

Encapsulation of oil in powder using spray drying and fluidised bed agglomeration

M. Fuchs, C. Turchiuli, M. Bohin, M.E. Cuvelier, C. Ordonnaud,
M.N. Peyrat-Maillard, E. Dumoulin *

ENSIA/UMR GenIAI, 1 Avenue des Olympiades, 91744 Massy Cedex, France

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Abstract

Many food active molecules such as antioxidants and aromas, exist in an oil form which may be easily oxidised. They are often prepared as a dosed, free-flowing powder, for storage protection and controlled release purposes. Oil encapsulation in powder was tested using a vegetable oil (VO) chosen as a model (5% w of dry matter) with a support of maltodextrin (MD) and acacia gum (AG) (ratio 3/2 w/w). Spray drying of a formulated aqueous emulsion (VO/MD/AG) led to small particles (<50 μm). Further, their size was increased (150 μm) by agglomeration in an air fluidised bed with spraying of water. Direct agglomeration of maltodextrin with an aqueous emulsion (VO/AG) represents a good process alternative, leading to particles of 200 μm . The two agglomerated powders consist of 5% of oil well dispersed in the support (MD/AG), with less than 0.5% on the particles surface. The protection against oil oxidation is good in comparison with unencapsulated oil. Also the agglomerated powders have suitable properties of flowability and wettability.

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1. Introduction

The encapsulation of active components in powders has become a very attractive process in the last decades, being adequate for food ingredients as well as for chemicals, drugs or cosmetics. The main objective is to build a barrier between the component in the particle and the environment. This barrier may protect against oxygen, water, light; avoid contact with other ingredients, in e.g. a ready meal; or control diffusion. The efficiency of protection or controlled release mainly depends on the composition and structure of the established wall

but also on the operating conditions during the production and use of these particles (temperature, pH, pressure, humidity). The barrier is generally made of compounds with chains to create a network, with hydrophilic and/or hydrophobic properties. The final powder has a specific composition regarding the active component, but it must also have good handling properties, mixing ability with water or other powders (size, shape, density). Flow properties of a powder may also depend on the properties of the wall material, and e.g. on the relative amount of lipids remaining unencapsulated on the surface of particles. Such a powder may require specific stability properties during heating or freezing processes, and for an optimal release of the encapsulated active component.

The usual *encapsulating agents* are proteins (e.g. milk, gelatine), gums (e.g. acacia, alginate), carbohydrates

* Corresponding author. Fax: +33 1 69935185.

E-mail address: dumoulin@ensia.fr (E. Dumoulin).

(e.g. sucrose, maltodextrins, modified starch, cyclodextrins, cellulose), lipids, fats, waxes, lecithins (emulsifiers) and fibers. Important parameters are temperature and humidity during processing and during the storage, on the one hand, and the end-use properties on the other. The initial formulation leading finally to powders, will be chosen to predict and to control physical phenomena like drying, melting, glass transition, crystallisation, caking. Blending two or more agents may provide the desired characteristics. Commercially available micro-encapsulated flavourings and ingredients include carrier materials which have been designed to suit the end foodstuff into which they will be added (Sinton, 1998).

Lipid encapsulation may be useful to retard lipid auto-oxidation, or as a carrier to solubilise substances like flavour, bitter components (Matsuno & Adachi, 1993). In the case of oil encapsulation, acacia gum is known for its emulsification role due to the presence of proteins. The gum is adsorbed at the oil/water interface, the recommended ratio oil/gum being inferior to 0.5 to lead to a desired droplet size ($<1 \mu\text{m}$). In the emulsion, used in spray drying, the rest of the wall material may be a less expensive water soluble carbohydrate (McNamee, O'Riordan, & O'Sullivan, 2002). During storage, volatile substances are retained by gums as long as the capsule structure remains protective, whatever encapsulation techniques (such as spray drying, extrusion, freeze drying or drum drying) used (Beristain, Azuara, Tamayo, & Vernon-Carter, 2003). The protection against oxidation of methyl linoleate encapsulated with acacia gum by spray drying and freeze drying, depends on the relative humidity of the environment (Minemoto, Adachi, & Matsuno, 1997). Extrusion is used to encapsulate minerals and vitamins in oil (saturated fat) in a glassy matrix (polysaccharides), with further grinding if a powder form is required (Van Lengerich & Lakis, 2002). An oxygen sensitive oil (tuna oil) is encapsulated in a mix of protein and carbohydrates (casein/sugar). These two components have been pre-heated together to form Maillard reactions products, which are antioxidants, giving a better resistance to oxidation. And an additional coating with triglycerides or starch is envisaged (Augustin & Sanguansri, 2003). For microcapsules with high oil content (40–70%), proteinaceous materials as sodium caseinate are effective wall materials, used in combination with maltodextrin, gums and emulsifier (Dian, Sudin, & Yusoff, 1996). Encapsulation of high-carotenoid canola oil and vitamin E in α -cyclodextrin increases the bioavailability of antioxidants in a water-soluble form and gives protection against heat, light, oxidation (Basu & Del Vecchio, 2001). α -tocopherol, a source of vitamin E, is liquid at room temperature. Its formulation in a solid dosage form is difficult but effective using a lipid-based excipient (Barker et al., 2003).

The development of commercial powdered ingredients for use in the preparation of food products, enriched with specific fatty acids, was observed. These fatty acids are very sensitive to oxidation, hereby leading to off-taste in the finished product. Microencapsulation may be used in combination with antioxidants and/or masking flavours. The addition of α -tocopherol (100 ppm) delays the oxidation of fish oil encapsulated in sodium caseinate with carbohydrates (25–50% w/w of oil). However, additional measures (packaging, neutral atmosphere) are recommended (Hogan, O'Riordan, & O'Sullivan, 2003).

Among several encapsulation techniques (Shahidi & Han, 1993), the *spray drying* of a formulated emulsion/suspension/solution is the most widely used to produce a powder where the active component is well dispersed in the matrix. In the case of emulsions, the wall material acts as a stabiliser and the spray-dried emulsion is able to disperse again in water. Usually the fraction of oil on the surface of powder, unprotected against oxidation, is low, and does not seem to be significant enough to influence shelf life (Buffo & Reineccius, 2000). Fish oil rich in polyunsaturated fatty acids is fixed onto a solid matrix of carbohydrates (modified starch, maltodextrin, cyclodextrin). Emulsification and spray drying are used to have a composition of 25–30% oil in matrix (Barrier & Rousseau, 1998). Encapsulation of liquid flavourings extract of a plant was accomplished by mixing this extract (e.g. garlic) with a 20% solution of acacia gum. After homogenisation, the encapsulated liquid mixture was spray dried. The heat stability in bakery products was attested (Owusu-Ansah & Green, 1999). Spray drying encapsulation of carotenoid pigments in gelatine or starch with sucrose provides a protection effect against degradation (Robert, Carlsson, Romero, & Masson, 2003).

The spray-dried powders usually have a small particle size, 10–100 μm , with poor handling and reconstitution properties. Some anti-caking agent may be added to improve the flowability, e.g. silica (2%), for spray-dried butteroil encapsulated in lactose, sucrose and all purpose flour (Konstance, Onwulata, & Holsinger, 1995). Very often a fluidised bed is associated with the spray dryer for further agglomeration of particles (e.g. milk), to improve or to change some of the properties like instantisation, or to decrease the content in fines. Solid bridging between particles, with the same material as the powder or a different material, occurs when spraying respectively water or a solution of agglomerating agent. Those agents are similar to those used for spray drying encapsulation: acacia gum, maltodextrins, starches with e.g. orange oil, vitamin A (Buffo & Reineccius, 2001; Buffo, Probst, Zehentbauer, Luo, & Reineccius, 2002). Spray drying is also used to obtain a stable dry emulsion of coconut oil (40%) encapsulated in HPMC which is both a carrier and an emulsifier. Wet granulation of

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