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Encapsulation of folic acid in food hydrocolloids through nanospray drying and electrospraying for nutraceutical applications

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ABSTRACT

In this work, two different technologies (electrospraying and nanospray drying) were evaluated for the encapsulation of folic acid using both a whey protein concentrate (WPC) matrix and a commercial resistant starch. The morphology of the capsules, molecular organization of the matrices upon encapsulation, encapsulation efficiency, and stability of the folic acid within the capsules under different storage conditions and upon thermal exposure were studied. Results showed that spherical nano-, submicro- and microcapsules were obtained through both techniques, although electrospraying led to smaller capsule sizes and to an enhanced control over their size distribution. Greater encapsulation efficiency was observed using WPC as encapsulating matrix, probably related to interactions between the protein and folic acid which favoured the incorporation of the bioactive. The best results in terms of bioactive stabilization in the different conditions assayed were also obtained for the WPC capsules, although both materials and encapsulation techniques led to improved folic acid stability, especially under dry conditions.

1. Introduction

The encapsulation of nutraceutical and functional ingredients is an area of increased interest over the last years which seek to protect these products from adverse environmental conditions and, thus, increase their shelf-life and assure their health-promoting properties. Moreover, the encapsulation of these components also enables their incorporation into different food matrices which results in novel functional food products with potential health benefits.

A variety of techniques have been used to encapsulate functional components, such as nanoemulsions (Silva, Cerqueira, & Vicente, 2012), coacervation (de Conto, Grosso, & Gonçalves, 2013; Tamjidi, Nasirpour, & Shahedi, 2012), extrusion methods (Li, Chen, Sun, Park, & Cha, 2011), fluidized bed coating (Zuidam & Shimoni, 2010), spray cooling (Gibbs, Kermasha, Alli, & Mulligan, 1999) or spray drying (Murugesan & Orsat, 2012). Among these methods, spray drying is nowadays the most common and cheapest technology in the food industry to produce microencapsulated additives for food applications (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). The active material to be encapsulated through the spray drying technique is dispersed in a carrier polymer solution which is atomized into small droplets. The solvent is evaporated using a warmed gas and the resulting solid capsules are collected as dry powder (Gharsallaoui et al., 2007; Gibbs et al., 1999). It is worth noting that this technology can be used with aqueous solutions, thus avoiding the use of organic solvents which could generate toxicity problems in contact with food. Nevertheless, it needs relatively high temperatures to eliminate the water from the polymeric/biopolymeric solutions, fact that could affect the stability of the bioactive ingredient. Apart from these well-known encapsulation techniques, electrospinning has recently arisen as an alternative technology that can also be used for encapsulation (Torres-Giner, Martinez-Abad, Ocio, & Lagaron, 2010; Lopez-Rubio & Lagaron, 2012). Besides being a very simple technique, some advantages of electrospinning for encapsulation include that neither temperature nor organic solvents are needed, thus, being an ideal method for protecting sensitive encapsulated ingredients. Electrospinning makes use of high voltage electric fields to produce electrically charged jets from viscoelastic polymer solutions which on drying, by the evaporation of the solvent, produce ultrathin polymeric structures (Li & Xia, 2004). The electrospun nanostructures morphology and diameter are affected by the solution properties and by the process parameters and, for certain materials, reduced size capsules can be obtained when

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adjusting both. In this case, the electrospinning process is normally referred to as "electrospraying" due to the non-continuous nature of the structures obtained (Lopez-Rubio & Lagaron, 2012). Capsules and, thus, electrospraying, are generally preferred for food and nutraceutical applications, since apart from facilitating handling and subsequent incorporation into different products, they also present greater surface to volume ratio and, thus, are expected to have better release profiles than fibers (Hong, Li, Yin, Li, & Zou, 2008).

Folic acid is an essential micronutrient which cannot be synthesized by humans and, thus, it must be ingested through the diet (Lopera, Guzmán, Cataño, & Gallardo, 2009). Folic acid is the synthetic form of folates, a broad group of compounds with vitamin functionality (Bakhshi, Nangrejo, Stride, & Edirisinghe, 2013). Specifically, folic acid is a water-soluble vitamin which is vital for a variety of physiological functions in humans. It plays an important role in the prevention of neural tube defects in infants and might decrease the likelihood of developing vascular diseases and some cancers (Liang, Zhang, Zhou, & Subirade, 2013). According to these beneficial effects, the European Regulations (Regulations 1924/2006; 1925/2006) allow the addition of folic acid in food and when its content provides more than 15% of the recommended daily amount, eight different health claims related to the beneficial effects of this vitamin are allowed in the food label (Regulation 432/2012). However, folic acid undergoes degradation reactions when it is exposed to light, temperature, moisture, acid or alkaline medium and oxygen atmosphere (Lopera et al., 2009). Therefore, the encapsulation of this bioactive ingredient is a plausible option to improve its stability and to assure its bioactivity within the food product during commercialization.

Regarding the matrix materials employed for encapsulation, food hydrocolloids are very convenient for nutraceutical and food applications, since many of them are soluble in aqueous solutions, thus, avoiding toxicity problems. Specifically, in the case of folic acid, there are some works which have shown the feasibility of spray drying for the encapsulation of this bioactive molecule within food hydrocolloids obtaining proper encapsulation efficiencies (Lopera et al., 2009). However, electrospinning from hydrocolloidal aqueous solutions has proven difficult due to several factors such as the polycationic nature or the low chain flexibility of these materials which complicates chain entanglements (essential for fiber formation) (Kriegel, Kit, McClements, & Weiss, 2009). Moreover, the high surface tension of water, as well as, the ionization of water molecules at high voltages in an air environment, also complicates the electrospinning process. Therefore, synthetic polymers such as polyethylene oxide (PEO) (Alborzi, Lim, & Kakuda, 2013) or multiple stages processes (Bakhshi et al., 2013) have been employed to date for the folic acid encapsulation through the electrospinning technology. Recent works have demonstrated that it is possible to obtain hydrocolloid-based encapsulation structures using electrospraying through the proper adjustment of the aqueous solution properties (mainly surface tension and viscosity) upon addition of several additives (surfactants and gums) (Pérez-Masiá, Lagaron, & Lopez-Rubio, 2014a, 2014b).

The aim of this work was to compare the more traditional spray drying technique (but using a novel nanospray drying device able to obtain smaller encapsulation structures) with the electrospraying methodology for folic acid encapsulation using two different food hydrocolloid matrices (a whey protein concentrate and a commercial resistant starch). Initially, whey protein concentrate (WPC) was used since it has excellent functional characteristics, it is a low cost ingredient and it has proven useful for the encapsulation of several functional ingredients through electrospinning (Lopez-Rubio & Lagaron, 2011, 2012). Additionally, a commercial resistant starch (derived from corn starch) with trade name Fibersol was also employed for folic acid encapsulation, as it was also the aim to compare the performance, in terms of protection, of a protein and a carbohydrate matrix. Specifically, a resistant starch was used as it could provide an additional beneficial prebiotic effect (Topping & Clifton, 2001). Both materials are dispersible in aqueous solutions and, thus, they are very convenient for nutraceutical applications. The encapsulation structures obtained through the two methodologies were characterized, and a comparative evaluation of the encapsulation efficiency and folic acid stability, under different storage conditions, and after applying a heating process were ascertained.

2. Materials and methods

2.1. Materials

Whey protein concentrate (WPC) was kindly donated by ARLA (ARLA Food Ingredients, Viby, Denmark). Under the commercial name Lacprodan® DI-8090, the composition per 100 g of product consisted of ~80 g of protein, ~9 g of lactose, and ~8 g of lipids, being the rest water and minerals like sodium and potassium. The commercial resistant starch was Fibersol® (www.fibersol.com) commercial grade, manufactured by ADM/Matsutani (Iowa, USA). Guar Gum was purchased at Capers Community Markets (Vancouver, Canada). Folic acid (>97% purity) and the surfactant Span 20 were supplied by Sigma–Aldrich (Spain). All products were used as received without further purification.

2.2. Preparation of the solutions

The solutions were prepared depending on the encapsulation technology by dissolving 0.4% or 20% w/v of the matrix hydrocolloids in water for the spray drying or the electrospraying technique, respectively. The concentration of the matrices used in the solutions had been previously optimized, so as not to block the spraying head (in the case of spray drying) and to get the required chain entanglements for capsule formation (in the case of electrospraying). Additionally, 0.5 wt.% of Guar gum with respect to the biopolymer matrix was incorporated, together with the hydrocolloid, in the resistant starch solutions. Span 20 was also added to the solutions to attain 5 wt.% with respect to the hydrocolloids weight. These solution compositions were selected as it had been previously optimized for these specific encapsulating matrices (Pérez-Masiá, Lagaron, & Lopez-Rubio, 2014b). When folic acid was incorporated, 1.5 wt.% of the bioactive with respect to the polymers weight was added. The solutions were stirred at room temperature until homogeneous dispersions of all the components were obtained.

2.3. Encapsulation through spray drying

The solutions with and without folic acid were spray-dried using a Nanospray-dryer B-90 (Büchi, Switzerland) with a 0.7 μm membrane cap. The solutions were introduced into the equipment through a silicone wire, which was connected to the spraying head of the equipment. The air flow was ~140 L/h with an inlet and outlet temperatures of 90 °C and 45 °C, respectively.

2.4. Encapsulation through electrospraying

The electrospinning apparatus, equipped with a variable high-voltage 0–30 kV power supply, was a Fluidnatek® LE-10 purchased from BioInicia S.L. (Valencia, Spain). Solutions with and without folic acid were introduced in a 5 mL plastic syringe and were electrospun under a steady flow-rate using a stainless-steel needle. The needle was connected through a PTFE wire to the syringe. The