Advanced Powder Technology 26 (2015) 462–469

Contents lists available at ScienceDirect

Advanced Powder Technology

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

Original Research Paper

Developing spray-dried powders containing anthocyanins of black raspberry juice encapsulated based on fenugreek gum

Shima Yousefi [a], Zahra Emam-Djomeh [a,*], Mohammad Mousavi [b], Farzad Kobarfard [c], Ireneusz Zbicinski [d]

[a] Transfer Phenomena Lab (TPL), Department of Food Science and Engineering, Faculty of Agricultural Engineering and Technology, University of Tehran, 31587-11167 Karaj, Iran
[b] Department of Food Science and Engineering, Faculty of Agricultural Engineering and Technology, University of Tehran, Karaj, Iran
[c] Department of Medicinal Chemistry, School of Pharmacy, Shahid Beheshti University of Medical Sciences, Tehran, Iran
[d] Faculty of Process and Environmental Engineering, Technical University of Lodz, Lodz, Poland

ARTICLE INFO

Article history:
Received 12 July 2014
Received in revised form 5 November 2014
Accepted 30 November 2014
Available online 11 December 2014

Keywords:
Encapsulation
Black raspberry
Anthocyanin
Fenugreek gum
Response surface methodology (RSM)

ABSTRACT

Spray-dried powder of black raspberry juice was optimized using response surface methodology (RSM). The juice was prepared by dispersions formulated with different coating materials including maltodextrin (MD with DE 6%, 6.59–26.41%), fenugreek gum (FG, 0.33–1.17%) and microcrystalline cellulose (MCC; 0.66–2.34%). Scanning electron microscopy and analyzing laser diffraction particle size were successfully used to relate the microstructure and dominant mechanisms on the stable dispersions to achieve the highest quality powder. Perturbation and 3D surface plots were drawn for each of the responses from the mathematical models. Second-order polynomial models with high R^2 (0.972–0.989) values were constructed for each powder physicochemical properties namely yield, solubility, total anthocyanin content (TAC), and total phenolic content (TPC). The linear effect of MD concentration had a significant ($p < 0.05$) term in all constructed models. The optimal conditions were: MD concentration of 20.0%, FG content of 0.93%, and MCC concentration of 1.74%. These optimum conditions were powder recovery of 82.72%, solubility of 82.2%, TAC of 280.76 mg Cy3G/100 mL and TPC of 2.0491 mg GAE/g dw. A high agreement between experimental and predicted values was found.

1. Introduction

Black raspberry (*Rubus occidentalis*) is a rich source of natural antioxidants and phytochemicals such as flavonoids (*anthocyanins*), tannins (*ellagitannins*), and phenolic acids [1]. This fruit due to the presence of anthocyanins is known for some biological functions such as, reducing eyestrain, DNA repair, anticancer, anti-neurodegenerative, and anti-inflammatory activities [2,3]. The anthocyanins stability is undesirably affected by environmental conditions including temperature, pH, oxygen presence, light, ascorbic acid, sugars and metal ions and co-pigments belonging to the various compounds classes [4].

Raspberry, as one of the most fragile and perishable fruits, is often processed into juice for its longer-term consumption [5], but color of black raspberry juice diminishes owing to enzymatic reactions in the liquid state. The main aim of converting raspberry juice into a powder is its easier use with a reduction in degradation of physicochemical properties of the final product [6]. Spray drying is a ubiquitous industrial operation in which the feed from a fluid state is transformed into a dried form by dispersing the feed into a heating medium [7]. Spray drying as the most common method of encapsulating food ingredients is used for coating or entrapping a functional component within a suitable carrier agent [8]. Quality of the obtained product and the powder efficiency are dependent on the operating conditions such as inlet- and outlet-air temperature, feed-flow rate, atomization speed or pressure, feed concentration and feed to carrier ratio [9].

Optimization of the conditions of spray drying method can highly maintain the natural colorants through capturing the ingredients in a coating material [10]. Typical carrier materials for the most encapsulation process include maltodextrin (MD), arabic gum and modified starch, which are widely used in food industry to improve the effectiveness of drying procedure [11]. MDs consist of D-glucose units linked mainly by $\alpha{-}1 \rightarrow 4$ glycosidic bonds, which were previously applied as common encapsulating agents during different spray drying processes to preserve bioactive compounds in acai pulp [12], mulberry juice [13] and water extract of sumac [8]. Carbohydrates like fenugreek gum (FG) as carrier materials are also suitably used to produce more stable emulsions and

* Corresponding author. Tel./fax: +98 26 32248804.
E-mail address: emamj@ut.ac.ir (Z. Emam-Djomeh).

http://dx.doi.org/10.1016/j.apt.2014.11.019
0921-8831/

to improve the powder yield [14]. FG (*Trigonella foenum-graecum* L.) is obtained from the annual herb of *Fabaceae* family, which is a galactomannan composed of ß-(1 → 4)-D-mannose units as the linear backbone and α-(1 → 6)-D-galactose units as the side chains. The clinical studies have shown anti-carcinogenic, anti-hyperlipidemic and anti-oxidant effects of fenugreek herb [15]. It has been extensively used as thickener, emulsifier, water absorbent and texture modifier in food industry [11]. Moreover, microcrystalline cellulose (MCC) is a common cellulose derivative which is used as thickener or viscosity modifier in the some food processes. It is a desirable coating agent due to its compatibility with a wide range of food compounds such as carbohydrates and hydrocolloids, along with its flavor retention capacity as well as water solubility [16].

The objective of this study was to optimize the formulation of wall materials in order to preserve powder physicochemical properties such as total anthocyanin content (TAC), total phenolic content (TPC), yield and solubility during encapsulation by MD and FG as wall materials and MCC as a supplementary coating material using response surface methodology (RSM). RSM is a collection of mathematical and statistical techniques that have been successfully used for developing, improving, and optimizing processes [17].

2. Materials and methods

2.1. Raw material and juice preparation

Fresh black raspberries (*R. occidentalis*) were selected from Valesh variety and collected from a commercial farm field in Siyahkal city located in Guilan province (Northern Iran) during June–July 2013. It was kept in a hard plastic container to protect the raspberries from direct light and temperature during transportation and used immediately on the same day. Raspberries (approximately 1020 g) were homogenized for 5 min using a crush blender (Master 10-speed Black & Decker, Madison, USA). The prepared black raspberry juice was then filtered through a cotton cloth (approximately 250 μm sieve) and instantaneously refrigerated at −20 °C. A batch of frozen black raspberry juice (1000 mL) was defrosted overnight at 4 °C and mixed with the appropriate aqueous solution of different formulations of the carriers as wall materials including maltodextrin (MD with DE 6%, 6.59–26.41%), fenugreek gum (FG, 0.33–1.17%) and microcrystalline cellulose (MCC, 0.66–2.34%); then, it was all homogenized for 6 min before the experiments. Total solid content of black raspberry juice was 10.0 ± 0.5 °Brix with 4.2 ± 0.2% of total sugars, 1.8 ± 0.1% of total acids, and the solid content of dried powder (96.8–98.5%), which depends on the carrier applied.

2.2. Carrier agents for spray drying

MD (DE value = 6) was prepared from Merck Chemical Co. (Darmstadt, Germany). FG and MCC were purchased from Canafen Gum® (Emerald Seed Products, SK, Canada) and Sigma-Aldrich Chemical Co. (St. Louis, MO, USA). They were mixed with the prepared raspberry extract for 5 min until their complete dissolution. Different formulations of the carriers as wall materials and black raspberry juice (Table 1) were mixed and homogenized at 12,000 rpm for 3 min using a rotor–stator blender (Ultraturrax, IKA, Staufen city, Germany) prior to the spray drying experiment.

2.3. Drying method

The spray drying procedure was conducted in a laboratory scale (Model Büchi B-290, Flawil, Switzerland) located in *Iranian Center of Chemistry and Chemical Engineering Research*. The dispersion of feed delivered through a two-fluid internal mixing nozzle with 0.7 mm in diameter. According to the preliminary studies, the feed flow rate, air flow rate and aspirator rate were 8.65 mL min^{-1} and 4.20 $m^3 h^{-1}$ and 55.87 $m^3 h^{-1}$, respectively. The inlet- and outlet-air temperatures (120 and 78 °C, respectively) were recorded using Testo-465 thermocouple (Testo, USA) with precision of ±0.1 °C [18]. Spray dryer was run for 10 min with distilled water before starting the process. The initial and final weight of powder container was measured using a digital balance. The prepared microcapsules were kept in an appropriate glass tube, and stored in a vacuum desiccator before its utilization.

2.4. Yield determination

The dry matter weight of the obtained powder and black raspberry juice consumed was used to calculate the yield of process based on the following equation [19].

$$Yield = \frac{P \times S_p}{L \times S_F} \times 100 \quad (1)$$

where P is the rate of powder production (g min^{-1}), L is the feed flow rate (g min^{-1}), S_p is the percent of total solids of the powder, and S_F is the percent of total solids of the feed. The powder on the drying chamber wall was manually swept and combined with those collected at the cyclone at the end of drying run. All experiments were done in triplicate.

2.5. Solubility measurement

For the solubility determination, 100 mL of distilled water was transferred into a blender jar. The powder sample (1 g, dry basis) was carefully added to the blender and operated at 15,000 rpm for 5 min. The solution was placed into a tube and centrifuged at 3000*g* for 5 min. An aliquot of 25 mL of the supernatant was then transferred to pre-weighed petri dishes and immediately oven-dried at 105 °C for 5 h. The results can be calculated to give the solubility index in terms of percentage of the soluble material from the weight of the residue [20]. All experiments were done in triplicate.

2.6. Total anthocyanin content

The concentration of the monomeric anthocyanins in the black raspberry juice was measured using the pH differential method [19]. Briefly, powder of black raspberry juice was adjusted with distilled water to give a final solution with total solid content equal to 10.0 °Brix. Then, 1.0 mL of reconstituted black raspberry juice was mixed with 24 mL of the corresponding buffer. The explanation of which proceeds as: the potassium chloride buffer was pH 1.0 (0.025 M) and sodium acetate buffer was pH 4.5 (0.4 M). The resulted sample was vortexed at high speed and then sonicated at 60 kHz for 20 min to allow complete dissolution and centrifuged at 2000*g* for 20 min using a centrifuge (Rotina 420 bench top, Hettich, Tuttlingen, Germany). In the next step, the samples were read against water blank at 510 and 700 nm, respectively. Absorbance (A) was measured as:

$$A = (A_{510} - A_{700})pH_{1.0} - (A_{510} - A_{700})pH_{4.5} \quad (2)$$

TAC of the samples was also calculated using the following equation:

$$TAC = \frac{(A \times MW \times DF \times 100)}{MA} \quad (3)$$

where MW is the molecular weight of cyanidin-3-glucoside (Cy3G, 449.2 g mol^{-1}), DF is the dilution factor (25.0) and MA is the molar extinction coefficient of Cy3G (26,900 L cm^{-1} mol^{-1}). The TAC was