Nano-particle encapsulation of fish oil by spray drying

Seid Mahdi Jafari\textsuperscript{a,}\textsuperscript{*}, Elham Assadpoor\textsuperscript{a}, Bhesh Bhandari\textsuperscript{b}, Yinghe He\textsuperscript{c}

\textsuperscript{a} Department of Food Science and Technology, Gorgan University of Agricultural Sciences and Natural Resources, Gorgan, Iran

\textsuperscript{b} School of Land and Food Sciences, University of Queensland, Brisbane, Australia

\textsuperscript{c} School of Engineering, James Cook University, Townsville, Australia

Received 26 August 2007; accepted 4 November 2007


corresponding author. Tel./fax: +98 171 4426 432.
E-mail address: smjafari@gau.ac.ir (S.M. Jafari).

Abstract

Nano-particle encapsulation by spray drying was undertaken by preparing sub-micron emulsions via high energy emulsifying techniques, namely Microfluidization and Ultrasonication. The encapsulation efficiency of fish oil as a core material was investigated. The attention was given to the surface oil content and surface oil coverage of encapsulated powders which are very significant parameters in the encapsulation process. Maltodextrin combined with a surface-active biopolymer (modified starch or whey protein concentrate) at a ratio of 3:1 were used as the wall material. Results showed that Microfluidization was an efficient emulsification technique resulting in fish oil encapsulated powder with the lowest unencapsulated oil at the surface of particles, mainly due to its capability to produce emulsions at the nano-range (d$_4$ of 210–280 nm).

Keywords: Surface oil content; Nano-emulsion; Emulsification; Surface oil coverage; Microfluidization

1. Introduction

Encapsulation is a rapidly expanding technology with a lot of potential in different areas including pharmaceutical and food industries. It is a process by which small particles of core materials are packaged within a wall material to form microcapsules (Gouin, 2004; Thies, 2001). One of the common techniques to produce encapsulated products is spray drying, which involves conversion of liquid oils and flavours in the form of emulsions into dry powders, as an important application of microencapsulation in the food industry (Bhandari, 2005; Reineccius, 2004). Over the last few years, the main emphasis of microencapsulation of food flavours and oils has concentrated on improving the encapsulation efficiency during spray drying, that is preventing volatile losses and extending the shelf-life of the products by minimizing the amount of unencapsulated oil at the surface of powder particles (Desai & Park, 2005; Madene, Jacquot, Scher, & Desobry, 2006; Reineccius, 2001). This is intended to produce high quality encapsulated powders with maximum recovery. The properties of wall and core materials as well as the emulsion characteristics and drying parameters are the factors that can affect the efficiency of encapsulation.

Emulsification plays a key role in optimising the encapsulation efficiency of food flavours and oils (Liu, Furuta, Yoshii, & Linko, 2000; Liu et al., 2001). It has been well documented that emulsion droplet size has a pronounced effect on the encapsulation efficiency of different core materials during spray drying (Risch & Reineccius, 1988; Soottitantawat, Yoshii, Furuta, Ohkawara, & Linko, 2003; Soottitantawat et al., 2005). These reports clearly show that reducing emulsion size can result in encapsulated powders with higher retention of volatiles and lower content of unencapsulated oil at the surface of powder particles. The presence of oil on the surface of the powder particles is the most undesirable property of encapsulated powders. This surface oil not only deteriorates the wettability and dispersability of the powder (Millqvist-Fureby, Elofsson, & Bergenstahl, 2001; Vega, Kim, Chen, & Roos, 2005),...
but also is readily susceptible to oxidation and the development of rancidity. Workers such as Kim, Chen, and Pearce (2002, 2005a, 2005b) by analysing industrial dairy powders with X-ray photoelectron spectroscopy (XPS), have found that there is a relatively high surface fat coverage on these powders (e.g., 53% for WPC powders). In another study, Keogh and O’Kennedy (1999) showed that milk fat encapsulated powders with whey proteins had more than 30% fat coverage. Some workers have also shown that the type of fat has a strong influence on the level of surface fat (Kim et al., 2005b; Millqvist-Fureby, 2003). Recently Vega and Roos (2006) have made a comprehensive review on spray-dried dairy emulsions with an emphasize on surface composition.

Much of the work in this area has been done by emulsions having a droplet size of more than one micron and the application of sub-micron (nano) emulsions in encapsulation of oils and flavours is scant in the literature. By the advent of modern emulsification systems and their potential application in encapsulation of food ingredients, understanding the influence of emulsion size in nano-range on surface oil content and coverage during spray drying is essential (Jafari, Assadpoor, Bhandari, & He, 2007b). In fact, there is no clear cut evidence on how sub-micron or nano-emulsions can improve the encapsulation efficiency of food flavours and oils. Recently, nano-emulsions have attracted considerable attention in various industrial fields including cosmetics, pharmaceuticals and agrochemicals (Jafari, He, & Bhandari, 2006; Jafari, He, & Bhandari, 2007a; Solans, Izquierdo, Nolla, Azemar, & Garcia-Celma, 2005; Tadros, Izquierdo, Esquena, & Solans, 2004). These emulsions are kinetically stable systems with very small droplet sizes that can be of real benefit for encapsulation purposes, since the stability and other features of the feedstock emulsion such as droplet size and distribution play a critical role on the retention and surface oil content of the product. Therefore, the objectives of this work are to determine the influence of sub-micron emulsions produced by different emulsification methods on encapsulation efficiency and investigate the encapsulated powder properties after spray drying for different emulsion droplet sizes and surface-active biopolymers. In this work, we have referred the encapsulation as “nano-particle encapsulation” since the core material in nano-size range will be encapsulated into the matrix of micron size powder particles.

2. Materials and methods

2.1. Materials

In this study, fish oil (HiDHA 25N, Nu-Mega Ingredients, Brisbane, Australia) was used as the core material (\( \rho = 850 \text{ kg/m}^3, \eta = 86 \text{ mPa s} \) at 25 °C, RI = 1.483). The wall material was an aqueous solution of a modified starch (Hi-Cap 100, National Starch and Chemical, NSW, Australia) and/or whey protein concentrate (WPC) (ALACEN, New Zealand Milk Products, Auckland, New Zealand) in a combination with maltodextrin (DE 16-20, Fieldose 17-C AP, Penford Limited, NSW, Australia). Analytical grade hexane and petroleum ether (BP 40–60 °C) were purchased from Sigma Chemicals Company (Sydney, Australia). Distilled water was used for the preparation of all solutions. All general chemicals used in this study were of analytical grade.

2.2. Preparation of emulsions

Hydrated solution of emulsion continuous phase was prepared by dissolving wall material powders in distilled water using a high speed blender (Model RW 20.n, IKA Works, Malaysia). They were produced one day before emulsification and kept overnight in a shaking water bath (Ratek Instruments, VIC, Australia) to warrant a full saturation of the polymer molecules. For starch-based biopolymers, the temperature of water bath was adjusted to 60 °C while for proteins, they were kept at ambient temperature to avoid changes due to temperature. In the case of WPC, their solutions were prepared by dispersing the desired amount of their powder (10 wt%) into buffer solution (5 mM phosphate buffer, pH 7). The pH of WPC solutions was adjusted back to pH 7.0 using 1 M HCL if required. The total concentration of dissolved solid was 40% (w/w) that was composed of 30 wt% maltodextrin and 10 wt% of emulsifying ingredients including one of the biopolymers of Hi-Cap or WPC.

All emulsions produced were of the oil-in-water type and being prepared in two stages: (a) pre-emulsions were obtained by a rotor–stator system (Model L2R, Silverson Machines Ltd., UK). The Silverson is a typical colloid mill with a stator composed of a metal grating in which, 2 mm holes are drilled. The core material (fish oil) in the ratio of 1:4 (core:wall) was progressively added to the continuous phase during pre-emulsion preparation and stirred for 10 min at the highest speed. (b) These coarse emulsions were then further emulsified using a Microfluidizer (Model M-110 L, Microfluidics, USA) at 60 MPa for one cycle, or an 24 KHz Ultrasound probe (Dr. Hielscher series, Model UP 400S) with 22 mm diameter at the highest power for 100 s. More details about emulsification conditions were presented in our previous work (Jafari & He et al., 2007a; Jafari & Assadpoor et al., 2007b). Sodium azide (0.02 wt%) was added to the emulsions as an antimicrobial agent. For each emulsifying device, about 1000 mL sample was prepared for the production of encapsulated powders by spray drying.

2.3. Spray drying

The infeed emulsions were transformed to encapsulated powder in a pilot-plant spray drier (Model SL 20, Saurin Group of Companies, Victoria, Australia). It was composed of a cylindrical chamber with a 1200 mm diameter and 2000 mm height, followed by a conical chamber of 500 mm high with a 60° angle. The dryer had a water evap-