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Olive leaves extract encapsulated by spray-drying in vacuum fried starch–gluten doughs

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ABSTRACT

Olive leaves extract (OLE) was microencapsulated with inulin (OLE-IN) by spray-drying using a central composite design. Oleuropein encapsulation efficiency and recovery values were over 87% in the OLE-IN microparticles obtained under optimal conditions. OLE or OLE microparticles were added into starch–gluten fried matrices with the aim of studying the effect of microencapsulation and the frying method on the polyphenols content and antioxidant activity, fat content and crispness. Vacuum starch–gluten fried samples absorbed slightly, but significantly, less oil than the atmospheric fried ones (except for the dough with 350 mg GAE/kg), but crispness was higher in the atmospheric fried products. Although vacuum fried matrices showed significantly higher content of polyphenols than atmospheric fried matrices, both types of matrices showed similar antioxidant activity, suggesting the formation of antioxidant metabolites derived from the Maillard reaction during the atmospheric frying. The results highlighted the importance of the microencapsulation of OLE to preserve the beneficial effects of polyphenols in processed food.

1. Introduction

Epidemiological evidence has demonstrated that diets rich in fruits and vegetables promote healthy effects, preventing oxidative stress-related diseases (Fraga et al., 2010), which has been associated to the content of bioactive compounds such as polyphenols. In the fast modern lifestyle, more processed foods are consumed instead of fresh fruits and vegetables, leading to the development of healthy ready-for-eat food products. Polyphenols have been extracted from several sources such as fruits, vegetables, seeds, flowers, leaves, and recently from agroindustrial wastes (Moure et al., 2001). A large amount of wastes are generated from olive oil industry, being the olive leaves (*Olea europaea* L.) one of the main by-products (Nunes et al., 2016). A wide profile of polyphenols has been described in olive leaves, where the oleuropein (OE) is the major compound. Moreover, beneficial effects of OE on human health, such as anti-atherogenic, anti-carcinogenic and anti-inflammatory have been reported (Bouaziz and Sayadi, 2005); therefore OE could play an important role as a food supplement.

The inclusion of polyphenol extracts from several natural sources as healthy/functional ingredients in food matrices has gained considerable attention in recent times (Robert et al., 2017). Regarding olive leave extracts (OLE), they have been used in different technological and functional applications such as biodegradable films for food packaging (Moudache et al., 2017), and food fortification (Nunes et al., 2016). Some examples of application of OLE in food matrices are enrichment of edible oils (Sahin et al., 2017), frying oils (Chiou et al., 2007, 2009), table olives, meat and meat products and fruit and fruit derivatives (Nunes et al., 2016). Furthermore, olive leave extracts have been included in biscuit formulations as a strategy for mitigation of dietary advanced glycation endproducts (Navarro and Morales, 2017).

Snack categories with the greatest growth are those that offer a wide range of product alternatives and declare healthy effects. Major

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salty snack manufacturing technologies are baking, extrusion, drying and frying; using doughs based on gluten and starch as starting material. Particularly frying, involves exposing the food to oxygen and high temperatures (usually, between 160 °C to 180 °C), processing conditions that may induce degradation of polyphenols (Dueik and Bouchon, 2011a, 2016). The stability of polyphenols during frying can be improved by modifying the process conditions. Several researchers have shown that vacuum frying is a technology able to retain polyphenols adequately (Dueik and Bouchon, 2011b; Da Silva and Moreira, 2008; Fang et al., 2011). Vacuum frying is an emerging frying technology carried out at pressure well below atmospheric conditions, which causes a great reduction of water boiling point, allowing frying at low temperatures (Garayo and Moreira, 2002; Dueik and Bouchon, 2011a).

Another useful technology to stabilize phenolic compounds is microencapsulation, wherein the active compounds are entrapped in a polymeric matrix to protect them and control their release at specific conditions (time and/or place) (Fang and Bhandari, 2010). Different encapsulation methods for OLE have been described, such as freeze-drying (Taghvaei et al., 2014; Mourtzinos et al., 2007; Ganje et al., 2016), W/O nanoemulsions and W/O/W double emulsion (Mohammadi et al., 2016a), electrostatic extrusion (Belščak-Cvitanovicí et al., 2011), spray-drying (Kosaraju et al., 2006, 2008). However, studies focused on the incorporation of encapsulated OLE in functional and/or healthy foods are scarce (Mohammadi et al., 2016b; Ganje et al., 2016), and none of these studies used the technology of spray-drying to encapsulate the extract. Spray-drying is a useful technique for the encapsulation of heat-sensitive materials because of its short drying times (5–30 s) (Desai and Park, 2005). Furthermore, this is simple, low-cost, reproducible and easy to scale-up, and the microparticles obtained by spray-drying have low water activity, simplifying storage, handling and transport (Gharsallaoui et al., 2007).

The objective of this work was to study the effect of both microencapsulation and frying method on the polyphenol content and quality parameters of starch–gluten fried doughs added with OLE or spray-dried OLE microparticles.

2. Materials and methods

2.1. Materials

Olive leaves (*O. europaea* L.) cv. Arbequina were collected at Melipilla (Chile, 2013). Inulin HP (DP > 23) Raftilina® was purchased from Alfa Chile S.A. (Chile). Matrices were formulated using wheat gluten (Asitec S.A., Chile) and native wheat starch (Blumos S.A., Chile). High oleic sunflower oil (HOSO) was donated by Camilo Ferrón Chile S.A. (Chile). Oleuropein (OE) and gallic acid standards were purchased from Sigma–Aldrich (Chile).

2.2. Preparation of the olive leaves extract

Polyphenols were extracted from olive leaves, previously blanched at 95 °C for 4.5 min, dried at 45 °C for 18 h in a forced-air oven (WTE, Germany). The dried olive leaves (400 g) were ground in a windmill (Fuchs-Müllen, Masch. Kom. N° 18791, Kriens, Switzerland) and macerated in ethanol:water (50:50 v/v, 1.6 L) for 24 h. The mixture was vacuum filtered (Whatman n°1) and the solid residue was reextracted twice and filtered. The extracts were combined and the volume was reduced in a rotatory evaporator (Buchi R-3, Switzerland) at 40 °C. The aqueous olive leaves extract (OLE) was filled up 1 L with distilled water and frozen at −20 °C in dark bottles.

2.3. Characterization of the olive leaves extract

Moisture and soluble solids (°Brix) of OLE were determined according to AOAC methods (1996).

2.3.1. Total polyphenol content

Total polyphenol content of OLE was determined by Folin Ciocalteau (Singleton and Rossi, 1965). The determination was carried out using a spectrophotometer (Unicam UV/vis ATI UNICAM, Cambridge, UK) at 765 nm, and the results were expressed as gallic acid equivalent (GAE) according to a calibration curve obtained using gallic acid solutions ranging from 0.1 to 1 mg/mL ($R^2 = 0.99$).

2.3.2. OLE analysis by HPLC

The OE content in the OLE was determined by HPLC at 280 nm according to Al-Rimawi (2014), using a Merck Hitachi L-6200 pump, a Waters 996 photodiode-array detector and a C18 column (5 μm × 4.6 mm i.d. × 250 mm, Symetry®, Waters, Ireland). An isocratic mobile phase of water:acetonitrile (80:20 v/v) containing 0.1% glacial acetic acid was used at a flow rate of 1 mL/min. OE was quantified using an OE standard calibration curve (20–200 μg/mL; $R^2 = 0.99$). Before injecting into the HPLC, the OLE was filtered (0.22 μm) and an aliquot (0.5 mL) was transferred to a volumetric flask filled up 25 mL with the mobile phase.

2.3.3. Antioxidant capacity

The antioxidant capacity of OLE was determined by 2.2-diphenyl-1-picryhydrazyl (DPPH) (Brand-Williams et al., 1995), ferric reducing ability of plasma (FRAP) (Benzie and Strain, 1996) and oxygen radical absorbance capacity (ORAC) (Dávalos et al., 2004) methods. DPPH was expressed as EC_{50}, whereas FRAP and ORAC were expressed as trolox equivalent (TE).

2.4. Preparation of olive leaves extract microparticles

The encapsulation of OLE was performed by spray drying, using inulin (IN) as encapsulating agent, according to a central composite experimental design with 12 runs. The OLE:IN ratio (1:0.34–1:2.15) and inlet air temperature (135–184 °C) were evaluated as independent variables. The dependent variables were oleuropein encapsulation efficiency (EE), recovery (R) and yield (Y). The data were fitted to a second-order regression model, according to Eq. (1). All of the experiments were conducted randomly to avoid systematic bials.

$$Y = \beta o + \sum_{i=1}^{2} \beta_i X_i + \sum_{i=1}^{2} \beta_{ii} X_i^2 + \sum_{i=1}^{1} \sum_{j=i+1}^{2} \beta_{ij} X_i X_j \quad (1)$$

where Y was the response; subscript i and j ranged from 1 to the number of variables (n = 2); β_o was the intercept term; β_i values were linear coefficients; β_{ii} values were the quadratic coefficients; β_{ij} values were the cross-product coefficient; and X_i and X_j were the levels of independent variables.

The analysis of variance (ANOVA), test of lack of fit, and determination of regression coefficients were performed with the software Statgraphics (5.0 program, Manugistics Inc., Rockville, MA). Response surface methodology (RSM) was used to determine the optimal conditions for each independent variable. Optimisation was performed using the desirability function (DF), where all the independent variables were maximized.