LWT - Food Science and Technology 70 (2016) 126–134

Contents lists available at ScienceDirect

LWT - Food Science and Technology

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

Optimization of coffee oil flavor encapsulation using response surface methodology

Adane Tilahun Getachew [a, b], Byung-Soo Chun [a, *]

[a] Department of Food Science and Technology, Pukyong National University, 45 Youngso-ro, Nam-Gu, Busan 48513, Republic of Korea
[b] School of Chemical and Bioengineering, Addis Ababa Institute of Technology, Addis Ababa University, P.O.Box 385, Addis Ababa, Ethiopia

ARTICLE INFO

Article history:
Received 19 August 2015
Received in revised form
2 February 2016
Accepted 8 February 2016
Available online 11 February 2016

Keywords:
Coffee oil
Encapsulation efficiency
Flavors
Optimization
PGSS

ABSTRACT

This study aimed the encapsulation of coffee oil flavor and optimizing the process conditions which affect the encapsulation efficiency (EE). Oil was extracted from roasted *Coffea arabica*, (Yrigachefe, Ethiopia) using supercritical carbon dioxide (Sc–CO_2). Then, the extracted oil was encapsulated by polyethyleneglycol (PEG) using particles from gas-saturated solutions (PGSS) process. Process parameters which affect the EE such as, temperature (T, 40–50 °C), pressure (P, 200–300 bar) and polymer-oil ratios (R, 5:1–10:1 g/g) were optimized via Box Behnken design (BBD). The optimal conditions were (T, 40.0 °C, P, 260.14 bar and R, 6.57:1 g/g) with a maximum EE of 79.78%. The encapsulated oil showed peroxide value of 4.56 meq peroxide/kg oil after 12 weeks of storage. Less than 2% loss of fatty acid composition was observed after encapsulation. Moreover, flavor profiles of particles obtained using optimal condition showed very good preservation of flavors. Therefore, microencapsulation using PGSS process could be employed for production of freely flowing powdered particles that can be used in food processing industries.

1. Introduction

Coffee is one of the main agricultural crops and one of the most traded commodities in the world, behind only to petroleum (Naidu, Sulochanamma, Sampathu, & Srinivas, 2008). Coffee oil is composed of triglycerides (75%) terpene esters (14%), partial acylglycerols (5%), free fatty acids (1%), free sterols (1.5%), sterol esters (1%) and polar lipids (<1%) (Nikolova-Damyanova, Velikova, & Jham, 1998). High triglyceride content makes it vulnerable to lipid oxidation (Oliveira, Cruz, Eberlin, & Cabral, 2005). The instability and susceptibility of edible oils to oxidative degradation result in a loss of nutritional quality and a development of off flavors, hence affecting shelf stability and sensory properties of products (Velasco, Dobarganes, & Márquez-Ruiz, 2003). Microencapsulation of oils in powder particles is a technological process addressed to protect polyunsaturated oils against oxidation, to mask or preserve flavors and aromas. Microencapsulation consists of involving a solid, liquid or gaseous component in a wall material, in order to form a particle that may offer protection against oxygen, heat, humidity and light. In addition, it offers the possibility of controlled diffusion of lipophilic functional food ingredients (Charve & Reineccius, 2009). Spray drying is a process widely used for microencapsulation of oils and flavors. However, most of the flavoring compounds which give foods their characteristic aroma are highly volatile with respect to water and hence, they are easily lost during spray-drying operation (Madene, Jacquot, Scher, & Desobry, 2006). Therefore, other technological solutions that minimize the loss of flavor are needed.

Particle formations using supercritical carbon dioxide (Sc–CO_2), such as rapid expansion of supercritical solutions (RESS), particles from gas-saturated solutions (PGSS), and supercritical antisolvent precipitation (SAS) have received much attention as alternative particle formation methods (Mishima, 2008). It is important for drug delivery systems to obtain composites or encapsulates which comprise an active compound loaded into a matrix of a carrier material, in order to improve product preservation as well as control the dissolution rate of the active compound (Cocero, Martín, Mattea, & Varona, 2009). The PGSS process involves the interaction of several experimental variables that affect the encapsulation efficiency (EE). Therefore, it is very important to select and optimize the most important variables that affect the EE.

Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for developing, improving and optimizing processes in which a response of interest is influenced

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

http://dx.doi.org/10.1016/j.lwt.2016.02.025
0023-6438/

by several variables, and the objective is to optimize this response (Baş & Boyacı, 2007). Analyzing the effects of the independent variables, this experimental methodology generates a mathematical model which describes the chemical processes within the experimental range (Myers, Montgomery, & Anderson-Cook, 2009).

Although a few attempts have been made to microencapsulate coffee oil using spray drying process, information on microencapsulation of coffee oil flavor using PGSS process and optimization of the process conditions using RSM is limited. Therefore, the aims of this work were to encapsulate roasted coffee oil by PGSS process using Polyethylene glycol (PEG) and optimize the process conditions using RSM. Moreover, the fatty acid composition and headspace aroma of coffee oil were also analyzed before and after encapsulation. The microcapsules obtained from optimized condition were also characterized.

2. Materials and methods

2.1. Materials

Green coffee beans, *Coffee arabica*, from Ethiopia, (Yirgacheffe grade 1) were supplied by Global Soft Commodities GSC International Coffee®, Seoul, Republic of Korea. All chemicals used were of either HPLC or analytical grades.

2.2. Sample preparation

Green coffee was medium roasted using home scale roaster (Hearthware Precision Coffee Roaster, Wheeling, IL, USA) at 220 °C for 900 s then the roasted coffee was finely ground by mechanical blender (PN SMKA-4000 mixer, PN Co., LTD, Republic of Korea) and sieved using a 710 μm stainless steel sieve. Then, the samples were immediately used for extraction of oil to protect the possible loss of flavor upon storage.

2.3. Oil extraction

The oil was extracted using laboratory scale Sc–CO_2 extraction system (Fig. 1). The extraction was done at a temperature of 45 °C and pressure of 250 bar. CO_2 flow rate was constant at 27 g/min during the whole extraction period of 2 h.

2.4. Microencapsulation process

The encapsulation process was carried out using laboratory scale PGSS apparatus designed and assembled by Department of Food Science and Technology. The schematic diagram of PGSS process used in this study is shown in Fig. 2. PGSS process began by placing PEG-8000 and coffee oil at different ratio into the reactor and then CO_2 was pumped from cylinder until the desired pressure was reached. Then mixture was stirred at 300 rpm and the nozzle size was 300 μm. The time of reaction for all runs was 1 h. During the reaction time the mixture melted and saturated with Sc–CO_2 .After 1 h, the mixture was depressurized by releasing through the nozzle to the precipitation chamber which was kept at ambient pressure. The solid particles were produced due to the intense cooling effect caused by the release of CO_2 (Cocero et al., 2009). The particle were collected from precipitation chamber and kept in air tight 100 mL plastic bottle until needed for further study.

2.5. Experimental design and statistical analysis

The RSM was applied to evaluate the effects of PGSS parameters and optimize conditions for various responses. Box–Behnken experimental design (BBD) with three numeric factors on three levels was used. This design consisted of fifteen randomized runs with three replicates at the central point to minimize the error. Independent variables used in experimental design were temperature (T, 40–50 °C), pressure (P, 200–300 bar) and polymer-oil ratio (R, 1:5–1:10 g/g). Each variable was coded variables in dimensionless numbers from −1 to 1 (Baş & Boyacı, 2007). Variables were coded according to Equation (2) (Gan & Latiff, 2011):

$$X = \left(\frac{X_i - X_0}{\Delta X}\right) \tag{1}$$

where X is the coded value, X_i is the corresponding actual value, X_0 is the actual value in the center of the domain, and Δ X is the increment of X_i corresponding to a variation of 1 unit of X. The natural and coded values of independent variables used in BBD are presented in Table 1. The response variable was fitted to the following second-order polynomial model (Equation (3)) which is generally able to describe relationship between the responses and the independent variables (Bezerra, Santelli, Oliveira, Villar, & Escaleira, 2008):

$$Y = \beta_0 + \sum_{i=0}^{3} \beta_i X_i + \sum_{i=0}^{3} \beta_{ii} X_i + \sum \sum_{i<j=1}^{3} \beta_{ii} X_i X_j, \tag{2}$$

where Y represents the response variable, Xi and Xj are the independent variables affecting the response, and β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms. Optimal encapsulation conditions were determined considering EE as response. Treatment of multiple responses and selection of optimal conditions were based on desirability function (Derringer, 1980). The experimental design and multiple linear regression analysis were performed using Design-Expert v.7 (Stat-Ease, Minneapolis, Minnesota, USA).

2.6. Encapsulation efficiency

The amount of unencapsulated oil (oil present at the surface of the powders) was measured using a method described by Tan, Chan, and Heng (2005) with some modifications. 15 mL of hexane was added to 2 g of powder in a 30 mL glass vial with a screw cap and shaken with a vortex mixer for 2 min at room temperature (25 ± 0.5 °C) to extract free oil. The solvent mixture was then decanted and filtered through a Whatman No. 1 filter paper. The collected powder on the filter paper was rinsed three times with 20 mL of hexane at each time by passing it through the powder. The residual powder was then dried to vaporize all residual solvent at 60 °C to a constant weight. Total oil was determined gravimetrically by ether extraction after solubilizing the wall material using deionized water (Bradley et al., 1992). The free oil content was then calculated as percentage by the weight difference in the powder before and after extraction and washing with hexane. Microencapsulation efficiency (EE) was calculated using the following equation:

$$EE = \left(\frac{T_0 - S_0}{T_0}\right), \tag{3}$$

where T_O, is the total oil content and S_O is the surface oil content.

2.7. Fatty acid analysis

An 6890 Agilent Technologies (Wilmington, DE, USA) gas chromatograph with a fused silica capillary column (length, 100 m;