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Short communication

Properties of encapsulated saffron extracts in maltodextrin using the Büchi B-90 nano spray-dryer

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ABSTRACT

The production, characterization and stability of nanoencapsulates of saffron hydrophilic apocarotenoids, i.e. crocins and picrocrocin, in maltodextrin using spray-drying are presented. The effect of mesh size and core:wall ratio (w/w) on the product yield and encapsulation efficiency of these apocarotenoids was examined. Nanoencapsulates were characterized and their stability was examined in the presence or absence of a strong phenolic antioxidant, the caffeic acid, under thermal and *in vitro* gastrointestinal conditions. Spherical particles were obtained. Product yield and encapsulation efficiency (%) of crocins and picrocrocin was found to be satisfactory. Thermal stability and bioaccessibility of these apocarotenoids was enhanced by nanoencapsulation. Further protection was provided by caffeid acid.

1. Introduction

Saffron, the dehydrated red three-branch styles of the plant *Crocus sativus* L. is the most expensive spice in the world (Melnyk, Wang, & Marcone, 2010) and a rich source of rare C20 apocarotenoids with a variety of functional properties (Kyriakoudi, Ordoudi, Roldán-Medina, & Tsimidou, 2015). These apocarotenoids, namely, crocetin (8,8′-diapocarotene-8,8′-dioic acid) sugar esters (CRTSEs) or crocins, picrocrocin [4-(β-D-glucopyranosyloxy)-2,6,6-trimethyl-1-cyclohexane-1-carboxaldehyde] and safranal (2,6,6-trimethyl-1,3-cyclohexadiene-1-carboxaldehyde), account for the ~50% w/w of the dried stigmas (Kyriakoudi, Chrysanthou, Mantzouridou, & Tsimidou, 2012). They also impart coloring and flavoring properties to the spice (Carmona, Zalacain, & Alonso, 2006; Ordoudi & Tsimidou, 2004). Crocins (the major one being the digentiobiosyl ester of crocetin, known as trans-4-GG) and picrocrocin are very hydrophilic compounds, a property that defines and confines their food and drink applications. These characteristics together with vulnerability to pH changes, thermal treatment or effect of intrinsic compounds or additives (Alonso, Varõn, Gõmez, Navarro, & Salinas, 1990; Maggi, et al., 2009; Ordoudi, Kyriakoudi, & Tsimidou, 2015; Orfanou & Tsimidou, 1995; Sánchez, Carmona, Ordoudi, Tsimidou, & Alonso, 2008; Tsimidou & Tsatsaroni, 1993) as well as the high value of the raw material, support the idea of protecting the bioactive compounds through encapsulation for food, drug and cosmetic uses (Mishra, 2016). Despite the large number of published scientific articles regarding the encapsulation of bioactive ingredients, limited are the data for the encapsulation of saffron bioactive apocarotenoids (Chranioti, Nikoloudaki, & Tzia, 2015; Rajabi, Ghorbani, Jafari, Mahoonak, & Rajabzadeh, 2015; Selim, Tsimidou, & Biliaderis, 2000) or the parent molecule crocetin (Kyriakoudi & Tsimidou, 2015; Zhou, Yuan, Zhao, Zhao, and Wang 2013). The methods used so far for this reason are freeze-drying, spray-drying and inclusion complexation and the most efficient wall materials employed were maltodextrin, polyvinylpyrrolidone (PVP40) and deoxycholic acid. To our knowledge limited are the applications on nanoencapsulation of saffron bioactive compounds (Esfanjani, Jafari, & Assadpour, 2017; Esfanjani et al., 2015; Mehrnia, Jafari, Makhmal-Zadeh, & Maghsoudlou, 2016; Mehrnia, Jafari, Makhmal-Zadeh, & Maghsoudlou, 2017). It is evident that more applications are to be expected because food applications at nano scale is an emerging field despite the current legislative restrictions for safety reasons (European Commission, 2018).

The present study aimed at encapsulating aqueous saffron extracts in maltodextrin by nano spray-drying, which results in a larger surface area per unit mass of the encalsupates, and offers advantages compared to the conventional mode, among which enhanced gastrointestinal stability, protection against oxidation, controlled release etc. (Anandharamakrishnan, 2014). Maltodextrin was selected among many other edible materials for its high water solubility, low viscosity, mild flavor and efficient use in the encapsulation of saffron extracts (Chranioti et al., 2015; Rajabi et al., 2015). The nanoencapsulates were characterized as in Kyriakoudi and Tsimidou (2015) and their stability was examined in the presence or absence of a selected strong phenolic antioxidant under thermal and *in vitro* gastrointestinal conditions. With this work we continue our efforts to widen applications of saffron

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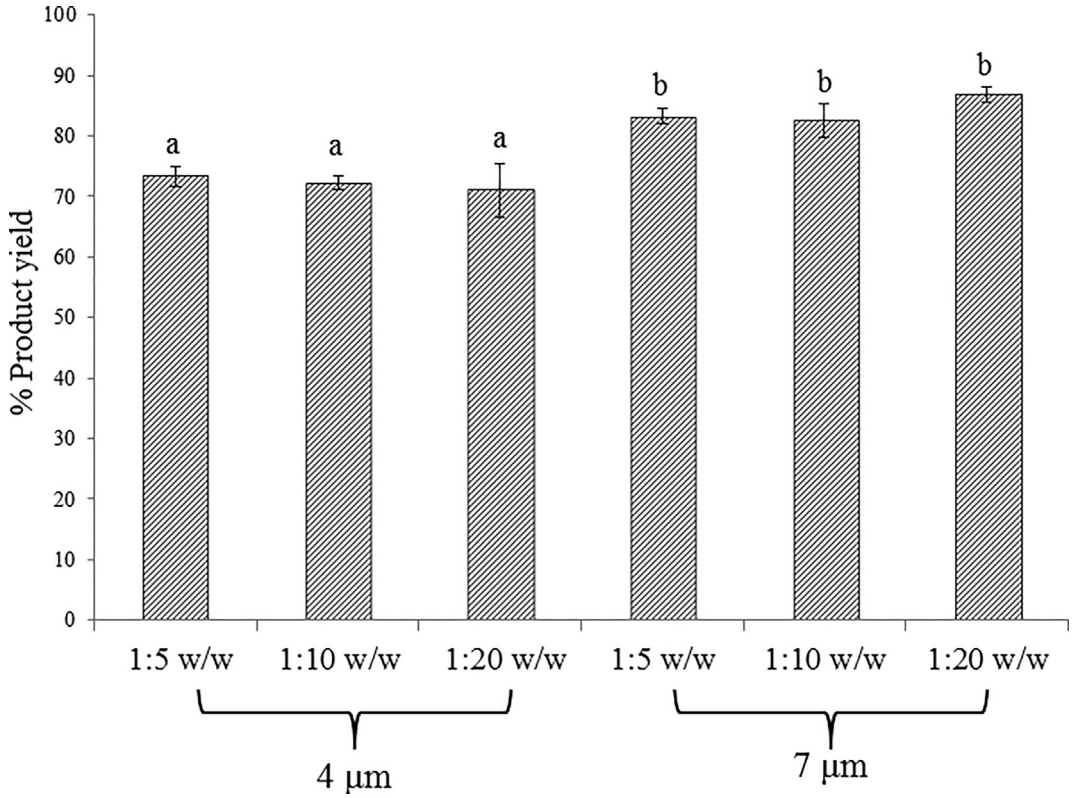

Fig. 1. Product yield (PY) (%) of the nanoencapsulates produced using 4 or 7 μm mesh size. Different lowercase letters indicate significant differences according to Duncan's test at $p < 0.05$; product yield (%) was calculated as following: $\frac{\text{Total amount of solids collected}}{\text{Total amount of solids initially used}} \cdot 100$.

ingredients in food products.

2. Materials and methods

2.1. Materials

Authentic Greek saffron (Saffron Cooperative of Kozani, Greece) was used. *Trans*-4-GG crocetin ester and picrocrocin were laboratory isolated (Kyriakoudi and Tsimidou, 2015; Sánchez, Carmona, Priscila del Campo, & Alonso, 2009). Purity (95% and 99%, respectively) was checked as described by Kyriakoudi, et al. (2012) using the same instrumentation. Maltodextrin with dextrose equivalents of 16.5–19.5 was from Carbosynth (Berkshire, UK). All chemicals were of the highest purity. Ultrahigh purity water was produced using a SG Ultra Clear Basic UV system (SG Wasseraufbereitung und Regenerierstation GmbH, Barsbüttel, Germany).

2.2. Encapsulation process

Crocins and picrocrocin in aqueous saffron extracts (Kyriakoudi and Tsimidou, 2015) and in the obtained nanoencapsulates were determined using RP-HPLC-DAD and were quantified using external calibration curves (*trans*-4-GG crocetin ester, $y = 28296.06x - 109972.40$, $R^2 = 1$; 9–455 ng/10 μL injected volume; $n = 7$ and picrocrocin, $y = 27653.09x - 170026.79$, $R^2 = 1.00$; 10–295 ng/10 μL injected volume; $n = 5$). Aqueous solutions of maltodextrin (10%, w/w) were prepared using a RW 20n homogenizer (IKA-Labortechnik, Staufen, Germany) (~400 rpm, 30 min, 25 °C) and then were kept overnight. Three different core:wall weight ratios (1:5, 1:10, 1:20, w/w) were tested. Feed solutions were also prepared in the presence of caffeic acid (CA). The total solids content was kept constant at 2.5% (w/w). Encapsulation was carried out using the Nano Spray Dryer B-90 (Büchi Labortechnik AG, Flawil, Switzerland) away from direct light and the feed solution was kept in an ice-bath. Nozzles with 4.0 μm and 7.0 μm spray-mesh were used. All feed solutions were filtered through a 0.45 μm RC membrane filter prior to the drying process. The inlet temperature was set at 100 °C based on preliminary trials and kept constant throughout the process. The air flow rate was 100 L/min. The relative spray rate was 80%. Each experiment was carried out in triplicate. The nanoencapsulates were stored in a desiccator at room temperature in the dark until analysis.

2.3. Characterization of the nanoencapsulates

The moisture content was determined by drying in an oven (130 °C, 2 h) and the water activity by an AquaLab LITE apparatus (Decagon, Pullman, WA, USA). Fourier transform-infrared (FT-IR) spectroscopic analysis, thermogravimetric analysis (TGA) and morphological analysis using scanning electron microscopy (SEM), were carried out as described by Kyriakoudi and Tsimidou (2015) using the same instrumentation.

2.4. Stability studies

Saffron (unencapsulated control sample) and nanoencapsulates with or without CA were sealed in glass containers and kept at 60 °C in the dark. The loss of crocins and picrocrocin was followed periodically by withdrawing aliquots and dissolving them in ultrahigh purity water under magnetic stirring (500 rpm, 25 °C, 5 min). Spectrophotometric monitoring (Shimadzu UV 1601, Kyoto, Japan) was carried out in the region 200–600 nm. Quantification of total crocins and picrocrocin was accomplished using calibration curves ($y = 0.52x - 0.031$; $R^2 = 0.99$; 1–50 mg/L; $n = 7$ and $y = 0.459x - 0.0201$; $R^2 = 1$; 1–50 mg/L; $n = 6$, respectively). Duplicate measurements were performed to calculate mean values. The *in vitro* digestion procedure was carried out as previously described by Ordoudi, et al. (2015). The obtained digestates were centrifuged (4.100*g*, 20 min, 4 °C) and the supernatants were filtered (0.45 μm RC membrane filter) and stored at −18 °C until further