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Phytochemical content and antioxidant activity of grapefruit (Star Ruby): A comparison between fresh freeze-dried fruits and different powder formulations

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Narirutin (PubChem CID: 442431)
2,2-Diphenyl-1-(2,4,6-trinitrophenyl) hydrazyl (PubChem CID: 2735032)
Potassium ferricyanide (PubChem CID: 26250)
Beta-carotene (PubChem CID: 5280489)
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ABSTRACT

Different grapefruit powders obtained by freeze drying and spray drying with prior addition of shell materials (arabic gum and bamboo fiber) were studied in order to evaluate the effect of these preservation processes on the retention of antioxidants, in comparison with the freeze-dried fruit with no carriers added. Freeze-dried samples showed above 90% retention of these phytochemicals, while spray-dried samples presented good retention of vitamins but a sharp decrease in of phenolic compounds. Pearson's correlation analysis showed that the most significant contribution to DPPH scavenging activity and inhibition of β-carotene bleaching was provided by phenolic compounds, mostly flavonoids, while the contribution to the reducing power was due to ascorbic acid and α-tocopherol. Therefore, the loss of these compounds in the spray-dried samples resulted in products with lower antioxidant activity. Naringin and narirutin were the major phenolic compounds in all grapefruit samples, although other flavanones present in lower concentration, like hesperidin, neohesperidin didymin, poncirin or melitidin, also showed high correlations with the antioxidant value of the samples.

1. Introduction

Grapefruit is a very common variety of citrus fruit and an important source of bioactive compounds such as vitamins C, E, A, phenolic compounds (flavonoids, phenolic acids and coumarins), and terpenic substances, such as carotenoids and limonoids (Kelebek, 2010; Zou, Xi, Hu, Nie, & Zhou, 2015). In recent years, the phenolic compounds present in grapefruit have been investigated,

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and some publications have suggested that they could play an important role in the antioxidant capacity of grapefruit juice (Gorinstein et al., 2005; Xu et al., 2008), which has been related with the prevention of different chronic diseases including heart disease, obesity, diabetes, cardiovascular diseases and cancer (Mertens-Talcott, Zadezensky, De Castro, Derendorf & Butterweck, 2006; Vanamala, Reddivari, Yoo, Pike, & Patil, 2006; Díaz-Juárez et al., 2009). Some epidemiological studies also pointed to the consumption of grapefruit brings benefits in weight loss and improve lipid metabolism (Dow, Going, Chow, Patil, & Thomson, 2012; Gorinstein et al., 2005). However, despite its high functional value, the consumption of fresh grapefruit is low, probably due to its strong bitter taste and also because it is produced on a seasonal basis, so that in many countries it may not be available in fresh conditions throughout the year. Dried and powdered products can overcome this problem, as they more stable than fresh fruit and easier to store and distribute, making them available all around the year. Freeze-drying and spray-drying are two techniques used for the production of fruit powder (Fernandes, Rodrigues, Law, & Mujumdar, 2011). Nevertheless, the process used to obtain the powder must ensure the maximal preservation of the bioactive or functional fruit compounds, with the type of shell materials used to protect those compounds playing an important role in the antioxidant capacity of the final product (Tonon, Brabet, Pallet, Brat, & Hubinger, 2009; Fang & Bhandari, 2012).

In this study, freeze-drying and spray-drying have been applied to obtain powdered grapefruit and their effects on the antioxidant capacity and the levels of ascorbic acid, α-tocopherol and phenolic compounds of the product have been investigated and discussed. The effect of arabic gum and bamboo fibre added as shell materials has been considered.

2. Materials and methods

2.1. Raw material

The study was carried out with different samples of grapefruit (*Citrus paradisi var. Star Ruby*) purchased in local supermarkets in Valencia (Spain). Grapefruits were washed and peeled with careful removal of the albedo. Arabic gum (AG, Scharlau, Spain) and bamboo fiber (BF, VITACEL®, Rosenberg, Germany) were added to the grapefruit pulp as shell materials for the drying process.

2.2. Sample's preparation

Prior to freeze-drying (FD), peeled grapefruits were cut and ground using a bench top food processor (Thermomix TM 21, Vorwerk, Spain), whereas for spray-drying (SD) they were liquidized in a domestic device (DeLonghi, Spain). Six formulations (4 for FD and 2 for SD) containing different proportions of the shell materials (AG and BF) or water content, selected according to a previous study (Agudelo, Igual, Camacho, & Martínez-Navarrete, 2016), were prepared (Table 1). For FD formulations, AG and BF were mixed with ground grapefruit and afterwards the samples were placed in aluminium pans (approximately 250 g in 0.5 cm thickness by pan) and immediately frozen at −45 °C (Liebherr Mediline, LCT2325, Germany) for 48 h before freeze-drying in a Telstar Lioalfa-6 Lyophyliser at 0.021 Pa and −59 °C. The obtained cakes were ground (Kenwood, CH 580, Spain) and sieved to obtain powder with a particle size lower than 0.7 mm. For SD formulations, AG and BF were dissolved in distilled water in the desired proportions and mixed with the liquidized grapefruit in relation 1:1 (AG-BF solutions: liquidized grapefruit). After that, the mixture was fed into a Büchi B-290 (Switzerland) mini spray dryer with the following operating conditions: aspirator rate 90% (35 m^3/h); atomisation air rotameter 40 mm (473 L/h) with a co-current flow; pump rate 30% (9 mL/min), and drying air inlet temperature 120 °C. After completion of the process and when the air inlet temperature fell below 50 °C, the samples were collected from the product collection vessel for further characterization. To verify the effect of using the carriers, the ground and liquidized grapefruit without shell materials added were also freeze-dried under the same conditions (GG and LG samples, Table 1). It was not possible to spray dry the liquidized sample without carriers.

Table 1
Freeze dried ground (GG) or liquidized (LG) grapefruit and different formulations of ground grapefruit used for freeze drying (FD) or liquidized grapefruit used for spray drying (SD).

	Formulation	Type of shell material and their content (g/100 g GG or GL)	
		Arabic Gum (AG)	Bamboo Fiber (FB)
Freeze dried grapefruit			
1	GG	–	–
2	LG	–	–
3	FD_1[a]	4.2	0.58
4	FD_2	4.2	0.58
5	FD_3	4.2	0
6	FD_4	0	0.58
Spray dried grapefruit			
7	SD_1	4	2
8	SD_2	4	0

[a] Prior to freeze drying the mixture was hydrated to a level of 90 g_{water}/100$g_{product}$.

2.3. Compound analyses

2.3.1. Ascorbic acid

Ascorbic acid was determined following a procedure previously described by Pereira, Barros, Carvalho, and Ferreira (2013) and the analysis was performed by ultra-fast liquid chromatography coupled to photodiode array detection (UFLC-PDA; Shimadzu Cooperation, Kyoto, Japan), using 245 nm as preferred wavelength. Results were expressed in g per 100 g of grapefruit's own solutes (GS).

2.3.2. Tocopherols

Tocopherols were determined following a procedure previously described by Barros, Heleno, Carvalho, and Ferreira (2010), using a HPLC system (Knauer, Smartline system 1000, Berlin, Germany) coupled to a fluorescence detector (FP-2020; Jasco, Easton, USA) programmed for excitation at 290 nm and emission at 330 nm, using the IS (tocol) method for quantification. The results were expressed in mg per 100 g GS.

2.3.3. Phenolic compounds

Grapefruit samples (1 g) were extracted with methanol/water (80:20, v/v, 30 mL) by mechanical maceration (150 rpm, 25 °C) during 1 h. Afterwards, the sample was filtered using a Whatman no. 4 paper and the residue was re-extracted with an additional portion of the solvent. The extracts were combined and the methanol was evaporated using a rotary evaporator (Büchi R-210; Flawil, Switzerland) and then the aqueous phase was further lyophilized (FreeZone 4.5, Labconco, Kansas City, MO, USA). Each extract (10 mg) was dissolved in water:methanol (80:20 v/v), filtered through 0.2 μm nylon filters and analysed by HPLC-DAD-ESI-MSn in a Hewlett–Packard 1100 equipment (Agilent Technologies, Waldbronn, Germany) connected to a mass spectrometer (API 3200 Qtrap, Applied Biosystems, Darmstadt, Germany) as previously described by the authors (Pinela et al., 2012). Results