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Inulin suitable as reduced-kilojoule carrier for production of microencapsulated spray-dried green *Cyclopia subternata* (honeybush) extract

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ABSTRACT

Retention of phenolic compounds during spray-drying of an anti-diabetic *C. subternata* extract and physicochemical characteristics of spray-dried powders (pure extract and extract-carrier mixtures) were evaluated. Extract-carrier mixtures contained three levels (250, 500 and 750 g/kg) of the microencapsulating agents, namely corn syrup solids, commonly used by the food industry, and inulin, a low-kilojoule alternative. The amorphous spray-dried powders ranged from nearly free-flowing to cohesive. Their moisture content and water activity fell within the range of their monolayer moisture values. The moisture sorption isotherm of the pure extract showed very little hysteresis, contrary to the mixtures containing carriers. Similar values for calculated and experimental heat flow, determined by isothermal microcalorimetry, indicated the carriers to be compatible with the extract, except when used in a mixture containing 750 g/kg corn syrup solids per total solids. Spray-drying had no detrimental effect on the individual phenolic content, in particular the heat labile mangiferin, isomangiferin and 3-β-D-glucopyranosyliriflophenone, and the total antioxidant capacity of the extract. Microencapsulation of *C. subternata* extract with inulin by spray-drying thus provides a stable low-kilojoule powder, suitable for formulation of single-serve beverage mixtures that can be used by diabetics.

1. Introduction

In the ongoing search for novel sources of bioactive compounds *Cyclopia* species (honeybush) have been identified for the production of value-added extracts for use in nutraceuticals and functional food products. Of particular interest is the xanthone, mangiferin, found in high concentrations in *Cyclopia* spp. Its potent antioxidant capacity and other health benefits, which include anti-diabetic properties, are well-documented (Vyas, Syeda, Ahmad, Padhye, & Sarkar, 2012). *Cyclopia subternata* also contains substantial quantities of isomangiferin with similar glucose-lowering activity *in vitro* to mangiferin (Schulze et al., 2016). Other compounds of interest are the benzophenone α-glucosidase inhibitors (Beelders et al., 2014), contributing to the anti-diabetic activity of *Cyclopia* extracts through postprandial regulation of blood glucose levels. An aqueous extract of "unfermented" *C. subternata* has been demonstrated to have glucose lowering (Schulze et al., 2016) and anti-obesity properties (Dudhia et al., 2013), further substantiating interest in honeybush for the production of functional products. Other phenolic glycosides present in this extract include flavanones, dihydrochalcones and a flavone (De Beer et al., 2012; Schulze et al., 2016).

A common application of tea and herbal tea extracts is the production of ready-to-drink iced teas and "instant" teas, i.e. dry formulated powder mixtures sold in bulk or convenient single-serve format. For these purposes a stable, free-flowing powder is required for further formulation. Spray-drying is used extensively to produce dry powders from natural extracts (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). It is common practice to microencapsulate natural extracts by spray-drying with carriers. Microencapsulation of the extract aids protection of phenolic

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Abbreviations

a_w	water activity
BET model	Brunauer, Emmett and Teller model
CS	corn syrup solids
CS25	*C. subternata* extract spray-dried with 250 g CS/kg
CS50	*C. subternata* extract spray-dried with 500 g CS/kg
CS75	*C. subternata* extract spray-dried with 750 g CS/kg
DE	dextrose equivalents
DP	degree of polymerisation
DSC	differential scanning calorimetry
DTA	differential thermal analysis
DTG	simultaneous TG/DTA
HPLC-DAD	high-performance liquid chromatography with diode-array detection
IN	inulin
IN25	*C. subternata* extract spray-dried with 25 g IN/kg
IN50	*C. subternata* extract spray-dried with 500 g IN/kg
IN75	*C. subternata* extract spray-dried with 750 g IN/kg
M_0	monolayer moisture content
MSI	moisture sorption isotherm
RH	relative humidity
SEM	scanning electron microscope
TAC	total antioxidant capacity
T_g	glass transition temperature
TG	thermogravimetry
TPC	total polyphenol content
XRPD	X-ray powder diffraction

compounds during spray-drying, as well as improves the physicochemical properties of the powder (Munin & Edwards-Lévy, 2011). A common carrier used by industry in a variety of food products is corn syrup solids, a cheap, versatile and readily available product. However, it has a similar metabolic effect to that of sugar (Gross, Li, Ford, & Liu, 2004) with an energy value of 16.3 kJ/g (Roberfroid, 1999) making it unsuitable for products aimed at health-conscious or diabetic consumers. Inulin, on the other hand, is a low-kilojoule (6.3 kJ/g) and indigestible prebiotic fibre (Barclay, Ginic-Markovic, Cooper, & Petrovsky, 2010; Schaller-Povolny, Smith, & Labuza, 2000) that stimulates the growth of beneficial bifidobacteria species (Kolida, Tuohy, & Gibson, 2002). Inulin is thus a healthy alternative to corn syrup solids for use in functional beverages.

The objectives of this study were to determine the effect of spray-drying on the total antioxidant capacity and phenolic composition of green *C. subternata* extract. Different ratios of the microencapsulating agents, corn syrup solids and inulin, added to the extracts were investigated to improve the physicochemical properties of the powder. Compatibility of the extract with corn syrup solids and inulin was evaluated to indicate the potential storage stability of the extract when microencapsulated with these carriers. Adsorption and desorption moisture isotherms, amorphous/crystalline nature, thermal characteristics and bulk properties of the spray-dried powders were determined to gain insight into their expected stability during storage.

2. Materials and methods

2.1. Chemicals, reagents, extracts and carriers

Authentic reference standards (purity > 950 g/kg) were from Sigma-Aldrich (St Louis, USA: mangiferin, hesperidin, maclurin, 3-β-D-glucopyranosyliriflophenone), Extrasynthese (Genay, France: luteolin) and Phytolab (Vestenbergsgreuth, Germany: eriocitrin, vicenin-2). Sigma-Aldrich supplied HPLC gradient grade acetonitrile for HPLC analysis. Deionised water, prepared using an Elix® water purification system (Merck-Millipore, Darmstadt, Germany), was further purified to HPLC grade using a Milli-Q™ Reference A+ System (Merck-Millipore). Analytical grade chemicals were obtained from either Sigma-Aldrich or Merck-Millipore.

A vacuum-dried, hot water extract of unfermented *C. subternata* with proven *in vivo* glucose-lowering activity (Schulze et al., 2016) was used to prepare the liquid feed solution for spray-drying with or without added carrier. Star Dri® 200 corn syrup solids with 20–23 dextrose equivalents (DE) was kindly donated by Tate and Lyle (Cape Town, South Africa). Orafti® HP inulin, a long-chain inulin food additive derived from chicory roots (*Cichorium intybus*) with 21–26 degrees of polymerisation (DP), was procured from Savannah Fine Chemicals (Pty) Ltd (Gardenview, South Africa).

2.2. Spray-drying treatments

The dry extract and carriers were mixed in predefined ratios to obtain a total mass of 40 g solids. The feed solution (100 g/L) was prepared by adding the dry powder mixtures slowly to deionised water (400 mL) at *ca* 55 °C, while stirring on a magnetic stirrer until completely dissolved (*ca* 10 min). Each carrier was added at three treatment levels, namely at 250, 500 and 750 g/kg of the total solids content. A control sample consisting of pure extract was also spray-dried. Each of the treatments was replicated 4 times (8 replicates for control) in a completely randomised design.

Spray-drying was conducted using a Büchi B-290 mini spray-dryer (Büchi Labortechnik AG; Flawil, Switzerland) equipped with a glass cyclone separator and a 1.5 mm nozzle aperture diameter. The feed solution was sprayed in a co-current direction using air as a drying medium. Following preliminary experiments to assess suitability of operating conditions in terms of powder yield (>600 g/kg) and moisture content, final operating conditions selected were: inlet temperature, 180 °C, aspirator rate, 35 m^3/h, peristaltic pump speed, 7.5 mL/min, atomisation air flow rate, 667 L/h, and nozzle cleaner, 8 strikes/min. The outlet temperature (*ca* 90–100 °C) varied depending on the composition of the feed solution. The collected powder was weighed in amber vials to determine the powder yield (calculated as g powder obtained per kg solids in the feed solution) and stored at ambient temperature in a desiccator with silica gel until analysis.

2.3. Characterisation of powders

2.3.1. Moisture content, water activity and moisture sorption isotherms

The moisture content of the spray-dried powders was determined by drying *ca* 2 g of sample at 100 °C for 60 min using an HR73 Halogen Moisture analyser (Mettler Toledo; Greifensee, Switzerland) and expressed as g/kg of the total mass of the product (wet basis), as well as g/kg of dry mass (dry basis). Water activity (a_w) was determined using a Novasina LabMASTER-aw electric hygrometer (Lachen, Switzerland).

Moisture sorption analyses of the spray-dried powders were