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Use of Supercritical Assisted Atomization to produce nanoparticles from olive pomace extract

Bahar Aliakbarian [a,*], Marco Paini [a], Renata Adami [b], Patrizia Perego [a], Ernesto Reverchon [b]

[a] Department of Civil, Chemical and Environmental Engineering, University of Genoa, Via Opera Pia 15, 16145 Genova, Italy
[b] Department of Industrial Engineering, University of Salerno, Via Giovanni Paolo II, 132, I-84084 Fisciano, Italy

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ABSTRACT

Olive pomace is one of the most interesting wastes containing bioactive compounds; the extraction of polyphenols can represent an innovative solution for the reduction of the environmental hazard of this solid and the simultaneous recovery of high-added value compounds. In this study, Supercritical Assisted Atomization (SAA) was employed for the encapsulation in maltodextrin of phenolic compounds extracted from olive pomace. The effect of the ratio of maltodextrin content to total solid content of the extract and drying temperature on physical characteristics, total phenolic content and antioxidant properties of the powdered product were studied. The results confirmed the efficiency of the SAA process to encapsulate phenolic compounds from olive pomace extract. Particles with average diameter of 712 nm with high total polyphenol content (105.0 ± 0.1 $mg_{Caffeic\ Acid\ Equivalent}/g_{Dry\ Powder}$) and antiradical power (98.8 ± 3.0 $mg_{DPPH}/mL_{extract}$) were obtained. These particles rich in bioactive compounds can be used as functional component in formulations of new food, cosmetic or pharmaceutical products.

Industrial relevance: Olive pomace is considered to be a low-cost and renewable source of high-added value compounds, such as polyphenols which can be valorized by several methodologies. In this work, we assessed the efficiency of Supercritical Assisted Atomization (SAA) in order to encapsulate phenolic compounds extracted from olive pomace. The particles obtained by SAA have spherical morphology with average diameter of 712 nm. The polyphenol-rich nanoparticles produced using this technique can be potentially used in the formulation of novel food or nutraceutical products.

1. Introduction

Olive pomace, a heterogeneous solid waste deriving from olive oil production, is one of the most widespread agro-industrial by-products in Mediterranean area. The amount of organic compounds with high chemical oxygen demand (COD) present in olive pomace (about 3500 mg/L) (Mavros, Xekoukoulotakis, Mantzavinos, & Diamadopoulos, 2008), forces the related industries to apply adequate treatments for this by-product. Olive pomace, being rich in bioactive molecules (Palmieri et al., 2012), could be considered a low-cost and renewable source of high-added value compounds, such as polyphenols. Recovering valuable bio-products like phenolic compounds from agri-food residues such as olive pomace has been recognized as a big scientific effort in the past decade (Aliakbarian, Casazza, & Perego, 2011) due to their potential application in food, nutraceutical, cosmetic and pharmaceutical fields (Fang & Bhandari, 2010).

The first problem that limits the application of polyphenols is the sensitivity to adverse environmental conditions like high temperature, light exposure, moisture, and presence of oxygen (Fang & Bhandari, 2011), that can lead to degradation of the bioactive compounds during processing and storage. Their low solubility in water must be also taken into account, that can preclude the addition of high amount of these compounds into water food systems and nutraceutical formulations, without proper process technology (Xiao, Shao, Yan, & Zhang, 2011; Zhang et al., 2015). Another limitation in the use of polyphenols is their bitter and astringent taste: in particular, astringency is due to the precipitation of mucopolysaccharides and salivary glycoproteins onto the tongue (Kosaraju, Labbett, Emin, Konczak, & Lundin, 2008). For the above reasons, encapsulation represents a suitable method to protect polyphenols from environmental conditions and enhance their solubilization in water and consequently their bioavailability and the astringency of these substances.

Supercritical fluids have been proposed for a wide range of industrial applications: among these, production of membranes (Baldino, Cardea, & Reverchon, 2016), micron and nanoparticles precipitation (Campardelli, Oleandro, & Reverchon, 2016; Prosapio, Reverchon, & De Marco, 2016; Santo et al., 2015).

Supercritical Assisted Atomization (SAA) has also been successfully used to produce microspheres active compounds + polymer

* Corresponding author.
E-mail address: Bahar.aliakbarian@unige.it (B. Aliakbarian).

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(Labuschagne et al., 2014; Petra et al., 2015; Reverchon & Adami, 2013), and is an efficient technique to coprecipitate thermolabile (Adami, Liparoti, & Reverchon, 2011) and low water soluble compounds (Liparoti, Adami, Caputo, & Reverchon, 2013). This micronization process takes advantage of combined liquid–gas properties of supercritical CO_2 (SC—CO_2). The key of the process is the solubilization of SC—CO_2 in a solution of the target compound in water or an organic solvent, to produce an expanded liquid solution. The resultant solution is further expanded through a nozzle in a drying chamber and gas expansion leads to the formation of fine liquid droplets, which can be easily dried in a warm nitrogen flow (Martin et al., 2013).

Supercritical based micronization techniques have also been used to coprecipitate several active molecules with antioxidant properties, such as phenolic compounds extracted from olive leaves (Chinnarasu et al., 2015), and curcuminoids from ethanolic turmeric extract (Osorio-Tobón, Carvalho, Rostagno, Petenate, & Meireles, 2016). Particles from Gas Saturated Solutions (PGSS) has been successfully used to coprecipitate a lipophilic component such as hydroxytyrosol from an aqueous solution using glycerol monostearate as the lipid carrier (Gonçalves, Poejo, Matias, Nogueira, & Duarte, 2014). In a more recent study, Salgado, Rodríguez-Rojo, Alves-Santos, and Cocero (2015) compared the use of PGSS-drying with Supercritical Anti-Solvent (SAS) to encapsulate resveratrol and tebuconazole using β-Glucans and soy lecithin as encapsulating agents. They concluded that SAS was not a suitable process to dry this material resulting to organic solvent trace in the product. Formulations were successfully dried by PGSS-drying technology.

SAS has been used to encapsulate polyphenols, extracted from green tea (Sosa, Rodriguez-Rojo, Mattea, Cismondi, & Cocero, 2011), and antioxidants extracted from rosemary (Visentin, Rodriguez-Rojo, Navarrete, Maestri, & Cocero, 2012). However, at the best of our knowledge, no supercritical based micronization techniques have been used for encapsulation of polyphenols extracted from olive pomace.

Therefore, the aim of this study was to assess the efficiency of SAA technology to encapsulate phenolic compounds from olive pomace, extracted by a previously established method. Maltodextin (MD) was used as the carrier. Different ratios of MD to total solids of the extract and drying chamber temperature were used as operating parameters. The moisture content, average size of the particles, rehydration properties, total content of polyphenols and antiradical power of the particles were measured.

2. Materials and methods

2.1. Chemicals

Methanol, acetic acid, ethanol, n-hexane, acetonitrile, maltodextrin, Foline Ciocalteau and 2,2- diphenyl-1-picrylhydrazyl (DPPH) reagents, and caffeic acid were purchased from Sigma Aldrich (St. Louis, MO, USA). Standard solutions were prepared with methanol and stored in dark bottles at 4 °C.

2.2. Olive pomace extract

Polyphenols were extracted from Taggiasca olive pomace by a high pressure-high temperature agitated reactor, HPTE, (model 4560, PARR Instrument Company, Moline, USA) using ethanol:water 50:50 as solvent, working at 200 rpm and 180 °C for 90 min. The reactor contained appropriate valves to allow introduction and removal of gases inside the reaction chamber. To avoid the effect of an extractive atmosphere for phenolic oxidation during the experiments, as an additional operative condition, all tests were carried out under nitrogen atmosphere by flushing nitrogen through the reactor for 2 min. The reactor was hermetically closed, hence pressure was directly proportional to the temperature. The maximum pressure reached in the reactor was 2.0 MPa. The ratio of olive pomace:extraction solvent was 1:10 (*w*/*v*). These operative conditions were based on the best operative parameters previously established in our laboratory (Aliakbarian et al., 2011; Paini et al., 2016). Extracts were centrifuged at 6000×*g* for 10 min (ALC PK131, Alberta, Canada), filtered (0.22 μm) and stored at 4 °C prior to experiments. Before encapsulation, the solid content of the olive pomace extract (expressed as mg/mL) was determined oven-drying at 105 °C a known volume of the extract for 24 h.

2.3. SAA apparatus and operative parameters

SAA laboratory apparatus (Fig. 1) consists of two high-pressure pumps (model 305, Gilson, Middleton, USA) which deliver the liquid solution (extract and carrier) and the liquid CO_2 to a mixer, as described elsewhere (Adami, Sesti Osséo, & Reverchon, 2009; Liparoti, Adami, & Reverchon, 2012; Reverchon & Adami, 2013).

The mixer is formed by a high pressure vessel (internal volume 25 cm^3) loaded with stainless steel perforated saddles, which assure a large contact surface between liquid solution and CO_2. The solution obtained in the mixer is then sprayed through an 80 μm diameter injection nozzle to the drying chamber (internal volume of 3 dm^3). A controlled flow of nitrogen (N_2) is taken from a cylinder, heated in an electric heat exchanger (model CBEN 24G6, Watlow, St. Louis, USA) and sent to the drying chamber to facilitate liquid droplets evaporation. Temperatures of mixer (Tmix), of the filter (Tf), and of drying chamber (Tc) can be controlled using band heaters (model STB3EA10, Watlow, St. Louis, USA). A stainless steel filter (0.1 μm) located on the bottom of the drying chamber allows the powder collection and the gaseous stream flow out. In this study temperatures of nitrogen flow (TN_2), Tf and Tmix were maintained constant at 95, 75 and 75 °C, respectively. Flow rate of hot N_2 was 1300 NL/h. SC—CO_2 and solution flow rates were kept at 10 and 4 g/min, respectively for all experiments. To operate below the atmospheric pressure, the plant was equipped with a vacuum system, formed by two vacuum pumps (DVP model ZA100P) operating downstream the drying chamber. The pressure in the drying chamber was 0.12 MPa. The system was completed by a condenser, which separates liquids from the gas stream.

Maltodextrin (MD:16.5–19.5 dextrose equivalents) was added into the ethanol:water extract and stirred at room temperature until homogeneous suspension was achieved. In this study, MD was selected as the carrier based on the suitable chemical and physical properties, including low viscosity at high solid contents, good solubility and notable heat protection capacity (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007; Paini et al., 2015). The quantity of MD, expressed as the percentage of the ratio of MD to total solids of the extract (% wt/wt_{TES}), and the temperature of the drying chamber (Tc), were selected as independent variables using a 3^2 full factorial design. MD content was varied from 10 to 50 (% wt/wt_{TES}) and Tc from 75 to 95 °C. According to experimental conditions, 50 mL of the suspension were used as feed. In this study, on the contrary of our previous research (Paini et al., 2015) in which spray drying was used working at 130 to 160 °C and higher amount of MD (100 to 500 g/L) as carrier, we decided to use the lower amount of the carrier (3.47 to 17.35 g/L of MD). The aim of the work was to evaluate the efficiency of SAA process to encapsulate phenolic compounds working at relatively low temperatures (75–95 °C) that can avoid degradation of the polyphenols while processing and using low amount of the carrier. The quantity of the powder recovered by the operator at each condition was measured, and the powdered products were analyzed in terms of physical characteristics (moisture content, size and morphology), rehydration properties, phenolic content and antioxidant power.

2.4. Powder recovery and characterization

Recovery of powdered products at the tested conditions was determined by weighing the total amount of particles recovered by the