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Resistant maltodextrin as a shell material for encapsulation of naringin: Production and physicochemical characterization

Dhananjay A. Pai [a], Venu R. Vangala [a,b], Jun Wei Ng [a], Wai Kiong Ng [a,*], Reginald B.H. Tan [a,c,*]

[a] Crystallisation and Particle Science, Institute of Chemical and Engineering Sciences, A* STAR, 1 Pesek Road, Jurong Island, Singapore 627833, Singapore
[b] Centre for Pharmaceutical Engineering Science, School of Pharmacy, University of Bradford, Bradford BD7 1DP, United Kingdom
[c] Department of Chemical & Biomolecular Engineering, National University of Singapore, 4, Engineering Drive 4, Singapore 117576, Singapore

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ABSTRACT

Herein the potential of a relatively new water soluble fiber, resistant maltodextrin (RMD) to encapsulate grapefruit polyphenol, naringin, using spray drying was evaluated. Full factorial Design Of Experiments (DOE) for spray drying with two levels of fiber–naringin ratio and spray dryer inlet temperature was executed. Resulting powders were characterized with respect to particle size and morphology, crystallinity, thermal properties, moisture sorption and naringin aqueous solubility increase. A 60–80% encapsulation was achieved. Thermal and moisture sorption behaviors of these dispersions were found to be dominated by RMD. By varying fiber–naringin ratio and spray drying temperatures, naringin was able to disperse in amorphous form in RMD matrix, which led to 20–55% increase in aqueous solubility. Solubility enhancement was found to correlate positively with increasing fiber: naringin ratio and spray drying temperature due to multiple factors discussed in this study. In conclusion, fiber–polyphenol bicomponent nutraceutical was successfully developed based on a well-established encapsulation technology i.e. spray-drying.

1. Introduction

Resistant maltodextrin (RMD) is a randomly linked alpha-glucoside oligosaccharide with an average degree of polymerization of 10–15. It is a low glycemic index (10% that of maltodextrin) (Goda et al., 2006), undigestible fiber with almost 40% being fermented into short chain fatty acids (SCFA) (Fastinger et al., 2008). Most of the research work on RMD reported so far in the literature has focused on the nutritional benefits such as increase in *bifidobacterium* spp. populations in colon (LeFranc-Millot et al., 2012; Vester Boler et al., 2011; Fastinger et al., 2008), increased satiety (Guérin-Deremaux et al., 2011), and increase in fecal weight (Timm et al., 2013; Vester Boler et al., 2011) which facilitates treatment of idiopathic chronic constipation (Braquehais and Cava, 2011). However, we have only come across one recent report (Chen et al., 2013) on use of RMD as an encapsulating material using spray and freeze drying processes which mainly focuses on sensory and stability profiles. Also, there have also been no reports on the fundamental solid state properties of RMD which could enable its usage for wider applications.

Naringin (NN) is the most prominent bioflavonoid in grapefruit juice and is also responsible for its bitter taste that leads to low consumer acceptance (Drewnowski et al., 1997). In a clinical study by Jung et al. (2003), an intake of 600 mg of naringin every day for 8 weeks was reported to a decrease of total cholesterol and LDL by 14% and 17% respectively. The bioavailability of flavonoids is limited by factors such as susceptibility to oxidation, degradation in acidic pH, poor water solubility and aqueous dissolution rates (Sansone et al., 2009). Naringin exhibits low water solubility (Lauro et al., 2007) which has been hypothesized as the rate limiting step for its absorption in the body (Kanaze et al., 2006a). Hence, solubility enhancement may be one of the ways, if not the ultimate one, to improve bioavailability. Despite demonstrated health benefits, the bitterness and poor water solubility limit its use in processed foods. Microencapsulation is a popular technique used to overcome these formulation challenges (Gharsallaoui et al., 2007). There are a few reports in the pharmaceutical literature for encapsulation of naringin such as production of microparticles for respiratory drug usage (Sansone et al., 2009) and gastroresistant microparticles (Lauro et al., 2007), both using spray drying. Kanaze et al. (2006a,b) attempted encapsulation of naringin in polyvinylpyrrolidone (PVP) and polyethylene glycol (PEG) matrices, using solvent evaporation method. These reported works were

* Corresponding authors at: Crystallisation and Particle Science, Institute of Chemical and Engineering Sciences, A* STAR, 1 Pesek Road, Jurong Island, Singapore 627833, Singapore. Tel.: +65 6796 3863.
E-mail addresses: ng_wai_kiong@ices.a-star.edu.sg (W.K. Ng), reginald_tan@ices.a-star.edu.sg (R.B.H. Tan).

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conducted using pharmaceutical grade shell materials which cannot be applied for application in food industry. Only Ficarra et al. (2002) and Cui et al. (2012) have reported formation of inclusion complex of naringin in food grade β-cyclodextrin that improves its solubility significantly. However, the high cost of β-cyclodextrin is an impediment for its use for food product development. Although research in this area is in preliminary stages, results have shown a very strong radical scavenging antioxidant capacity of a fiber–polyphenol combination in the digestive system. In a recent work, Saura-Calixto (2011) has discussed the important role of indigestible fibers in carrying polyphenols to the colon, followed by metabolism of these polyphenols into health beneficial end products. The goal of current work was to investigate the potential of RMD as an encapsulating shell material for naringin using spray drying. The specific objective is to understand the influence of RMD:NN ratio in the formulation and spray drying inlet temperature, on properties related to solid state stability as well as solubility enhancement of encapsulated naringin.

2. Materials and methods

2.1. Materials

Naringin was purchased from Sigma Aldrich chemical company (St. Louis, MO, USA). Resistant maltodextrin (Promitor 85™) was obtained from Tate & Lyle Co. (Chicago, IL, USA). Ultrapure water was obtained from Millipore Milli-Q® Gradient A10 water system. Analytical grade ethanol was obtained from JT Baker (Center Valley, PA, USA).

2.2. Methods

2.2.1. Spray drying DOE and preparation of physical mixtures

A 2^2 full factorial study was conducted for spray drying with the following variables: RMD solution concentration and spray drying inlet temperatures. A total of four, 100 mL naringin (3% w/v) suspensions were prepared by slowly adding the polyphenol to a solution of RMD prepared in ultrapure water, accompanied by stirring with a magnetic bar at 1000 rpm for 20 min. The RMD solutions were prepared at two concentrations (20 and 40% w/v). Spray drying was conducted in an amber colored spray drying chamber since polyphenols are usually light sensitive (Fang and Bhandari, 2010) at temperatures of 155 and 180 °C using a nozzle of 0.7 mm diameter and constant feed flow rate of 3 mL/min. The full experimental design and assigned formulation codes in shown in Table 1. Only one trial was carried out per combination of RMD concentration and spray drying inlet temperature.

Naringin (3% w/v) suspensions in water (100 mL) without any added fiber were also spray dried at 155 °C and 180 °C (coded as NN-155 and NN-180) to be used as controls for selected characterization measurements. All final powders from the spray dryer were stored in screw capped amber colored bottles within a dry box. Two physical mixtures of the same ratios as the one used for spray drying, PM1 (93 RMD:7 NN) and PM2 (87 RMD:13 NN) were prepared by blending the weighed powders in a Turbula mixer (WAB, Switzerland) at 15 rpm for 20 min. The final mix was stored in a dry box maintained at 20% RH in amber colored glass bottles. The spray drying outlet temperatures were found to range from 95 ± 3 to 110 ± 4 °C for inlet temperatures of 155 to 180 °C respectively. There was minimal sticking of powder to the spray drying chamber surface and the chamber was easy to be cleaned, both of which are desirable aspects for an industrial scale process.

Table 1
Spray drying full-factorial DOE.

Formulations with 3% w/v naringin	Concentration of RMD in spray drying slurry (% w/v)	Spray drying inlet temperature (°C)
A	20	155
B	20	180
C	40	155
D	40	180

2.2.2. Particle morphology and size distribution

The morphology of pure RMD, NN, physical mixtures as well as final spray dried formulations were observed under JEOL JSM-6700F Field Emission Scanning Electron Microscope (FESEM) (JEOL Ltd, Japan), operating at an accelerating voltage of 5 kV under lower secondary electron imaging (LEI) mode. Each sample was dispersed onto a carbon adhesive-coated metal stubs and sputter coated with platinum for 1 min at 20 mA using Cressington 208 HR Sputter Coater (Cressington Scientific Instruments Inc., UK) prior to analysis.

The particle size distribution of spray dried formulations was determined using laser diffraction technique (Mastersizer 2000, Malvern Instruments Ltd., UK). The analysis was conducted in a dry mode (Scirocco dry dispersion unit, Malvern Instruments Ltd., UK) for these measurements. All samples were measured in triplicates at a feed pressure of 3 bars, which was finalized following pressure titration and a feed vibration rate of 50%. All samples were passed through a sieve with ball bearings in order to ensure breaking up of agglomerates and obtain reproducible results.

2.2.3. Encapsulation efficiency measurement

Encapsulation efficiency (EE) of naringin was defined in Eq. (1) as:

$$EE = \frac{\text{Experimental naringin content}}{\text{Theoretical naringin content}} \quad (1)$$

In general, HPLC is the preferred method for quantification studies. Herein Folin–Ciocalteu method which is based on UV–Vis spectrometry and a specific method for polyphenol estimation was chosen for quantification of naringin. Specifically for naringin, Lauro et al. (2007) have reported UV–Vis spectrometry to be as reliable as HPLC measurements. It was found that RMD showed an absorbance peak at 286 nm, interfering with that of narignin, thus preventing direct use of UV–Vis measurement. Hence, the naringin content in each of the spray dried samples was measured using Folin–Ciocalteu method which is a specific method of analysis for polyphenols. A 2000 ppm stock solution was prepared by dissolving naringin in pure ethanol. Standard solutions of naringin (125–1000 μg/mL) were prepared to construct the standard curve, by suitably diluting the stock solution with ultrapure water. A 10 mL solution for final absorbance measurement was prepared by adding the necessary chemicals in the following order: 0.5 mL naringin standard solution +4.5 mL ultrapure water +0.2 mL Folin's reagent +0.5 mL saturated sodium carbonate solution +4.3 mL ultrapure water. The samples were then kept at room temperature for 1 h for full color development, following which the absorbance was measured in a 3 mL quartz cuvette at 975 nm using a UV–Vis spectrophotometer (Varian Cary 50, Agilent Technologies, US). Although the standard protocol for Folin–Ciocalteau method for detection of bluish complex is around 730–760 nm, this work has followed the measurement of peak at 975 nm according to the work of Espinosa Bosch et al. (2008).

For measurement of naringin content in spray dried samples, 100 mg of each spray dried formulation was completely dissolved in 5 mL of 1:1 ultrapure water–ethanol solvent mixture and stirred for 1 h to ensure complete solubility of the powders. The solutions were then passed through a 0.2 μm Nylon filter. A 10 mL aliquot for absorbance reading was prepared, composed of 0.5 mL sample