFICATIE VAN HET ONDERWERP
INV. A23L1/22 A23L1/00 A23P1/04 A23P1/06 A61K9/113

Volgens de Internationale Classificatie van octrooien (IPC) of zowel volgens de nationale classificatie als volgens de IPC.

B. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

Onderzochte miminum documentatie (classificatie gevolgd door classificatiesymbolen)
A23L A23P A61K

Onderzochte andere documentatie dan de mimimum documentatie, voor dergelijke documenten, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

Tijdens het onderzoek geraadpleegde elektronische gegevensbestanden (naam van de gegevensbestanden en, waar uitvoerbaar, gebruikte trefwoorden)
EPO-Internal, WPI Data, FSTA, BIOSIS

C. VAN BELANG GEACHTE DOCUMENTEN

Categorie °	Geciteerde documenten, eventueel met aanduiding van speciaal van belang zijnde passages	Van belang voor conclusie nr.
X Y	US 4 933 192 A (DARLING DONALD F [GB] ET AL) 12 juni 1990 (1990-06-12) conclusies 1-12; voorbeelden I-V ----	1-3,6, 8-11 4,5,7, 12,13
X	FECHNER ET AL: "Stability and release properties of double-emulsions stabilised by caseinate-dextran conjugates" FOOD HYDROCOLLOIDS, ELSEVIER, deel 21, nr. 5-6, 15 maart 2007 (2007-03-15), bladzijden 943-952, XP005925154 ISSN: 0268-005X	8
Y	bladzijde 945, kolom 1 bladzijde 951 ----	4,5,12, 13

-/--

[X] Verdere documenten worden vermeld in het vervolg van vak C.

[X] Leden van dezelfde octrooifamilie zijn vermeld in een bijlage

° Speciale categorieën van aangehaalde documenten

"A" niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijft

"D" in de octrooiaanvrage vermeld

"E" eerdere octrooi(aanvrage), gepubliceerd op of na de indieningsdatum, waarin dezelfde uitvinding wordt beschreven

"L" om andere redenen vermelde literatuur

"O" niet-schriftelijke stand van de techniek

"P" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur

"T" na de indieningsdatum of de voorrangsdatum gepubliceerde literatuur die niet bezwarend is voor de octrooiaanvrage, maar wordt vermeld ter verheldering van de theorie of het principe dat ten grondslag ligt aan de uitvinding

"X" de conclusie wordt als niet nieuw of niet inventief beschouwd ten opzichte van deze literatuur

"Y" de conclusie wordt als niet inventief beschouwd ten opzichte van de combinatie van deze literatuur met andere geciteerde literatuur van dezelfde categorie, waarbij de combinatie voor de vakman voor de hand liggend wordt geacht

"&" lid van dezelfde octrooifamilie of overeenkomstige octrooipublicatie

Datum waarop het onderzoek naar de stand van de techniek van internationaal type werd voltooid	Verzenddatum van het rapport van het onderzoek naar de stand van de techniek van internationaal type
15 April 2009	

Naam en adres van de instantie	De bevoegde ambtenaar
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	De Jong, Ellen

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ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Nummer van het verzoek om een onderzoek naar de stand van de techniek
NL 2002046

C.(Vervolg). VAN BELANG GEACHTE DOCUMENTEN

Categorie °	Geciteerde documenten, eventueel met aanduiding van speciaal van belang zijnde passages	Van belang voor conclusie nr.
X	BENICHOU ET AL: "W/O/W double emulsions stabilized with WPI-polysaccharide complexes" COLLOIDS AND SURFACES. A, PHYSICACHEMICAL AND ENGINEERING ASPECTS, ELSEVIER, AMSTERDAM, NL, deel 294, nr. 1-3, 2 februari 2007 (2007-02-02), bladzijden 20-32, XP005870823 ISSN: 0927-7757	8
Y	bladzijde 21 bladzijde 31	4,5,12, 13

X	WO 03/049548 A (UNILEVER NV [NL]; UNILEVER PLC [GB]; LEVER HINDUSTAN LTD [IN]; APPELQV) 19 juni 2003 (2003-06-19)	8
A	bladzijde 6, regels 9-32; conclusies 1-19	1-7,12, 13

X	MUSCHIOLIK ET AL: "Multiple emulsions for food use" CURRENT OPINION IN COLLOID AND INTERFACE SCIENCE, LONDON, GB, deel 12, nr. 4-5, 1 oktober 2007 (2007-10-01), bladzijden 213-220, XP022266053 ISSN: 1359-0294	8
Y	het gehele document	4,5,12, 13

A	US 6 440 336 B1 (WEINREICH BERND [DE] ET AL) 27 augustus 2002 (2002-08-27) in de aanvraag genoemd	1-6,8-13
Y	conclusies 1-20	7

A	EP 1 757 361 A (FEYECON DEV & IMPLEMENTATION B [NL]) 28 februari 2007 (2007-02-28) alineas [0016], [0027]; voorbeelden 1-3	1-13

A	US 6 482 433 B1 (DEROOS KRIS BART [CH] ET AL) 19 november 2002 (2002-11-19) kolom 4, regel 14 - kolom 5, regel 61	1-13

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ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Informatie over leden van dezelfde octrooifamilie

Nummer van het verzoek om een onderzoek naar de stand van de techniek: NL 2002046

In het rapport genoemd octrooigeschrift	Datum van publicatie	Overeenkomend(e) geschrift(en)		Datum van publicatie
US 4933192	A 12-06-1990	AU	590694 B2	09-11-1989
		AU	7052287 A	01-10-1987
		CA	1297722 C	24-03-1992
		DE	3779348 D1	02-07-1992
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WO 03049548	A 19-06-2003	AU	2002349035 A1	23-06-2003
US 6440336	B1 27-08-2002	AT	204787 T	15-09-2001
		CA	2307370 A1	15-04-1999
		DE	59704488 D1	04-10-2001
		DK	1021241 T3	27-12-2001
		WO	9917868 A1	15-04-1999
		EP	1021241 A1	26-07-2000
		ES	2163138 T3	16-01-2002
		JP	3492317 B2	03-02-2004
		JP	2001518391 T	16-10-2001
		PT	1021241 E	30-01-2002
EP 1757361	A 28-02-2007	AU	2006282152 A1	01-03-2007
		CA	2618894 A1	01-03-2007
		CN	101247885 A	20-08-2008
		JP	2009505820 T	12-02-2009
		WO	2007024133 A1	01-03-2007
		US	2008213380 A1	04-09-2008
US 6482433	B1 19-11-2002	AT	292393 T	15-04-2005
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		CA	2313011 A1	30-12-2000
		DE	60019199 D1	12-05-2005
		DE	60019199 T2	09-03-2006
		JP	2001072773 A	21-03-2001
		SG	86410 A1	19-02-2002
		ZA	200003120 A	02-01-2001

OCTROOICENTRUM NEDERLAND

WRITTEN OPINION

File No.	Filing date *(day/month/year)*	Priority date *(day/month/year)*	Application No.
SN51640	01.10.2008		NL2002046

International Patent Classification (IPC)
INV. A23L1/22 A23L1/00 A23P1/04 A23P1/06 A61K9/113

Applicant
Friesland Brands B.V. te Meppel

This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the application
- ☐ Box No. VIII Certain observations on the application

	Examiner
	De Jong, Ellen

Application number

WRITTEN OPINION

NL2002046

Box No. I Basis of this opinion

1. This opinion has been established on the basis of the latest set of claims filed before the start of the search.

2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the application and necessary to the claimed invention, this opinion has been established on the basis of:

   a. type of material:

   ☐ a sequence listing

   ☐ table(s) related to the sequence listing

   b. format of material:

   ☐ on paper

   ☐ in electronic form

   c. time of filing/furnishing:

   ☐ contained in the application as filed.

   ☐ filed together with the application in electronic form.

   ☐ furnished subsequently for the purposes of search.

3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4. Additional comments:

Box No. V Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty	Yes:	Claims	4,5,7,12,13
	No:	Claims	1-3,6,8-11
Inventive step	Yes:	Claims	
	No:	Claims	1-13
Industrial applicability	Yes:	Claims	1-13
	No:	Claims	

2. Citations and explanations

see separate sheet

Ad V

1. Reference is made to the following documents:

D1: US-A-4 933 192 (DARLING DONALD F [GB] ET AL) 12 juni 1990 (1990-06-12)
D2: FECHNER ET AL: "Stability and release properties of double-emulsions stabilised by caseinate-dextran conjugates" FOOD HYDROCOLLOIDS, ELSEVIER, deel 21, nr. 5-6, 15 maart 2007 (2007-03-15), bladzijden 943-952, XP005925154 ISSN: 0268-005X
D3: BENICHOU ET AL: "W/O/W double emulsions stabilized with WPI-polysaccharide complexes" COLLOIDS AND SURFACES. A, PHYSICACHEMICAL AND ENGINEERING ASPECTS, ELSEVIER, AMSTERDAM, NL, deel 294, nr. 1-3, 2 februari 2007 (2007-02-02), bladzijden 20-32, XP005870823 ISSN: 0927-7757
D4: WO 03/049548 A (UNILEVER NV [NL]; UNILEVER PLC [GB]; LEVER HINDUSTAN LTD [IN]; APPELQV) 19 juni 2003 (2003-06-19)
D5: MUSCHIOLIK ET AL: "Multiple emulsions for food use" CURRENT OPINION IN COLLOID AND INTERFACE SCIENCE, LONDON, GB, deel 12, nr. 4-5, 1 oktober 2007 (2007-10-01), bladzijden 213-220, XP022266053 ISSN: 1359-0294
D6: US-B1-6 440 336 (WEINREICH BERND [DE] ET AL) 27 augustus 2002 (2002-08-27) in de aanvraag genoemd

2. D1 discloses a method of preparing a w/o/w emulsion by rehydrating a powder prepared by spray drying a w/o/w emulsion, which was obtained by dispersing a w/o emulsion in water containing sodium caseinate and maltodextrin (Example I).

The powder and the w/o/w emulsion are considered to anticipate the subject-matter of claims 8-11.
Furthermore, since the w/o emulsion is spray dried "in the presence of sodium caseinate and maltodextrin" (i.e. being in a w/o/w emulsion), the subject-matter of claims 1-3 and 6 is also considered to be anticipated.

3. D2 teaches a w/o/w emulsion containing wherein the W_2 phase contains sodium caseinate-dextran conjugates (p.945 and 951).

D3 discloses a w/o/w emulsion, wherein the external oil-water interface contains a WPI-xanthan gum complex (p.21, 31).
D4 discloses a duplex emulsion (w/o/w) wherein the outer water phase contains an emulsifier selected from proteins, gums, modified starches (p.6 l.9-32).
D5 refers to a w/o/w emulsion prepared by emulsifying a w/o phase into the outer w-phase containing whey protein and xanthan gum (par.2.3.3).

D2-D5 are considered to anticipate the subject-matter of claim 8.

4. Extrapolating the method of D1 to emulsions having an active component inside the water or oil phase is not considered to involve an inventive step in view of D2, D3 and D5. The technique of spraying an emulsion using supercritical CO_2 was known from D6.

Thus, the subject-matter of claims 4, 5, 7, 12 and 13 is not considered to involve an inventive step.