Powder Technology 255 (2014) 103–108

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Use of different supports for oil encapsulation in powder by spray drying

C. Turchiuli [a,b,c,*], M.T. Jimenez Munguia [d], M. Hernandez Sanchez [a], H. Cortes Ferre [a], E. Dumoulin [a,b,c]

[a] AgroParisTech, UMR 1145 Ingénierie Procédés Aliments, 1 avenue des olympiades, 91300 Massy, France
[b] INRA, UMR 1145 Ingénierie Procédés Aliments, 1 avenue des olympiades, 91300 Massy, France
[c] CNAM, UMR 1145 Ingénierie Procédés Aliments, 1 avenue des olympiades, 91300 Massy, France
[d] Universidad de las Américas, Sta Catarina Martir, 72810 Puebla, Mexique

ARTICLE INFO

Available online 28 August 2013

Keywords:
Dry emulsion
Spray drying
Powder
Encapsulation

ABSTRACT

Spray drying of oil-in-water emulsions containing hydrophilic carriers is used to encapsulate lipophilic compounds into powders. Oil droplets are dispersed within the solid matrix of carriers acting like a barrier. To study the influence of the nature of the carrier on both the properties of the initial and dry emulsion and on the spray drying process, α-tocopherol dispersed in olive oil (weight ratio 1/4) was used as a model lipophilic molecule.

Eight initial oil-in-water emulsions containing 4% w/w oil phase and 36% w/w carrier consisting of different food polymers as maltodextrin DE12, acacia gum and inulin, mixed in different proportions were prepared by rotor-stator homogenization and characterized for their size, size distribution and viscosity.

They were spray dried in a pilot spray dryer in the same conditions (inlet and outlet air temperatures of 180 °C and 90 °C respectively, emulsion flow rate 57 $g \cdot min^{-1}$) and the properties of the dry emulsions produced were characterized.

Whatever the support used, the powder yield of the spray drying process was higher than 50% without optimization of the operating conditions. The dry emulsions produced had similar properties (size, size distribution, density and flowability) and contained more than 73% of the initial oil with only 5% of the oil phase on the particle surface (unencapsulated). After powder dissolution in water, the reconstituted emulsions had a size distribution similar to that of the initial emulsions, indicating that spraying did not modify the emulsion structure. Due to its emulsifying and film forming properties, the use of acacia gum, in combination with maltodextrin and/or inulin, allowed obtaining more stable initial emulsions with controlled size distribution (~2 μm, monodispersed) leading to higher powder yield for spray drying (e.g., superior to 65%).

Agave inulin was found to be a possible alternative to maltodextrin to produce powders with increased health benefits.

1. Introduction

Encapsulation of active molecules aims at creating a barrier between the molecules and the environment. It is used to protect active molecules against light, humidity and oxygen in order to avoid or delay their degradation and stabilise them during storage before use. It also allows limiting or controlling their transfer to the environment in order to avoid losses, to mask some of their properties (taste, odour, catalytic activity) or to get a controlled (at given time and place) and dosed (total or progressive) delivery [1].

Encapsulation is widely used in the food, chemical and pharmaceutical industry where active molecules are often lipophilic compounds (aroma, vitamins, antioxidants, drugs), not or poorly soluble in water. Their encapsulation may consist in creating an oil in water emulsion by dispersing them, eventually after dilution in oil, in an hydrophilic continuous phase containing long chain polymers. These polymers will protect the oil active molecules by isolating them from the environment and limiting their mobility.

Removing water from the aqueous phase by spray drying allows obtaining a powdered dry emulsion where oil droplets containing the active molecules are dispersed within the solid polymer matrix of powder particles. The powder form is stable and allows easier dosage and mixing with other powders. However, for efficient encapsulation, the proportion of unencapsulated molecules on the particle surface has to be low; and for easy handling, the powder must have good flowability and mixing ability and allow the reconstitution of the initial wet emulsion by rehydration in water [2]. Encapsulation efficiency and powder properties are influenced by the different steps of the spray drying process comprising (1) the preparation of the initial oil in water wet emulsion with a given structure, (2) its spraying in small drops into hot air and (3) the drying of each individual drop leading finally to dry

* Corresponding author at: AgroParisTech, UMR 1145 Ingénierie Procédés Aliments, 1 Avenue des Olympiades, 91300 Massy, France. Tel.: +33 1 69 93 50 71; fax: +33 1 69 93 50 05.
E-mail addresses: christelle.turchiuli@agroparistech.fr (C. Turchiuli), mariat.jimenez@udlap.mx (M.T. Jimenez Munguia), mariadelrayo.hernandezsanchez@agroparistech.fr (M. Hernandez Sanchez), elisabeth.dumoulin@agroparistech.fr (E. Dumoulin).

0032-5910/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.powtec.2013.08.026

solid particles [3–6]. The behaviour of the product during the spraying and drying steps depends on the emulsion properties. Especially, the composition of both hydrophilic and lipophilic phases, their weight ratio, the dry matter content and oil droplet size distribution of the emulsion and its viscosity were found to be key parameters for aroma encapsulation [7–11]. Different studies have shown that the decrease of the emulsion oil droplets size (down to 2 μm) causes an increase of the encapsulation efficiency, e.g., a better retention of the active molecule during spray drying, a lower quantity of non encapsulated oil on the particle surface or a better protection of the oil phase regarding oxidation [12,13]. The initial emulsion oil droplet size distribution is therefore a key parameter to control. The choice of the carrier materials that will constitute the dry solid matrix is also important [14,15]. They must have good spray drying ability: allow forming aqueous solutions with reasonable viscosity to be pumped and sprayed, be not too sticky or hygroscopic products for rapid and efficient drying and good stability of the powder during storage. In the food industry, the main carriers used for oil encapsulation are polysaccharides, starches, celluloses, gums and proteins. They are used alone or in combination. To produce fine and stable emulsions and to have a better protection of the oil active molecule during spray drying, emulsifying and film forming compounds are also often necessary [16]. Local products, with good availability and offering some health benefits, may be preferred [17,8]. Acacia gum, a natural hydrocolloid obtained by exudation from acacia and known for its emulsifying and film forming properties, and maltodextrin, a neutral and inexpensive starch hydrolyzate, are often associated for the encapsulation of oil with an efficiency which is a function of proportions of each [18,19]. Due to its technical and nutritive properties, inulin may also be an interesting possible encapsulation agent. It is a fructooligosaccharide with prebiotic effects and dietary fiber action. It causes no increase of the glycemic index, which makes it a potential ingredient for diabetic food, and it is known to improve calcium biodisponibility [20]. Agave inulin is extracted from the blue agave. Unlike other types of inulin, it is very soluble in cold water and has more calcium and minerals, with a neutral and mildly sweet flavour. Properties of agave inulin make it a potential support for encapsulation, especially in Mexico where it is produced in large quantities, that will add health benefits to the product. α-Tocopherol is a lipid-soluble antioxidant contained in vitamin E. Its high oxidative sensitivity requires protection during storage.

In this study, α-tocopherol was prediluted in olive oil and encapsulated into dry emulsions by spray drying. Different support materials, maltodextrin DE12[1] (MD), acacia gum[2] (GA) and agave inulin[3] (I), were used alone or mixed in different proportions. The size, size distribution and viscosity of the initial emulsions, containing 40% w/w total dry matter with 8% w/w olive oil and 2% w/w of α-tocopherol, were measured in order to follow their evolution according to the dry matter composition and to study their influence on the spray drying powder yield, on the properties of the powders obtained and on the encapsulation efficiency.

2. Material and methods

2.1. Products

Oil phase represented 4% w/w of initial oil in water emulsions. It consisted of α-tocopherol (Sigma, Ge) and olive oil (Lesieur, Fr—15% w/w saturated, 77% w/w mono-unsaturated and 8% w/w poly-unsaturated fatty acids) with a weight ratio of 1:4. In the aqueous phase, different hydrophilic carriers were used alone or in mixes with different ratios (Table 1): maltodextrin DE12 (MD) (Glucides, Roquette, Fr), agave inulin (I) (Oligofructine, Nutriagaves, Mx) and acacia gum (GA) (Instantgum AA, Nexira, Fr). The total dry matter content of all initial emulsions (hydrophilic and lipophilic compounds) was 40% w/w in which the lipophilic compounds represented 10% w/w (comprising 2% w/w α-tocopherol).

Table 1
Mass composition of emulsions studied (40% w/w dry matter).

Emulsion		Dry matter					Water
		MD	GA	I	Olive oil	α-Tocopherol	
1 polymer	MD	36.4	–	–	2.9	0.7	60
2 polymers	MD-I (1:1)	18	–	18	3.2	0.8	
	MD-GA (1:1)	18	18	–			
	GA-I (1:1)	–	18	18			
3 polymers	MD-GA-I (1:1:1)	12	12	12			
	MD-GA-I (1:2:1)	9	18	9			
	MD-GA-I (2:1:1)	18	9	9			
	MD-GA-I (1:1:2)	9	9	18			

2.2. Initial emulsions

Aqueous solutions were prepared by slow dissolution of the carrier(s) in water at 40 °C under mechanical stirring and, for the oil phase, α-tocopherol was mixed with olive oil under magnetic stirring. Emulsions were obtained using a rotor–stator homogenizer (AXR, Silverson Machines Ltd, Fr) to disperse the oil phase into the aqueous one. Emulsions were homogenized for 20 min with a rotation speed of 3500 rpm. Homogenization caused an increase of the emulsion temperature up to 60 °C that seemed to be favourable to obtain fine and homogeneous emulsions (size about 1 μm).

2.3. Spray drying

Initial emulsions were spray dried in a Niro Minor pilot scale spray dryer (Niro, Dk). It is a one step co-current spray dryer with an evaporative capacity comprised between 1 and 4 kg·h^{-1}. The emulsion feed flow rate was fixed to 57.6 g·min^{-1}, corresponding to a water flow rate of 34.6 g·min^{-1}. For spraying, a rotary wheel was used with a rotation speed of 25000 rpm (5 bar compressed air). Drying air was taken from the ambient by a fan (43 Hz) with a flow rate of 110 kg·h^{-1}. Its inlet and outlet temperatures (T_{IN} and T_{OUT}) were measured using thermocouples (K type). Exit moist air and dry powder were separated by a cyclone at the outlet of the chamber. The powder was collected continuously in glass jars where its temperature was measured, and it was stored in sealed refrigerated jars until analysis.

Assuming total removal of water during drying and no product loss, spray drying of 1 kg of initial emulsion should allow producing 400 g of dry emulsion (powders) containing 10% w/w encapsulated oil comprising 2% w/w α-tocopherol.

2.4. Emulsions and powders characterization

The density of emulsions (20 °C) was measured by pycnometry. Their apparent viscosity μ (Pa·s) and their rheological behaviour at 25 °C were measured using a rotational rheometer with coaxial cylinders (Rheomat R180, Lamy, GB). Apparent viscosity was deduced from shear stress values τ (Pa) measured for different velocity gradients γ (s^{-1}) imposed between the rotor and the stator (Newton's law $\mu = \tau \cdot \gamma^{-1}$).

The size and size distribution of emulsions and powders were measured by laser granulometry (Mastersizer 2000, Malvern, GB). For the emulsions, the analysis was performed in wet mode, dispersing a few drops of emulsion in deionized water (Hydro 2000). Powders were analyzed in dry mode (Scirocco 2000) with a compressed air pressure of 1 bar ensuring the dispersion of the particles.

[1] MD: maltodextrin DE12.
[2] GA: acacia gum.
[3] I: agave inulin.