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Effects of the spray-drying temperatures on the physiochemical properties of an encapsulated bitter melon aqueous extract powder

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ABSTRACT

Bitter melon (*Momordica charantia* L.) is a medicinal fruit often used for the treatment of diabetes, due to its content of saponins, phenolics and flavonoids and its antioxidant capacity. The aims were to use response surface methodology (RSM) to optimise the inlet (125.6, 130, 140, 150, 154.1 °C) and outlet (72.9, 75, 80, 85, 87.1 °C) temperatures for the spray-drying encapsulation of a bitter melon aqueous extract using a combination of maltodextrin and gum Arabic as encapsulating agent and to determine the stability of the optimised encapsulated powder under various storage conditions. The RSM models were adequate to describe and predict the responses for the process yield, the retentions of saponins, phenolics, flavonoids and antioxidant activity, the moisture content and the water solubility index with an overall $R^2 \geq 0.91$. The optimal inlet and outlet temperatures were determined to be 140 °C and 80 °C, respectively. The optimised spray-dried powder had high values for process yield (71.4 ± 1.4%), retention of bioactive compounds and antioxidant activity (≥87.9 ± 2.6%), water solubility index (89.9 ± 0.51%) and had a low moisture content (2.2 ± 0.1%), which was below the $M_o = 5.71$ predicted by the BET model. However, in terms of the morphology of the powder particles under scanning electron microscopy and loss of the bioactive compounds and antioxidant activity, the safest range for preserving the powder at 25 °C was determined to range from 22.5% to 33.8%. The encapsulated powder was also slightly more stable at −20 and 10 °C than at 30 °C, over 150 days. Therefore, it can be concluded that spray-drying with the inlet temperature at 140 °C and the outlet temperature at 80 °C resulted in a very stable encapsulated powder of the bitter melon aqueous extract.

1. Introduction

Bitter melon (*Momordica charantia* L.) has long been used as a food and medicine in Asia and Africa. The whole fruit and the seeds of bitter melon contain bioactive compounds such as saponins, phenolics and flavonoids [1–4]. These bioactive compounds are plant secondary metabolites and their phytochemical and pharmacological properties have been widely investigated [5,6].

The bitter melon phytochemicals are considered to be important ingredients in herbal remedies, especially for diabetes; they have long been suggested to be a safe alternative or complementary therapy for lowering blood glucose levels [7–10]. For example, in a study in diabetic rats, a powder prepared from a bitter melon aqueous extract was as effective at lowering blood glucose as glibenclamide, a known synthetic antidiabetic drug [10,11].

Recently, the aqueous conditions for extracting saponins, phenolics and flavonoids from bitter melon have been optimised by Tan et al. [2–4]. However, it is desirable to produce a dry powder from this aqueous extract since powder is easier to handle and it can have a longer shelf life than an aqueous extract because it is less susceptible to oxidation and degradation, especially if the extracted bioactives are encapsulated. Recently, polyphenols from olive pomace [12] and star fruit [13] were successfully encapsulated by spray-drying and the encapsulation of saponins from *Ophiopogon japonicus* has also been reported [14].

Encapsulation by spray-drying is a well-known technique where a homogenised liquid mixture, of a core material of interest and an encapsulating agent, is converted into a dry powder, in which the core material is surrounded by the encapsulating agent, through rapid heating and spraying to quickly evaporate the solvent [15–17]. In encapsulation, the material of interest is coated with at least one

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layer of encapsulating agent(s), thereby preserving the bioactive compounds by protecting them from exposure to air and reducing their deterioration [18]. Spray-drying is the most commonly used technique for this process because the production cost is low and the resulting products are of high quality [15].

The spray-drying conditions, especially the inlet and outlet temperatures, are important factors contributing to the efficiency of the encapsulation process [19–23]. Several studies have reported that the physical and chemical properties of encapsulated powder can be affected by either too high or too low drying temperatures [22,24,25]. Therefore, it is desirable to optimise the inlet and outlet temperatures in order to obtain the highest possible quality of encapsulated powder, in terms of physical and chemical properties. Moreover, following encapsulation, it is also important to determine whether the quality of the powder is affected during storage by environmental factors, such as temperature, oxygen and relative humidity [26,27], in order to determine the best conditions for the storage of the powder.

Therefore, the aims of this study were 1) to use response surface methodology to optimise the inlet and outlet temperatures for the spray-drying encapsulation of the bitter melon aqueous extract in terms of process yield, retentions of saponins, phenolics, flavonoids and antioxidant activity, moisture content, water activity, water solubility index, water absorption index, particle size distribution, bulk density and particle morphology and 2) to determine the stability of the optimised encapsulated powder under various storage conditions.

2. Materials and methods

2.1. Chemicals

Ethanol was obtained from Fronine (Taren Point, NSW, Australia). Folin-Ciocalteau (FC) reagent, sodium carbonate, vanillin, sulphuric acid, sodium nitrite, aluminium chloride, sodium hydroxide (NaOH), sodium acetate trihydrate, acetic acid, 2,4,6-tripyridyl-s-triazine (TPTZ), ferric (III) chloride hexahydrate, trolox, gallic acid, rutin and aecsin, were purchased from Sigma-Aldrich (Castle Hill, NSW, Australia). Lithium chloride (LiCl), potassium acetate (CH_3COOK), magnesium chloride ($MgCl_2$), potassium carbonate (K_2CO_3), magnesium nitrate ($Mg(NO_3)_2$), sodium chloride (NaCl) and potassium chloride (KCl) were obtained from Bacto Laboratories (Mount Pritchard, NSW, Australia). Maltodextrin (DE 18) and gum Arabic (GA) were purchased from the Melbourne Food Depot (Brunswick East, VIC, Australia).

2.2. Plant material

Bitter melons (Moonlight variety) were purchased from the Sydney Markets (Sydney, NSW, Australia). They were immersed in liquid nitrogen before freeze-drying in a FD3 freeze dryer (Rietschle Thomas, Seven Hills, NSW, Australia) at −40 °C and 0.2 mbar for 72 h. The freeze-dried bitter melons were then pulverised using a Waring blender (John Morris Scientific, Chatswood, NSW, Australia), and passed through a 1 mm EFL 2000 stainless steel sieve (Endecotts, London, UK). The ground freeze-dried bitter melon preparation was then stored at −20 °C until use.

2.3. Aqueous extraction

The ground freeze-dried bitter melon preparation (1 g) was extracted with 20 ml of deionised water at 40 °C for 15 min using a shaking water bath (Ratek Instruments, Boronia, VIC, Australia) as previously optimised [4]. The extract was cooled on ice for 10 min and then vacuum-filtered through several layers of cheesecloth and finally through a Whatman No. 1 filter paper (Lomb Scientific, Taren Point, NSW, Australia) to remove insoluble bitter melon residues.

2.4. Infeed solutions

A 1:1 (w/w) combination of maltodextrin (MD) and gum arabic (GA) was chosen as the encapsulating agent due to its high stability in infeed solutions and the high quality of encapsulated powder it can produce [19,22]. To prepare the encapsulating agent stock solution, 35 g of the encapsulating agents (MD/GA, 1:1 w/w) was homogenised with 100 g of deionised water using a Silverson L4RT high shear mixer (J L Lennard Pty. Ltd., Silverwater, NSW, Australia) at 4500 rpm for 10 min. Then, 180 g of the bitter melon aqueous extract and 120 g of the encapsulating agent stock solution (a ratio of 1.5:1 w/w) were mixed to give a total solid content in the final infeed solution of 11% (w/w). The mixture was homogenised for 10 min and then placed in an ultrasound water bath (Ultrasonik 57X, Extech Equipment Pty. Ltd., Wantirna South, VIC, Australia) for 20 min to promote the microencapsulation process [28].

2.5. Experimental design

The optimal inlet and outlet temperatures for the encapsulation of the bitter melon aqueous extract by spray drying were determined using response surface methodology. The 2^3 Central Composite Design (CCD) was used to obtain a second-order model for the prediction of dependent responses, including process yield (Y_1), retentions of saponins (Y_2), phenolics (Y_3), flavonoids (Y_4), and antioxidant activity (Y_5) and for moisture content (Y_6), the water solubility index (Y_7), water activity (Y_8) bulk density (Y_9), particle size distribution (Y_{10}) and water absorption index (WAI) (Y_{11}).

The two independent variables, inlet temperature (X_1) and outlet temperature (X_2), were considered at five levels as shown in Table 1. The experimental data were fitted to a second-order polynomial equation as follows:

$$Y_i = a_o + a_1X_1 + a_2X_2 + a_{11}X_1^2 + a_{22}X_2^2 + a_{12}X_1X_2; \quad (1)$$

where Y is the dependent response; X_1 and X_2 are the levels of the independent variables, and a_o, a_i, a_{ii} and a_{ij} are the regression coefficients of the variables for the offset, linear, quadratic and interaction terms, respectively.

2.6. Encapsulation by spray-drying

The infeed solutions were spray-dried using a laboratory-scale spray drier (Buchi Mini Spray Drier B-290, Noble Park, VIC, Australia) according to a method described by Fang and Bhandari [29] with slight modifications. The drying air flow rate and compressed air flow were set at 35 m^3/h and 473 L/h, respectively. Based on the model in Table 1 and preliminary experiments, the inlet temperature (X_1) values tested were 125.6, 130, 140, 150 and 154.1 °C and the outlet temperature (X_2) values were 72.9, 75, 80, 85 and 87.1 °C. The inlet temperatures were directly set, the outlet temperatures were controlled using the feed flow rate and the aspiration was set at 100%.

After spray-drying, the encapsulated powder was cooled in a desiccator containing silica gel to prevent moisture absorption and weighed to determine the yield of powder. The encapsulated powder

Table 1
Inlet and outlet temperature levels for the CCD RSM model.

Coded variable levels	Inlet temperature X_1 (°C)	Outlet temperature X_2 (°C)
+1.414	154.1	87.1
+1	150	85
0	140	80
−1	130	75
−1.414	125.6	72.9