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Volatile compounds and physicochemical characteristics during storage of microcapsules from different fish oil emulsions

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ABSTRACT

Spray-drying of double (DE) and multilayered (ME) fish oil emulsions, was used to produce two different types of microcapsules (DM and MM, respectively). Stability of emulsions and physicochemical characteristics, oxidative stability and volatile profile of the microcapsules at initial time and after 1 month of storage at 20 and 4 °C were analyzed. ME showed better stability and held higher amount of fish oil than DE (2.5 vs 0.625% w/w). Microencapsulation yield was similar for both types, whereas moisture, microencapsulation efficiency and oxidative stability were higher for MM. The type of microcapsule influenced the volatile profile and specifically the 28 selected volatiles related to oxidation. Both types of microcapsules appear as feasible alternatives to the bulk fish oil as a way to provide ω-3 storage and for enrichment purposes, but MM seems to be more appropriate in terms of oxidation during storage.

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1. Introduction

Polyunsaturated fatty acids (PUFA), mainly eicosapentaenoic acid (EPA, C20:5ω-3) and docosahexaenoic acid (DHA, C22:6ω-3), are compounds with bioactive properties and several beneficial effects for human health (McClements et al., 2007). The importance of increasing EPA and DHA intake has been recognized by several health agencies worldwide (Barrow et al., 2007). As a result, consumer's interest in boosting the intake of ω-3 fatty acids (FA) has increased. The main dietary source of ω-3 FA is fish oil. However, as a consequence of current trends in eating habits, this source is not enough to reach the recommended daily dose of EPA and DHA (Taneja and Singh, 2012). For this reason, there is a growing interest in the development of functional foods and supplements as a source of EPA and DHA (McManus et al., 2011).

Several researchers have examined the possibility of incorporating fish oil (and thereby the beneficial ω-3 FA) into food products (Carneiro et al., 2013). Nevertheless, ω-3 FA are exceptionally susceptible to oxidation processes. The breakdown of the ω-3 chain during oxidation involves nutritional loss and a detrimental sensory, with an unacceptable rancidity and fishy off-flavour (Taneja and Zhu, 2006). This fact leads to the main challenge for the production of ω-3 FA

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enriched products: the prevention of lipid oxidation (Jacobsen, 2008). Microencapsulation of ω-3 by spray-drying has been suggested as a strategy to solve these drawbacks. This technique involves the production of an emulsion by means of a homogenization process, which allows the emulsifiers adsorb the oil droplets, reducing the interfacial tension and producing a protective layer that avoids droplet aggregation. This simple method leads to traditional emulsions with only a single layer of emulsifier around every single oil droplet, namely monolayered emulsions. Alternative strategies, such as the modification of the emulsion preparation to obtain multiple or multilayer emulsions have also been developed (Grigoriev and Miller, 2009; Shaw et al., 2007).

Double emulsions occur when a single emulsion is itself homogenized to form droplets within another (added) continuous phase of the opposite hydrophobicity/hydrophilicity to that making up the continuous phase of the original single emulsion (and similar to the discrete phase of the original single emulsion). One thus produces "droplets within droplets". For example, in an "oil-in-water-in-oil" double emulsion (O-W-O), there are very small droplets of oil inside larger droplets of water which are themselves surrounded by a continuous oil phase. The encapsulation of oil by this technique is prepared as an oil-in-water-in-oil (O1/W/O2) emulsion, in which the encapsulated oil consists on the inner oil phase (O1) (Edris and Bergnstahl, 2001; Dwyer et al., 2013; O'Dwyer et al., 2013). It requires the combination of two emulsifiers: an emulsifier of hydrophilic nature for the stabilization of the O1/W interface and an emulsifier of hydrophobic nature for the stabilization of the O2/W interface. Multilayered emulsions can be defined as emulsions in which lipid droplets are surrounded by multiple layers instead of a single layer of coating material, these layers being formed by a combination of an emulsifier and one or more polyelectrolyte of opposite charges. This technique is known as "layer-by-layer" (LbL) and has been recently developed (Grigoriev and Miller, 2009; Jiménez-Martín et al., 2015; Klinkesorn et al., 2005a, b, c, 2006; Shaw et al., 2007).

Microencapsulation of fish oil as a source of ω-3 by spray-drying can be considered successfully performed when high retention of the core material is achieved and the majority of the oil is encapsulated, with low amounts of surface oil on the microcapsule powders (Jafari et al., 2008). This objective can be reached by the use of wall and core materials with adequate properties, optimizing formulation and preparation of emulsions and adjusting the drying process conditions (Gharsallaoui et al., 2007). Selection of the emulsifier and coating wall materials represents a very important step during the optimization of the microencapsulation by spray-drying and has a main influence on the final efficiency and stability of the global process (Anwar and Kunz, 2011). Emulsifiers commonly used in the food industry are proteins, polysaccharides, phospholipids and small molecule surfactants that differ in their emulsifying properties and the stability of the produced emulsions. As for the coating materials, polysaccharides and proteins are the most used materials. It is important to obtain a balance between economical aspect, ease of use and encapsulating properties.

Most studies on fish oil microcapsules by spray-drying are focused on their physical stability rather than on their volatile profile. Volatile compound profile or specific volatiles extracted by solid-phase microextraction (SPME) and analyzed with gas chromatography-mass spectrometry (GC/MS) appears as a useful tool for addressing lipid oxidation (Jacobsen, 1999). The complete volatile profile of the microcapsules, besides providing information of the oxidation level of the powders, can be also very useful as an approach to predict their flavor. Indeed, a recent study showed that sensory properties of fish oils were predicted using key oxidative volatiles, the results being comparable to the evaluation with a sensory panel (Ritter and Budge, 2012).

The stabilization of ω-3 FA by spray-drying microencapsulation has been assessed by complementary methods, such as peroxide value (PV), TBARs (Thiobarbituric Acid Reactive substances) index (Jiménez-Martín et al., 2015) and p-anisidine value determination (Omar et al., 2009), headspace propanal determination (Augustin et al., 2006) and non-isothermal differential scanning calorimetry (Pedroza-Islas et al., 2002). Dobarganes et al. (Dobarganes et al., 2009) also applied and validated the accelerated test Rancimat to evaluate the oxidative stability of dried microencapsulated oils.

Several authors have studied different microencapsulated fish oils (menhaden oil, tuna oil) using multilayered emulsions (LbL technique) and lecithin-chitosan emulsifier combination with different carbohydrates as coating material (Jiménez-Martín et al., 2015; Klinkesorn et al., 2005a, b, c, 2006; Shaw et al., 2007). On the other hand, to date, researches on microencapsulation of O/W/O emulsions that can be found in the literature are scarce, and, to the best of our knowledge, only two works have tackled this issue. In the study of Edris and Bergnstahl (Edris and Bergnstahl, 2001), orange oil was encapsulated in the inner compartment of a O1-W-O2 double emulsion, where O1 was orange oil, W was water and O2 was vegetable oil and was secondarily coated with lactose and caseinate as wall materials using spray-drying. Liao et al. (Liao et al., 2012) encapsulated fish oil in a O/W/O double-emulsion produced with succinic acid deamidated wheat gluten (SDWG) followed by heat-polymerization of emulsified SDWG.

This work was aimed to study the effect of microencapsulating fish oil by spray-drying from multiple and multilayered emulsions and to provide for the first time a comparison of both types of emulsions and their resulting microcapsules in terms of physicochemical characteristics and oxidative stability, including the analysis of the volatile profile by SPME-GC/MS during storage at ambient and refrigeration temperatures.

2. Material and methods

2.1. *Material*

Fish oil extracted by cold pressing from cod liver (kindly provided by Biomega Natural Nutrientes S.L., Galicia, Spain) was used as source of ω-3 FA (5.96% EPA, 25.83% DHA, and 0.02% BHT). Sodium caseinate (kindly provided by Anvisa S.A., Madrid, Spain), lactose monohydrate (Scharlau, Sentmenat, Spain), extra virgin olive oil (Hacendado, Madrid, Spain), polyglycerolpolyricinoleate (kindly provided by Cargill, Barcelona, Spain), soybean lecithin (Across Organics, Madrid, Spain), chitosan with 95% of deacetylation (Chitoclear FG 95, kindly provided by Trades, Murcia, Spain), maltodextrin with a dextrose equivalent of 12% (Glucidex 12, Roquette, Lestrem, France) and glacial acetic acid (Scharlau, Barcelona, Spain) were used for the preparation of the emulsions. Hydrochloric acid and petroleum ether (Scharlau, Barcelona, Spain) were used for the oil extraction of the microcapsules. For the oxidative stability, 1-butanol and isopropanol (Scharlau, Barcelona, Spain) were purchased as solvents and 2-thiobartituric acid (TBA, Serva, Heidelberg, Germany), trichloroacetic acid (Fisher,

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