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Encapsulation of antioxidant phenolic compounds extracted from spent coffee grounds by freeze-drying and spray-drying using different coating materials

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ABSTRACT

Freeze-drying and spray-drying techniques were evaluated for encapsulation of phenolic compounds (PC) extracted from spent coffee grounds. Additionally, the use of maltodextrin, gum arabic and a mixture of these components (ratio 1:1) as wall material to retain the PC and preserve their antioxidant activity was also assessed. The contents of PC and flavonoids (FLA), as well as the antioxidant activity of the encapsulated samples were determined in order to verify the efficiency of each studied condition. Additional analyses for characterization of the samples were also performed. Both the technique and the coating material greatly influenced the encapsulation of antioxidant PC. The best results were achieved when PC were encapsulated by freeze-drying using maltodextrin as wall material. Under these conditions, the amount of PC and FLA retained in the encapsulated sample corresponded to 62% and 73%, respectively, and 73–86% of the antioxidant activity present in the original extract was preserved.
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1. Introduction

Phenolic compounds are secondary metabolites synthesized by many plants during their normal development or as a response to environmental stress conditions (Beckman, 2000). These compounds present important functional properties being, therefore, of great interest for chemical, pharmaceutical and food industries. In green coffee, phenolic compounds have been mainly identified as chlorogenic acid and related to substances including caffeoylquinic acid, dicaffeoylquinic acid, feruloylquinic acid, and *p*-coumaroylquinic acid, which are partially transformed during the coffee roasting process (Farah & Donangelo, 2006; Mussatto, Machado, Martins, & Teixeira, 2011). Numerous benefits for the health have been reported as a consequence of the ingestion of phenolic compounds present in coffee (Mussatto, 2015), particularly for chlorogenic acid, including antioxidant activity and anti-obesity (Cho et al., 2010), anti-inflammatory (Shin et al., 2015), anti-diabetic (Karthikesan, Pari, & Menon, 2010) and anti-cancerous effects (Kasai, Fukada, Yamaizumi, Sugie, & Mori, 2000).

However, phenolic compounds are very vulnerable to an oxidizing environment, for example, to light, oxygen, moisture, among others, due to the existence of unsaturated bonds in the molecular structures. Thus, they must be encapsulated to enhance their storage stability, making safer as food ingredients and providing benefits to the consumers. Apart from stabilizing these bioactive compounds, the encapsulation process also helps to mask unpleasant flavours in food provided by these functional compounds, including bitter taste and astringency of polyphenols. A large variety of materials can be used for encapsulation in food applications, being polysaccharides such as maltodextrin, gum arabic, hydrophobically modified starches and chitosan, as well as mixtures of them, the most commonly used coating materials (Gouin, 2004; Nedovic, Kalusevic, Manojlovic, Levic, & Bugarski, 2011; Ray, Raychaudhuri, & Chakraborty, 2016).

Encapsulation techniques are often based on drying processes due to the liquid nature of the extracts that contain the bioactive compounds. Spray-drying, spray-bed-drying, fluid-bed coating and freeze-drying are some examples of encapsulation techniques. Among these technologies, spray-drying is one of the most widely used for food industry due to its low-cost and flexibility (Fang & Bhandari, 2010), together with freeze-drying, which is very suitable for drying of heat sensitive compounds since it conserves almost intact the initial functional properties of those components (Ceballos, Giraldo, & Orrego, 2012). However, the drying technique and the material used as coating usually affect the retention capacity of compounds within the matrix. Therefore, it is of great

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importance to properly select both, the coating material and the encapsulation technique in order to maximize the incorporation and retention of the functional compounds within the encapsulation matrix. Maltodextrin, for example, is a relatively low-cost polysaccharide with neutral taste and aroma and that acts as an effective protection to flavours (Fernandes, Borges, & Botrel, 2014). Gum arabic is also a polysaccharide commonly used in encapsulation processes due to its good emulsifying and film-forming capacities (Silva et al., 2013). Several researchers have studied the use of these two coatings -unmixed and mixed- to encapsulate bioactive compounds such as essential oils (Fernandes et al., 2014), anthocyanins (Flores, Singh, Kerr, Pegg, & Kong, 2014; Mahdavee Khazaei, Jafari, Ghorbani, & Hemmati Kakhki, 2014), cherry pomace phenolic extracts (Cilek, Luca, Hasirci, Sahin, & Sumnu, 2012), propolis (Silva et al., 2013), among others, being the retention capacity highly dependent on the type of phenolic compound encapsulated and on the coating composition.

Most of the bioactive compounds that are encapsulated into these matrices have been extracted from natural sources. Spent coffee grounds (SCG), which is the main residue of coffee industry obtained from soluble coffee preparation (Mussatto, Machado, et al., 2011), has attracted an increased interest as source of bioactive compounds, specially due to its high content of phenolic compounds (Ballesteros, Ramirez, Orrego, Teixeira, & Mussatto, 2017; Conde & Mussatto, 2016; Murthy & Naidu, 2012; Mussatto, Ballesteros, Martins, & Teixeira, 2011; Panusa, Zuorro, Lavecchia, Marrosu, & Petrucci, 2013; Zuorro & Lavecchia, 2012). However, the encapsulation of these compounds for the maintenance of their properties has never been reported. Phenolic compounds extracted from SCG present important properties like antioxidant activity, for example, which make possible their application in different areas. Encapsulation of these compounds is an important strategy to preserve their properties for longer periods since the phenolic compounds would be protected from oxidation by the coating material that acts as a barrier to oxygen and water, improving their stability and use as a food additive (Lavelli, Harsha, & Spigno, 2016). In this sense, the present study evaluated the encapsulation of antioxidant phenolic compounds extracted from SCG by using two different encapsulation techniques, namely freeze-drying and spray-drying. The efficiency of maltodextrin, gum arabic and a mixture of these components as wall material to retain the phenolic compounds and preserve their antioxidant activity within the encapsulated matrix was also evaluated.

2. Materials and methods

2.1. Raw material and chemicals

Spent coffee grounds (SCG) were provided by the Portuguese coffee industry Nova Delta-Comércio e Indústria de Cafés S.A. (Campo Maior, Portugal). The material was dried in an oven at 60 °C until 5% moisture content being then stored for further use in the extraction experiments. All the chemicals used were analytical grade. Maltodextrin (dextrose equivalent 20 (DE20)) and gum arabic were purchased from Sigma–Aldrich (Chemie GmbH, Steinheim, Germany). Ultrapure water from a Milli-Q System (Millipore Inc., USA) was used.

2.2. Extraction procedure

The extraction of antioxidant phenolic compounds from SCG was performed by autohydrolysis using the conditions optimized in a previous study (Ballesteros et al., 2017). Briefly, ultrapure water and SCG (15 ml/g) were mixed into 160-ml cylindrical stainless steel reactor (Parr Instruments Company, Illinois, USA), which was duly closed and placed into an oil-bath with open heating circulator and temperature control (Julabo, Labortechnik GmbH, Seelbach, Germany). The reactor was maintained in the bath for 50 min at 200 °C, being subsequently removed and immediately cooled down in an ice-bath for 10 min to stop the reaction. The total content of the reactor was centrifuged (2500*g*, 20 min) and the supernatant (SCG extract) was filtered through 0.22 μm filters and stored at −20 °C until further use. The volume of extract recovered after centrifugation was quantified and used for calculations.

In order to evaluate the structural properties of the extracted phenolic compounds, SCG extract was submitted to a reaction for the phenolic compounds precipitation. In brief, the extract was mixed with ethyl acetate (1:3 v/v) and the mixture was kept at room temperature during 24 h, being then centrifuged (2500*g*, 20 min) and the precipitated dried at 100 °C.

2.3. Encapsulation process

Encapsulation of the SCG extract was carried out using maltodextrin and gum arabic as coating materials. For the assays, 100 ml of extract were mixed with 20 g of coating material and the mixture was homogenized at 6000 rpm in an IKA T-25D Ultra-turrax homogenizer until obtaining a good dispersion. Three matrices were evaluated: i) 100% maltodextrin; ii) 100% gum arabic; and iii) a mixture of maltodextrin and gum arabic at ratio 1:1. A blank consisting of distilled water instead of SCG extract was also prepared for each matrix. All the samples were prepared in triplicate and the total soluble solids (°Brix) were measured using a digital refractometer. Afterward, the samples were subjected to freeze-drying and spray-drying processes. For freeze-drying, the samples were previously frozen and then put into a chamber at −60 °C under pressure of 0.05 bar, being maintained under these conditions for 48 h. A Christ alpha 1–4 LD equipment (SciQuip, UK) was used. Spray-drying was carried out in an equipment mini Buchi model 191 (Büchi Laboratoriums Technik, Switzerland) using a liquid feed volumetric flow rate of 108 ml/h, drying air inlet temperature of 100 °C, nozzle air flow-rate, 600 NL (litters at normal conditions)/h and aspiration 75% (28 m^3/h).

The moisture content of the dry powders was determined in a moisture analyser model MAC 50/1/NH (Radwag, Poland) and they were stored at room temperature and protected from the light until further analyses.

2.4. Analytical methodology

2.4.1. Chemical characterization of SCG extract

High performance liquid chromatography was used to analyse the compounds present in the SCG extract. Chlorogenic acid, hydroxymethylfurfural, and furfural were identified and quantified in the extract using the following conditions (Mussatto, Ballesteros, et al., 2011): UV detector at 276 nm and a Nucleosil 120-5 C18 5 μm (4.6 × 250 mm) column at room temperature. Acetonitrile/water (ratio 1/8) with 10 g/l of glacial acetic acid (pH adjusted to 2.5 with phosphoric acid) was used as mobile phase at 0.9 ml/min. The responses of the detector were integrated using the D-7000 HPLC System Manager software (Hitachi).

2.4.2. Structural characterization

Morphology and crystalline phases of SCG extract and encapsulated phenolic compounds were evaluated by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively (Ballesteros, Teixeira, & Mussatto, 2014a). For the SEM analyses, the samples were covered with a very thin film (35 nm) of Au-Pd (80–20 wt%) and the images were obtained by applying an

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