Food Chemistry 139 (2013) 448–457

Contents lists available at SciVerse ScienceDirect

Food Chemistry

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

# Microcapsule production employing chickpea or lentil protein isolates and maltodextrin: Physicochemical properties and oxidative protection of encapsulated flaxseed oil

Asli Can Karaca, Michael Nickerson, Nicholas H. Low *

*Department of Food and Bioproduct Sciences, University of Saskatchewan, 51 Campus Drive, Saskatoon, SK, Canada S7N 5A8*

ARTICLE INFO

*Article history:*
Received 9 August 2012
Received in revised form 7 December 2012
Accepted 15 January 2013
Available online 29 January 2013

*Keywords:*
Flaxseed oil
Microencapsulation
Legume protein
Freeze-drying
Oxidative stability

ABSTRACT

Flaxseed oil was microencapsulated, employing a wall material matrix of either chickpea (CPI) or lentil protein isolate (LPI) and maltodextrin, followed by freeze-drying. Effects of oil concentration (5.3–21.0%), protein source (CPI vs. LPI) and maltodextrin type (DE 9 and 18) and concentration (25.0–40.7%), on both the physicochemical characteristics and microstructure of the microcapsules, were investigated. It was found that an increase in emulsion oil concentration resulted in a concomitant increase in oil droplet diameter and microcapsule surface oil content, and a decrease in oil encapsulation efficiency. Optimum flaxseed oil encapsulation efficiency (~83.5%), minimum surface oil content (~2.8%) and acceptable mean droplet diameter (3.0 μm) were afforded with 35.5% maltodextrin-DE 9 and 10.5% oil. Microcapsules, formed by employing these experimental conditions, showed a protective effect against oxidation versus free oil over a storage period of 25 d at room temperature.



## 1. Introduction

Flaxseed oil is rich in essential fatty acids (e.g., α-linolenic acid) which are purported to induce a variety of health benefits upon consumption. These health benefits include, reducing the risk of coronary heart diseases (Li, Attar-Bashi, & Sinclair, 2003) and the prevention of breast and prostate cancers (Bougnoux & Chajès, 2003). Despite these purported health-promoting properties, flaxseed oil remains underutilised by the food industry due to its susceptibility to oxidation because of its high polyunsaturated fatty acid (PUFA) content, and due to its lack of miscibility in aqueous food systems (Łukaszewicz, Szopa, & Krasowska, 2004; Bozan & Temelli, 2008). However, through the use of encapsulation technologies these limitations can be circumvented so as to offer PUFA oxidative protection to the harsh environmental conditions experienced during food processing and storage, targeted release during digestion and improved flaxseed oil miscibility in foods.

Microencapsulation is defined as a process whereby an active ingredient becomes enclosed or packaged within micron-sized carrier matrices, which in turn segregate and protect the inner core from the surrounding environment (Gibbs, Kermasha, Alli, & Mulligan, 1999). Depending on the active ingredient and food matrix several physical and chemical methods for capsule production are available (Gouin, 2004; Madene, Jacquot, Scher, & Desobry, 2006). Although gelatin is one of the most widely used encapsulating materials (Dong, Touré, Jia, Zhang, & Xu, 2007; Prata, Zanin, Ré, & Grosso, 2008; Liu, Low, & Nickerson, 2010), it suffers from a number of perceived safety concerns (e.g., prion disease), and religious and dietary restrictions. Therefore, the development of plant protein-based encapsulation systems, as an alternative to animal proteins, is of considerable interest and importance. Legume proteins can serve as a potential source for this purpose because of their high nutritional value, low cost and purported beneficial health benefits, including, but not limited to, reducing the risk of cardiovascular disease, as an aid in glycemic control in diabetic individuals, and in the prevention of digestive tract diseases (Boye et al., 2010; Duranti, 2006). Literature reports of the use of legume proteins as wall materials for lipid encapsulation are few (Ducel, Richard, Saulnier, Popineau, & Boury, 2004; Ducel, Richard, Popineau, & Boury, 2005; Pereira et al., 2009). To the best of our knowledge, the microencapsulation of flaxseed oil in legume protein-based matrices has not been previously reported, however, it has been entrapped within other non-legume and non-gelatin protein matrices with some success.

Grattard, Salaün, Champion, Roudaut, and Le Meste (2002) encapsulated flaxseed oil into a matrix composed of maltodextrin, lecithin and xanthan gum, via freeze-drying. They indicated that the resulting microcapsules efficiently protected flaxseed oil from oxidation. Quispe-Condori, Saldaña, and Temelli (2011) microencapsulated flaxseed oil by spray-drying and freeze-drying methods, using zein as the coating material, and investigated the effects of

* Corresponding author. Tel.: +1 306 966 5037; fax: +1 306 966 8898.
*E-mail address:* nicholas.low@usask.ca (N.H. Low).

0308-8146/$ - see front matter Crown Copyright © 2013 Published by Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.foodchem.2013.01.040

zein and flaxseed oil concentration on microcapsule efficiency. They reported significantly higher encapsulation efficiency for spray-drying (93.3%) than for freeze-drying (59.6%). The objectives of this study were to study flaxseed oil microencapsulation potential of chickpea and lentil protein isolates and maltodextrin, as wall materials, and to investigate the physicochemical properties, surface microstructure, and flaxseed oil oxidative protection of the produced microcapsules.

## 2. Materials and methods

### 2.1. Materials

Chickpea (CDC Frontier, Kabuli) and lentil (CDC Grandora) seeds were provided by the Crop Development Centre at the University of Saskatchewan (Saskatoon, SK, Canada). Maltodextrin samples (DE 9, Dry MD™ 01918 and DE 18, Dry MD™ 01909-Z) were donated by Cargill Inc. (Cargill Texturizing Solutions, Cedar Rapids, IA, USA). Flaxseed oil was kindly donated by Bioriginal Food & Science Corp. (Saskatoon, SK, Canada). All chemicals used were of reagent grade and purchased from Sigma–Aldrich (Oakville, ON, Canada). The water used in this research was produced from a Millipore Milli-Q™ water system (Millipore Corp., Milford, MA, USA).

### 2.2. Proximate analysis

#### 2.2.1. General

Proximate composition analyses for protein isolates and maltodextrin-DE samples were conducted according to AOAC Official Methods 925.10 (moisture), 923.03 (ash), 920.85 (lipid), and 920.87 (crude protein, by using $\%N \times 6.25$) (AOAC, 2003). Carbohydrate content was determined on the basis of percent difference from 100%.

#### 2.2.2. Proximate analysis of maltodextrin samples

The chemical composition of maltodextrin-DE 9 was determined to be: 4.6% moisture, 0.0% protein, 0.0% lipid, 95.0% carbohydrate and 0.4% ash. For maltodextrin-DE 18 the results were: 4.7% moisture, 0.0% protein, 0.0% lipid, 95.0% carbohydrate and 0.3% ash.

### 2.3. Protein isolate preparation

Whole chickpea and lentil seeds were ground into a fine flour, using an IKA A11 basic analytical mill (IKA Works Inc., Wilmington, NC, USA) for 1 min, and then defatted using hexane (1:3 [w/v] flour:hexane ratio) for 40 min. The mixture was then filtered, employing Whatman Gr. 1 paper (110 mm; Whatman International Ltd., Maidstone, United Kingdom), and air-dried in a fume hood. This defatting procedure was repeated twice for each flour.

Chickpea protein isolate (CPI) was prepared according to the method of Papalamprou, Doxastakis, and Kiosseoglou (2010). In brief, defatted flour (100 g) was mixed with water at a 1:10 ratio (w/v), adjusted to pH 9.0 using 1.0 M NaOH and stirred at 500 rpm for 45 min at room temperature (21–23 °C). The suspension was then centrifuged at 4500×*g* for 20 min at 4 °C, using a Sorvall RC-6 Plus centrifuge (Thermo Scientific, Asheville, NC, USA) to collect the supernatant. The resulting pellet was re-suspended in water at a ratio of 1:5 (w/v), adjusted to pH 9.0, stirred for an additional 45 min, followed by centrifugation (4500×*g*, 20 min, 4 °C). Supernatants were pooled and adjusted to pH 4.6, using 0.1 M HCl to precipitate the protein. The protein was recovered by centrifugation as above, collected and stored at −30 °C prior to freeze-drying, which was performed using a Labconco FreeZone 6 freeze drier (Labconco Corp., Kansas City, MO, USA), to yield a free flowing powder. Proximate analysis of CPI showed a chemical composition of, 85.4% protein, 6.52% moisture, 3.05% ash, 4.11% carbohydrate and 0.92% lipid.

Lentil protein isolate (LPI) was produced by employing the combined methods of Bamdad, Goli, and Kadivar (2006) and Lee, Htoon, and Paterson (2007). Defatted flour (100 g) was mixed with water at a 1:10 ratio (w/v), adjusted to pH 9.5 with 1.0 M NaOH, and stirred at 500 rpm for 1 h at room temperature. The mixture was kept static at 4 °C overnight to allow for non-protein sedimentation. After centrifugation at 1600×*g* for 30 min at 4 °C, the supernatant was collected, and pH was adjusted to 4.5 with 0.1 M HCl. The precipitated protein was collected by centrifugation (1600×*g*, 30 min, 4 °C) and stored at −30 °C prior to freeze-drying. Proximate analysis of LPI showed a chemical composition of, 81.9% protein, 5.04% moisture, 3.63% ash, 9.00% carbohydrate and 0.43% lipid.

### 2.4. Emulsion preparation

Protein solutions were prepared by dissolving the isolates (corrected on a weight basis for protein content) in water, followed by adjustment to pH 3.0 or 7.0 with either 0.1 M NaOH or 0.1 M HCl. The resulting mixtures were stirred at 500 rpm overnight at 4 °C to ensure complete dissolution. Maltodextrin solutions were prepared by dissolving either DE 9 or 18 in water, followed by stirring at 300 rpm overnight at 4 °C. Prior to sample homogenisation, the pH of the protein solutions was re-adjusted to 3.0 or 7.0, as described above. Twenty-eight oil-in-water emulsions were prepared (Table 1) by homogenising (Polytron PT2100, Kinematica AG, Lucerne, Switzerland) varying amounts of protein isolate, maltodextrin solutions and flaxseed oil in 15 ml plastic centrifuge tubes, employing a 12 mm PT-DA 2112/2EC generating probe at 13,000 rpm for 3 min.

### 2.5. Droplet size measurements

#### 2.5.1. General

Droplet size distributions of initial and reconstituted emulsions were measured using a Mastersizer 2000 laser light scattering instrument (Malvern Instruments Ltd., Worcestershire, United Kingdom) equipped with a Hydro 2000S sample handling unit (containing water). Emulsion samples were taken from the bottom of the tube, immediately after homogenisation, for analysis. This sample was stirred continuously within the sample cell to ensure homogeneity at room temperature. Obscuration in all the measurements was kept at ~14% by water addition. Droplet size distributions were calculated by the instrument according to the Mie Theory which uses the refractive index difference between the droplets and the dispersing medium to predict the intensity of the scattered light. The ratio of the refractive index of flaxseed oil (1.479) to that of the dispersion medium (1.330) was 1.112. Droplet size measurements were reported as volume/mass moment mean ($d_{4,3}$) which is expressed as:

**Table 1**
Formulations of CPI- and LPI-stabilised emulsions prior to freeze-drying.

| Protein (%) | Maltodextrin (%) | Oil (%) | Water (%) |
|---|---|---|---|
| 4.0 | 40.7 | 5.3 | 50.0 |
| 4.0 | 38.1 | 7.9 | 50.0 |
| 4.0 | 35.5 | 10.5 | 50.0 |
| 4.0 | 32.8 | 13.2 | 50.0 |
| 4.0 | 30.2 | 15.8 | 50.0 |
| 4.0 | 27.6 | 18.4 | 50.0 |
| 4.0 | 25.0 | 21.0 | 50.0 |