LWT - Food Science and Technology 92 (2018) 523–530

Contents lists available at ScienceDirect

LWT - Food Science and Technology

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

Microencapsulation of omega-3 polyunsaturated fatty acids and astaxanthin-rich salmon oil using particles from gas saturated solutions (PGSS) process

Monjurul Haq[a,b], Byung-Soo Chun[a,*]

[a] Department of Food Science and Technology, Pukyong National University, 45 Yongso-ro, Nam-Gu, Busan 48-513, Republic of Korea
[b] Department of Fisheries and Marine Bioscience, Jessore University of Science and Technology, Jessore 7408, Bangladesh

ARTICLE INFO

Keywords:
Microencapsulation
PGSS
Salmon oil
ω-3 PUFAs
Astaxanthin

ABSTRACT

Salmon oil rich in omega-3 polyunsaturated fatty acids and astaxanthin was microencapsulated in polyethylene glycol-6000 using particles from gas saturated solutions process. The process conditions viz. temperature (45–55 °C), pressure (15–25 MPa), oil and polymer ratio (1:2.5–1:10), and nozzle size (300–500 μm) were optimized for maximizing encapsulation efficiency. The maximum encapsulation yield of 79.20% was obtained in the microparticle produced at a temperature of 50 °C, pressure of 25 MPa, oil and polymer ratio of 1:5, and nozzle diameter of 400 μm. Astaxanthin content of 40.60 μg/g oil in microparticle; bulk density of 0.26%; cohesiveness and flowability measured by the Carr index of 12.76, and wettability of 5.91 min of the microparticles were found. Scanning electron microscopic analysis showed microparticles of different morphologies, from spherical or elongated to amorphous-shaped, with the size of the microparticles between 0.37 μm and 449 μm. Fourier transform infra-red spectroscopy spectra and fatty acid compositions of salmon oil before and after microparticle formation suggested that there was no remarkable alteration due to the PGSS process. Microparticles showed significant thermogravimetric stability up to 350 °C, and in vitro release of oil in fluids stimulating gastric conditions was faster than in distilled water.

1. Introduction

Omega-3 polyunsaturated fatty acids (ω-3 PUFAs) are specially characterized by their availability mostly in marine sources, and they exert crucial health functions, which cannot be substituted by fatty acids of other origins. Many researches have proven the health benefit of ω-3 PUFAs as playing an essential role for the proper growth and normal development of brain tissues and the nervous system; exerting positive effects on coronary heart disease; controlling hypertension; decreasing rheumatoid arthritis, diabetes, Alzheimer's, clinical depression, and anxiety; and being advantageous for cancer, inflammatory and immune disorders (Correa, Peixoto, Goncalves, & Cabral, 2008; Su, Huang, Chiu, & Shen, 2003; Yashodhara et al., 2009). Therefore, interest regarding supplementing ω-3 PUFAs in food industries and the development of pharmaceutical products are growing fast. Additionally, carotenoid pigment astaxanthin is related with the reduction of age related macular degeneration, ischemic problems, exhibits very strong antioxidant activity (Haq, Ahmed, Cho, & Chun, 2017a), and crucial in preventing cancer and cardiac problems, and helping in the enhancement of the immune response and liver functions (Nishino et al., 2005; Pashkow, Watumull, & Campbell, 2008).

Both the ω-3 PUFAs and the astaxanthin pigment are highly unstable and prone to oxidation, especially when in contact with atmospheric oxygen at elevated temperatures, require measures to keep the quality intact. Because the oxidation process not only damages nutritional value of fish oil but also generates toxic compounds and off-flavors (Wu, Zhang, Xue, Xue, & Wang, 2017). In this case, microencapsulation may be a suitable approach to overcome the above mentioned obstacles and ensure good quality ω-3 PUFAs and astaxanthin-rich salmon oil to consumers. In this process, the targeted core liquid material is enclosed inside small capsules that has superior stability against light and oxidative degradation and that is easier to apply in various food matrices (Karim et al., 2017). A number of conventional techniques have been applied for fish oil encapsulation, such as spray drying, liquid anti-solvent crystallization, freeze-drying, and milling. However, there are several drawbacks to these processes like the production of coarse particles, product degradation due to thermal or mechanical stress, contamination with undesired harmful solvents or toxic substances, etc. Therefore, researchers are investigating alternative supercritical fluid based techniques to overcome these drawbacks (Martin & Cocero, 2008), and recently, some promising technologies such as particles from gas saturated solutions (PGSS), rapid

* Corresponding author.
E-mail address: bschun@pknu.ac.kr (B.-S. Chun).

https://doi.org/10.1016/j.lwt.2018.03.009
Received 28 November 2017; Received in revised form 4 February 2018; Accepted 4 March 2018
Available online 06 March 2018

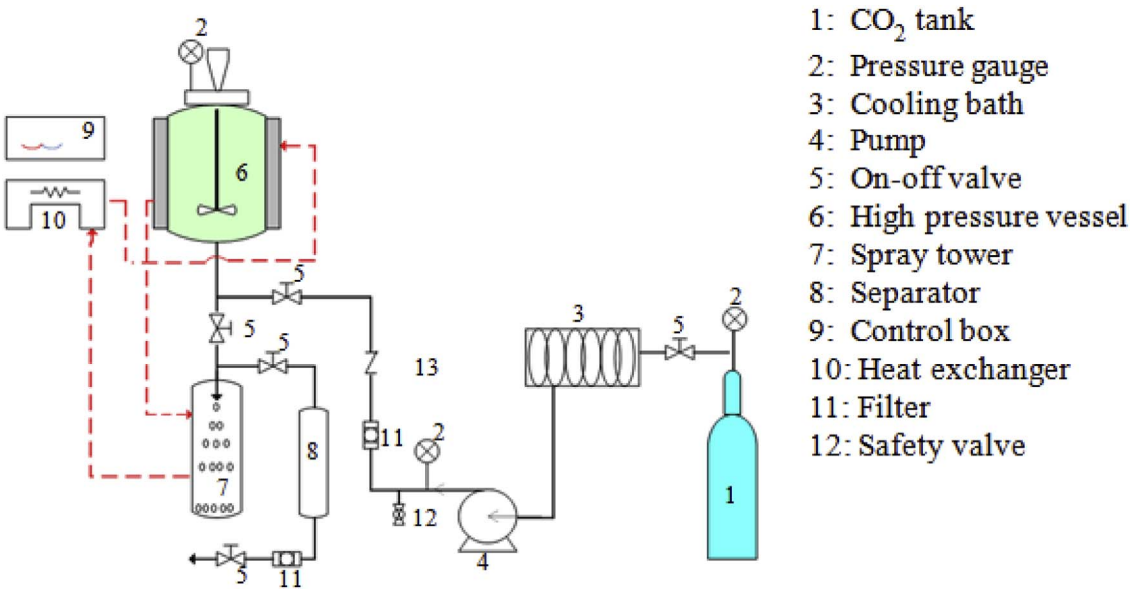

Fig. 1. Schematic diagram of PGSS instrument used in this study.

expansion of supercritical solutions (RESS), and supercritical anti-solvent (SAS) are being used for microencapsulation of bio-functional compounds. These techniques are advantageous as they require less energy, are cheaper, and the final product is free from harmful solvents (Yun, Lee, Asaduzzaman, & Chun, 2013). PGSS is a successful encapsulation technique based on the solubility of SC-CO_2 in molten biopolymers, oils, and fats (Martín, Pham, Kilzer, Kareth, & Weidner, 2010). The PGSS process is advantageous as it is operated under mild conditions, produces microparticles of homogenous and narrow size distribution, and is vital for the production of solvent free final products in the food and pharmaceutical industries (Levai et al., 2017). Moreover, particles of different morphologies like spherical and completely solid, distorted, agglomerated, spherical and hollow, etc., can be produced using this process. Polyethylene glycol (PEG) of high molecular form is a very common and popular material for the production of microparticles of water insoluble, non-polar bioactive compounds such as fish oil and astaxanthin using the PGSS process because of its hydrophilic nature and solubility in SC-CO_2 (Yun et al., 2013). Due to toxicological safety, the use of PEG in food and pharmaceutical industry is approved up to a daily maximum dose of 10 mg/kg body weight (Barrett, Dehghani, & Foster, 2008). So, in this research work, it was selected a PEG with molecular weight of 6000 g/mol (PEG-6000) due to its good miscibility with SC-CO_2, its water solubility, and its non-toxic property.

The efficiency of microparticles for encapsulating bioactive compounds is related to pre-expansion conditions such as temperature, pressure, ratios of core materials and polymer, and nozzle size. As far as we know, there is no report on ω-3 PUFAs and astaxanthin rich salmon oil encapsulation by PGSS technology using PEG-6000. Therefore, the objectives of this study were optimizing suitable process conditions (temperature, pressure, oil and polymer ratio, nozzle size, etc.) for encapsulation of ω-3 PUFAs and astaxanthin-rich salmon oil by the PGSS process and characterizing the produced particles for a wide range of physico-chemical parameters, i.e., encapsulation yield, PUFAs and astaxanthin stability, microparticle density, microparticle size distribution, scanning electron microscopic (SEM) view, microparticle size analysis, Fourier transform infra-red spectroscopy (FT-IR), thermogravimetric analysis (TGA), and in vitro release.

2. Materials and methods

2.1. Materials and reagents

Carbon dioxide (CO_2) gas (99.99%) was provided by KOSEM, Yangsan, Korea. PEG with an average molecular weight of 6000 g/mol, fatty acid methyl esters (FAMEs), and astaxanthin standard were collected from Sigma-Aldrich Co., St. Louis, Missouri, USA.

2.2. Sample collection and ω-3 PUFAs rich salmon oil preparation

Atlantic salmon frame bone was obtained from Seawell Co., Ltd. (imported from Norway), Haeundae-gu, Busan, Korea. The sample was prepared, and oil was extracted using SC-CO_2 technique (Haq et al., 2017b). Omega-3 PUFAs and astaxanthin-rich salmon oil were produced by urea complexation and the enzyme-catalyzed transesterification process (Haq, Park, Kim, Cho, & Chun, 2017c).

2.3. Microencapsulation of salmon oil by the PGSS process

Microencapsulation of salmon oil by the PGSS process was performed using the two-step optimization process. In the first step, the pre-expansion temperature (45 °C–55 °C) and pressure (15–25 MPa) were optimized at fixed oil and PEG-6000 ratio of 1:5 w/w and diameter nozzle of 300 μm. In the second step, considering the optimized condition of temperature and pressure, the oil and PEG-6000 ratio (1:2.5–1:10, w/w) and diameter nozzles (300–500 μm) were optimized. In both steps, the optimization was based on the maximum encapsulation yield.

The laboratory scale instrument for the PGSS process used in this study is shown in Fig. 1. Salmon oil and PEG-6000 at specified amounts were loaded into the reactor and the cap of the reactor, to which a stirrer was attached, was plugged carefully to prevent gas leakage. CO_2 gas was pumped and delivered to the reactor until the desired pressure was reached. The reaction process was continued for 1 h after which the oil with PEG was released through the nozzle and collected from a separator.

2.4. Encapsulation efficiency (EE)

EE of the salmon oil in microparticle was determined following the procedure of Varona, Kareth, Martina, and Cocero (2010) with slight