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

Running title: Antioxidants in grapefruit fresh freeze-dried fruits and powder formulations

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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.

Chemical compounds:

Ascorbic acid (PubChem CID: 54670067); Alpha-Tocopherol (PubChem CID: 14985); Naringin (PubChem CID: 25075); 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), Thiobarbituric acid (PubChem CID: 2723628), Trolox (PubChem CID: 40634).

Keywords: Spray-drying/Freeze-drying; Antioxidant activity; Bioactive compounds; Arabic gum; Bamboo fiber

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, and some publications have suggested that they could play an important role in the antioxidant capacity of grapefruit juice (Gorinstein et al., 2005; Xu, Liu, Chen, Ye, Ma & Shi, 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, Tenorio-López, Zarco-Olvera, Valle-Mondragón, Torres-Narváez & Pastelín-Hernández, 2009). Some epidemiological studies also pointed to the consumption of grapefruit brings benefits in weight loss and improve lipid metabolism (Gorinstein et al., 2005; Dow, Going, Chow, Patil & Thomson, 2012). 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 & Mujundar, 2011). Nevertheless, the process used to obtain

71 the powder must ensure the maximal preservation of the bioactive or functional fruit
72 compounds, with the type of shell materials used to protect those compounds playing an
73 important role in the antioxidant capacity of the final product (Tonon, Brabet, Pallet, Brat
74 & Hubinger, 2009; Fang & Bandari, 2012).
75 In this study, freeze-drying and spray-drying have been applied to obtain powdered
76 grapefruit and their effects on the antioxidant capacity and the levels of ascorbic acid, α-
77 tocopherol and phenolic compounds of the product have been investigated and discussed.
78 The effect of arabic gum and bamboo fibre added as shell materials has been considered.
79

80 **2. Materials and methods**

81

82 *2.1. Raw material*

83 The study was carried out with different samples of grapefruit (*Citrus paradisi var. Star*
84 *Ruby*) purchased in local supermarkets in Valencia (Spain). Grapefruits were washed and
85 peeled with careful removal of the albedo. Arabic gum (AG, Scharlau, Spain) and bamboo
86 fiber (BF, VITACEL®, Rosenberg, Germany) were added to the grapefruit pulp as shell
87 materials for the drying process.
88

89 *2.2. Sample's preparation*

90 Prior to freeze-drying (FD), peeled grapefruits were cut and ground using a bench top food
91 processor (Thermomix TM 21, Vorwerk, Spain), whereas for spray-drying (SD) they were
92 liquidized in a domestic device (DeLonghi, Spain). Six formulations (4 for FD and 2 for
93 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 ºC (Liebherr Mediline, LCT2325, Germany) for 48 h before freeze-drying in a Telstar Lioalfa-6 Lyophyliser at 0.021 Pa and -59 ºC. 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 ºC. After completion of the process and when the air inlet temperature fell below 50 ºC, 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.

2.3. Compound analyses

2.3.1. Ascorbic acid. Ascorbic acid was determined following a procedure previously described by Pereira et al. (2013) and the analysis was performed by ultra-fast liquid chromatography coupled to photodiode array detection (UFLC-PDA; Shimadzu

117 Coperation, Kyoto, Japan), using 245 nm as preferred wavelength. Results were expressed
118 in g per 100 g of grapefruit's own solutes (GS).
119
120 *2.3.2. Tocopherols.* Tocopherols were determined following a procedure previously
121 described by Barros et al., (2010), using a HPLC system (Knauer, Smartline system 1000,
122 Berlin, Germany) coupled to a fluorescence detector (FP-2020; Jasco, Easton, USA)
123 programmed for excitation at 290 nm and emission at 330 nm, using the IS (tocol) method
124 for quantification. The results were expressed in mg per 100 g GS.
125
126 *2.3.3. Phenolic compounds.* Grapefruit samples (1 g) were extracted with methanol/water
127 (80:20, v/v, 30 mL) by mechanical maceration (150 rpm, 25 ºC) during 1 h. Afterwards, the
128 sample was filtered using a Whatman no. 4 paper and the residue was re-extracted with an
129 additional portion of the solvent. The extracts were combined and the methanol was
130 evaporated using a rotary evaporator (Büchi R-210; Flawil, Switzerland) and then the
131 aqueous phase was further lyophilized (FreeZone 4.5, Labconco, Kansas City, MO, USA).
132 Each extract (10 mg) was dissolved in water:methanol (80:20 v/v), filtered through 0.2 μm
133 nylon filters and analysed by HPLC-DAD-ESI-MS_n in a Hewlett–Packard 1100 equipment
134 (Agilent Technologies, Waldbronn, Germany) connected to a mass spectrometer (API 3200
135 Qtrap, Applied Biosystems, Darmstadt, Germany) as previously described by the authors
136 (Pinela et al., 2012). Results were expressed mg/100 g GS.
137
138 The dehydrated samples possessed different proportions of added solutes, so that in order to
139 make the results comparable to evaluate the effects of the dehydration processes on the

vitamins content and phenolic compounds, the results were referred to the grapefruit's own solutes (GS) according to Eq. 1 and Eq. 2.

$$m_i = \frac{(m_{ip}/m_p)}{(1-x_w{}^p)(x_{GS/TS})} \quad (1)$$

$$X_{GS/TS} = \frac{m_g(1-x_w{}^g)}{(m_{AG}+m_{FB}\pm m_{H20}(1-x_w{}^g)} \quad (2)$$

Where: m_i is the mass of each compound refered to grapefruit solutes (mg/ g GS); m_{ip} is the mass of each compound analysed in the powder (mg/g), $x_w{}^p$ is the water content of the powder (g_{water}/g_{powder}), $x_{GS/TS}$ is the mass fraction of grapefruit solutes (GS) to total solutes (TS), m_g, m_{AG} and m_{BF} are the mass of ground or liquidized grapefruit, arabic gum and bamboo fibre, respectively, in the sample and $x_w{}^g$ is the water content of the ground or liquidized grapefruit (w/w).

2.4. Antioxidant activity

The methanol/water (80:20, v/v) extracts described above (section 2.3.3) were re-dissolved (methanol/water, 80:20, v/v) to a concentration of 10 mg/mL (stock solution). Six successive dilutions were made starting from the stock solution and further submitted to the different *in vitro* antioxidant assays as previously described by Fernandes, Barreira, Antonio, Oliveira, Martins and Ferreira (2016). The antioxidant activity was evaluated using four in vitro assays: DPPH radical-scavenging activity, reducing power, inhibition of β-carotene bleaching in the presence of linoleic acid radicals and inhibition of lipid peroxidation using TBARS in brain homogenates. The extract concentrations providing 50% of antioxidant activity or 0.5 of absorbance (EC_{50}) were calculated from the graphs of antioxidant activity percentages (DPPH, β-carotene bleaching and TBARS assays) or

absorbance at 690 nm (reducing power assay) against extract concentrations. Trolox was used as standard.

2.5. Statistical analysis

Analyses of variance (ANOVA) were carried out to evaluate the effect of drying treatments. When the *p* value was lower than 0.05, significant differences between samples were considered. Furthermore, a Pearson's correlation analysis between the antioxidant activity and all the analysed compounds was carried out, with a 95% significance level. All the statistical analyses were performed using Statgraphics Centurion XV.

3. Results and discussion

3.1 Effects on vitamins and phenolic compounds

The freeze-drying process works with either whole or ground fruits, while spray drying requires an input feedstock with low viscosity and small particle size. For this reason, the grapefruit was liquidized and diluted to obtain a fluid that met the conditions of the spray dryer. **Table 2** collects the levels of acid ascorbic and α-tocopherol in the different analysed preparations. In general, the values obtained for the content of these vitamins in GG and LG samples were similar to those shown in the literature for ascorbic acid (Moraga, Igual, García-Martínez, Mosquera & Martínez-Navarrete, 2012) and α- tocopherol (Chun, Lee, Ye, Exler & Eitenmiller, 2006 ; USDA Natl. Nutrien Database, 2011) in pink grapefruit varieties. Significant differences ($p<0.05$) were found in the contents of both vitamins between the two samples without shell materials added (GG and LG), with a better vitamin retention in the liquefied fruits further used for preparation of the spray-dried (SD) samples.

186 According to Park, Lee & Eun (2016) freeze-drying usually conduct to lower losses in
187 comparison with other techniques like hot air drying, because the low temperature and the
188 absence of oxygen in the drying chamber, this latter being the main cause of losses due to
189 ascorbic acid browning reactions. Similar results were reported by Vanamala et al. (2005)
190 and Moraga et al (2012), which found that freeze-drying did not reduce significantly
191 vitamin C content in different varieties of grapefruits. As it is shown in **Table 2**, the
192 retention of this vitamin in relation to the non-formulated fruit was higher in the FD (97-
193 100%) than in the SD samples (92-94 %). Although spray-drying process caused a
194 significant ($p<0.05$) decrease in the content of ascorbic acid, the retention levels were high.
195 Despite the high temperature used in the process, the drying occurs instantaneously, so that
196 the sample does not stay in contact for a long time with the high temperature, which can
197 guarantee the preservation of sensitive compounds (Agudelo et al., 2016). Moreover a
198 slightly greater protective effect was observed when arabic gum and bamboo fiber were
199 added together (SD_1), with 94% of retention for 92% in the sample containing only AG
200 (SD_2). The degradation of vitamin C by effect of the high temperature applied during spray-
201 drying was also found by Langrish (2009) and Solval, Sundararajan, Alfaro & Sathivel
202 (2012), whereas the protective effects of AG addition were reported by Ali, Maqbool,
203 Ramachandran & Alderson (2010), among others.
204 As for α-tocopherol, the levels were maintained in spray-dried samples (SD) compared to
205 LG sample, whereas a significant loss ($p < 0.05$) was observed in GG in relation to the
206 formulated freeze-dried samples (FD). This may be explained by the protection afforded by
207 the shell materials added. Arabic gum (AG) is acknowledged to be an effective
208 encapsulation agent due to its high water solubility, the low viscosity of its concentrated
209 solutions relative to other hydrocolloid gums, and its ability to act as oil in water-emulsifier

(Glicksman, 1983), which may explain the good retention of α-tocopherol observed in the dried preparations. Bamboo fiber (BF) has not been used with this purpose in the literature, although the properties reported by the commercial company for the product (Vitacel®), as a solute with synergistic effects with proteins, capillary effects (water and oil-binding) and binding characteristics independent of the temperature or the pH value, and no quality changes in extreme processing conditions, would also explain the efficiency in α-tocopherol preservation.

The phenolic chromatographic profile of *Citrus paradisi var.* Star Ruby (grapefruit) recorded at 280 nm is shown in **Figure 1**. Compound characteristics, tentative identities and quantitative results are presented in **Tables 3** and **4**. Compounds were identified based on their chromatographic and UV and mass spectra characteristics. Up to eighteen compounds were detected, four of which were phenolic acid derivatives and fourteen flavonoids, mainly from the group of flavanones (**Table 3**). Most of these compounds have been previously reported by other authors in grapefruit or different Citrus species (Dugo, Presti, Öhman, Fazio, Dugo & Mondello, 2005; Peterson et al., 2006; Gattuso, Barreca, Gargiulli, Leuzzi & Caristi, 2007; Mullen, Marks & Crozier, 2007; Djoukeng, Arbona, Argamasilla & Gomez-Cadenas, 2008; Xu et al., 2008; Kelebek, 2010; Igual, García-Martínez, Camacho & Martínez-Navarrete, 2011; Zhang, Duan, Zang, Huang & Liu, 2011; Abad-García, Garmón-Lobato, Berueta, Gallo & Vicente, 2012a, Abad-García, Berueta, Garmón-Lobato, Urkaregi, Gallo & Vicente, 2012b; Anagnostopoulou & Kefalas, 2012; Goulas & Manganaris, 2012; Moraga et al., 2012; Barreca et al., 2013; Sun, Qiao, Shen, Jiang, Chen & Ye, 2013; García-Castello, Rodriguez-Lopez, Mayor, Ballesteros, Conidi & Cassano, 2015). Nonetheless, to the best of our knowledge, compounds 1, 3 and 9 have not been previously described in grapefruit. Compound 1 ($[M-H]^-$ at *m/z* 329) and 3 ($[M-H]^-$ at

m/z 325) releasing MS^2 fragments at *m/z* 167 (-162 u; [3,4-dihydroxyphenylacetic acid-H]$^-$) and *m/z* 179 (-146 u; [caffeic acid-H]$^-$), respectively, were tentatively assigned as 3,4-dihydroxyphenylacetic acid hexoside and caffeic acid rhamnoside. Compound 9 ([M-H]$^-$ at *m/z* 563) presented a UV spectrum characteristic of a flavone and a fragmentation pattern that was coherent with an *O*,*C*-diglycoside of apigenin bearing pentosyl and hexosyl residues. The loss of -120 u leading to the ion at *m/z* 443 supported the presence of a *C*-attached hexose, while the absence of an ion [(M-H)-90]$^-$ pointed to a 6-*C* attachment. The lack of an ion [(M-H)-132]$^-$ from the loss of the pentosyl residue suggested that this sugar was not linked to the aglycone but to the other sugar; this was confirmed by the presence of an abundant [(M-H)-150]$^-$ ion at *m/z* 413, which according to Ferreres, Gil-Izquierdo, Andrade, Valentao & Tomás-Barberán (2007) would be characteristic of an *O*-attached pentose on the *C*-glycosylating hexose. The *O*-glycosylation should not take place in the positions 6'', 4'', or 3'' of the hexose, otherwise the fragment [(M-H)-120]$^-$ would not be produced. The ion at *m/z* 293 would result from the fragment at *m/z* 413 by further loss of a fragment of 120 u (partial loss of the *C*-attached hexose). All in all, compound 9 was tentatively identified as apigenin 2''-*O*-pentosyl-6-*C*-hexoside.

Flavanones were the dominant flavonoids in all grapefruit samples, representing about 93% of total flavonoids (**Table 4**). These results are similar to those compiled by Peterson et al. (2006). Various flavanone neohesperidosides (naringin, neohesperidin, poncirin) and rutinosides (narirutin, hesperidin, eriocitrin, and didymin) were identified in the analyzed grapefruit samples, with naringin and narirutin being the predominant phenolic compounds, as also reported by other authors (Vanamala et al., 2006; Gattuso et al., 2007; Moraga et al., 2012). Naringin is a characteristic component of grapefruit juices and the principal responsible for the bitter taste of this fruit (Mullen et al., 2007). Its mean concentration

ranged between 560 and 680 mg/100g dw in the two samples without carriers added (GG and GL), values similar to those reported by Moraga et al. (2012).

In general, the freeze-drying of formulated samples did not cause important changes in the phenolic composition, with percentages of retention of 90-95% in the content of total phenolics in relation to the GG sample when expressed in relation to grapefruit own solutes (**Table 4**), whatever the type of shell material added. The lower relative retention observed in the sample FD_1 might be a consequence of the rehydration it was submitted before freeze-drying. Much greater losses of phenolic compounds were produced by the spray drying process, with mean percentages of retention around 58% in the content of total phenolics with respect to the starting material (LG). This might be explained by an increased degradation favoured by the applied temperature.

3.2 Effects on antioxidant activity

In order to evaluate the effects of freeze-drying and spray-drying on the antioxidant activity, four chemical and biochemical *in vitro* assays were performed (**Table 5**). The antioxidant activity was expressed as EC_{50} values (mean ± SD). In general, LG and GG samples showed greater antioxidant capacity (lower EC_{50} values) than dried samples, being the LG extract the most active in all assays, consistent with its higher levels of vitamins and phenolic compounds. Relatively good retention of the antioxidant capacity was found in the formulated freeze-dried powders in relation to the non-formulated material, but in the case of the β-carotene bleaching assay, where a sharp decrease of the activity was observed in most of the processed samples. On the contrary, the spray-dried samples showed the lowest antioxidant activity, which is coherent with a greater loss was produced in their levels of

281 phenolic compounds. Pearson's statistical correlation analysis was used to establish
282 correlations between the antioxidant capacity and the studied bioactive compounds. The
283 obtained results showed that the most significant contribution to DPPH scavenging activity
284 (-0.82, $p<0.05$) and inhibition of β-carotene blanching (-0.76, $p<0.05$), and was provided by
285 total phenolic compounds specifically by flavonoids. However, these compounds did not
286 present significant correlations with the reducing power (-0.43, $p>0.05$) and TBARS
287 formation inhibition (-0.32, $p>0.05$)
288 The antioxidant activity of flavonoids as electron or hydrogen donors relates to the
289 reduction potentials and reactivity of the substituent reactive groups, so in DPPH
290 scavenging activity the compounds, didymin (-0.91, $p<0.05$), naringin (-0.8405, $p<0.05$),
291 narirutin (-0.81, $p<0.05$), poncirin (0.81, $p<0.05$) and hesperidin (-0.73, $p<0.05$) presented
292 the best correlations, while in the inhibition of β-carotene bleaching, melitidin (-0.94,
293 $p<0.05$), nehosperidin (-0.90, $p<0.05$), and apigenin 2''-*O*-pentosyl-6-*C*-hexoside (- 0.84,
294 $p<0.05$) were the most promising compounds.
295 There are many studies in the literature that also described a high correlation between
296 phenolic compounds content and antioxidant capacity of many fruits (Deepa, Kaur, George,
297 Singh & Kapoor, 2007; Contreras-Calderón, Calderón-Jaimes, Guerra-Hernández &
298 García-Villanova, 2011), attributing this behaviour to the redox properties of these
299 compounds, which allow them to act as reducing agents, hydrogen donors and singlet
300 oxygen quenchers (Miranda et al., 2010). In extracts from Rio Red grapefruit,
301 Jayaprakasha, Girennavar & Patil (2008) also reported a high correlation ($R^2>0.94$)
302 between total polyphenol content and radical scavenging activity by the DPPH method.

303 Ascorbic acid (-0.7890, $p<0.05$) and α-tocopherol (-0.54, $p<0.05$) contributed to increase
304 the reducing power, in addition to some individual phenolic compounds suggesting that all
305 these compounds can work synergistically in the protection against oxidative damages.
306

307 **4. Conclusions**

308 The results obtained in the present study showed that adding arabic gum and bamboo fiber
309 to obtain grapefruit powder by freeze-drying is a good alternative, maintaining the
310 functional components of the fruit, namely antioxidant vitamins and phenolic compounds,
311 and antioxidant properties. However, in the case of spray-drying it lead to a loss of
312 bioactive compounds affecting the functional quality of the fruit. In both cases, the addition
313 of arabic gum helps protect especially the α-tocopherol against degradation by acting as
314 encapsulation agents. Bamboo fiber added together with the gum showed a protective effect
315 against ascorbic acid and total phenols degradation. Clearly the largest contribution to the
316 antioxidant capacity of the studied samples is provided by the presence of phenolic
317 compounds, mainly flavonoids that can effectively scavenge various reactive oxygen
318 species or free radicals under *in vitro* conditions.
319

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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).

		Type of shell material and their content (g/100g GG or GL)	
	Formulation	Arabic Gum (AG)	Bamboo Fiber (FB)
Freeze dried grapefruit			
1	GG	-	-
2	LG	-	-
3	FD_{1*}	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

* prior to freeze drying the mixture was hydrated to a level of 90 $g_{water}/100g_{product}$

Table 2. Contents of antioxidant vitamins referred to grapefruit's own solutes (GS). The results are presented as mean ± SD

	α-Tocopherol mg/100 g GS	Ascorbic acid g/100 g GS
Freeze dried grapefruit powder		
GG	0.60 ± 0.02^{e}	0.333 ± 0.003^{d}
LG	0.66 ± 0.03^{cd}	0.381 ± 0.003^{a}
FD_1	0.96 ± 0.03^{a}	0.3331 ± 0.0002^{d}
FD_2	0.95 ± 0.03^{ab}	0.32592 ± 0.00003^{e}
FD_3	0.93 ± 0.02^{ab}	0.33263 ± 0.00003^{d}
FD_4	0.84 ± 0.04^{bc}	0.3240 ± 0.0007^{e}
Spray dried grapefruit powder		
SD_1	0.677 ± 0.009^{cd}	0.3584 ± 0.0003^{b}
SD_2	0.73 ± 0.04^{cd}	0.351 ± 0.002^{c}

Different letters within the same column indicate significant differences ($p<0.05$)

Table 3. Retention time (Rt), wavelengths of maximum absorption in the visible region ($_{max}$), mass spectral data and tentative identification of phenolic compounds in grapefruit samples.

Peak	Rt (min)	λ_{max} (nm)	Pseudomolecular ion $[M-H]^-$ (*m/z*)	MS^2 (*m/z*)	Tentative identification	References*
1	5.5	278	329	167(100)	3,4-Dihydroxyphenylacetic acid hexoside	-
2	5.8	332	341	179(29),161(100),135(18)	Caffeic acid hexoside	1,10
3	8.3	328	325	179(100)	Caffeic acid rhamnoside	-
4	9.2	284,336	595	287 (100)	Eriodictyol-7-*O*-rutinoside (eriocitrin)	1, 2,3,7,8,11
5	9.9	332	355	193(100)	Ferulic acid hexoside	1, 2
6	11.6	336	593	505(14),473(24),383(18),353(29),325(11)	Apigenin 6,8-*C*-diglucoside	1, 2,7,8,10
7	12.4	286,336	741	433(47),271(100)	Naringenin-7-*O*-rutinoside-4'-*O*-glucoside	1, 2,6,10
8	13.8	286,334	741	433(14),271(5)	Naringenin-7-*O*-neohesperidoside-4'-*O*-glucoside	1, 2,10
9	18.3	336	563	443(30),413(100),341(28),313(15),293(48)	Apigenin 2''-*O*-pentosyl-6-*C*-hexoside	-
10	18.5	284,336	595	271(100)	Naringenin-*O*-dihexoside	2,10
11	20.7	282,336	579	271 (100)	Naringenin-7-*O*-rutinoside (narirutin)	1,3,4,5,7,8,9,10,11,12,13,14,15,16
12	22.1	284,336	579	459(22),313(3),295(3),271(20)	Naringenin-7-*O*-neohesperidoside (naringin)	1,3,4,5,7,8,9,10,11,12,13,14,15,16
13	23.4	284,338	609	301(100)	Hesperetin-7-*O*-rutinoside (hesperidin)	1,3,4,5,8,9,10,12,14.15
14	24.8	284,338	609	301(100)	Hesperetin-7-*O*-neohesperidoside (neohesperidin)	1,3,4,8,9,10,12,14,16
15	27.6	284,332	621	579(5), 501(14),459(7), 271(12)	Acetyl naringin	11
16	27.9	284,336	723	661(13), 621(44),579(100), 271(18)	3-Hydroxymethylglutaryl naringin (Melitidin)	11
17	31.4	286,324	593	285(100)	Isosakuranetin-7-*O*-rutinoside (didymin)	1, 2,3,5,6,8,10,15,16
18	32.4	286,338	593	285(100)	Isosakuranetin-7-*O*- neohesperidoside (poncirin)	1, 2,3,5,7,8,10,15,16

*References: 1-Mullen et al. (2007); 2- Abad-García et al. (2012a) ; 3- Gattuso et al. (2007) ; 4- García-Castello et al. (2015) ; 5- Goulas & Manganaris (2012) ; 6- Barreca et al. (2013) ; 7- Djoukeng et al. (2008); 8 – Dugo et al. (2005) ; 9- Kelebek (2010) ; 10- Abad-García et al. (2012b); 11 -Zhang et al. (2011); 12- Xu et al. (2008) ; 13- Anagnostopoulou & Kefalas (2011); 14- Sun et al. (2013); 15-Igual et al. (2011); 16- Moraga et al. (2012).

Table 4. Contents of phenolic compounds in freeze-dried grapefruit (GG and LG) and formulated samples (FD and SD) referred to grapefruit's own solutes (mg/100 g GS). The results are presented as mean ± SD

Peak	GG	LG	FD_1	FD_2	FD_3	FD_4	SD_1	SD_2
1	6.9 ± 0.5 ^{a}	1.71 ± 0.02 ^{f}	2.35 ± 0.03 ^{d}	1.19 ± 0.03 ^{g}	3.44 ± 0.08 ^{c}	2.31 ± 0.01 ^{d}	4.9 ± 0.2 ^{b}	1.01 ± 0.04 ^{g}
2	tr	tr	tr	tr	tr	tr	tr	tr
3	tr	tr	tr	tr	tr	tr	tr	tr
4	10.2 ± 0.4 ^{a}	10.4 ± 0.3 ^{a}	8.46 ± 0.09 ^{b}	7.2 ± 0.3 ^{c}	8.4 ± 0.2 ^{b}	8.3 ± 0.6 ^{b}	5.8 ± 0.3 ^{d}	6.70 ± 0.18 ^{c}
5	14.6 ± 0.5 ^{a}	8.7 ± 0.2 ^{b}	7.89 ± 0.18 ^{c}	6.7 ± 0.1 ^{e}	7.32 ± 0.06 ^{d}	5.93 ± 0.12 ^{f}	6.8 ± 0.3 ^{de}	5.58 ±0.03 ^{f}
6	38 ± 2 ^{a}	38.1 ± 1.2 ^{a}	26.6 ± 0.2 ^{b}	25.5 ± 0.7 ^{b}	25.9 ± 0.3 ^{b}	28 ± 2 ^{b}	22.3 ± 0.7 ^{c}	25.6 ± 1.6 ^{b}
7	10.8 ± 0.5 ^{a}	9.3 ± 0.5 ^{b}	9.98 ± 0.13 ^{ab}	9.7 ± 0.4 ^{b}	9.53 ± 0.03 ^{b}	9.4 ± 0.5 ^{b}	6.1 ± 0.2 ^{c}	5.4 ± 0.2 ^{c}
8	8.7 ± 0.3 ^{a}	7.9 ± 0.3 ^{b}	6.96 ± 0.05 ^{c}	7.8 ± 0.2 ^{b}	7.9 ± 0.3 ^{b}	7.7 ± 0.3 ^{b}	4.92 ± 0.07 ^{d}	4.41 ± 0.09 ^{e}
9	9.2 ± 0.3 ^{a}	8.5 ± 0.5 ^{a}	3.61 ± 0.14 ^{c}	5.55 ± 0.05 ^{b}	6.1 ± 0.7 ^{b}	5.6 ±0.4 ^{b}	2.58 ± 0.05 ^{d}	2.91 ± 0.12 ^{cd}
10	tr	0.68 ± 0.02	tr	tr	tr	tr	tr	tr
11	129.9± 1.3 ^{b}	142.4 ± 0.3 ^{a}	113.38 ± 0.06 ^{e}	118.9 ± 1.2 ^{c}	117.2 ± 0.8 ^{d}	120.18 ± 0.09 ^{c}	75.7 ± 0.4 ^{f}	73.69 ± 0.07 ^{g}
12	560 ± 2 ^{b}	680.9 ± 1.8 ^{a}	525 ± 4 ^{e}	545 ± 5 ^{c}	532 ± 4 ^{d}	548.7 ± 1.1 ^{c}	331 ± 3 ^{f}	322.9 ± 0.6 ^{g}
13	8.2 ± 0.5 ^{a}	8.17 ± 0.08 ^{a}	6.52 ± 0.02 ^{d}	7.4 ± 0.3 ^{bc}	6.92 ± 0.06 ^{cd}	7.57 ± 0.12 ^{ab}	5.5 ± 0.3 ^{e}	4.9 ± 0.3 ^{f}
14	10.2 ± 0.3 ^{b}	13.13 ± 0.13 ^{a}	8.16 ± 0.13 ^{c}	8.7 ± 0.2 ^{c}	8.48 ±0.08 ^{c}	8.8 ± 0.6 ^{c}	6.64 ± 0.04 ^{d}	6.1 ± 0.2 ^{e}
15	8.4 ± 0.3 ^{c}	8.52 ± 0.02 ^{c}	8.24 ± 0.08 ^{c}	10.2 ± 0.4 ^{a}	9.8 ± 0.4 ^{ab}	9.6 ± 0.2 ^{b}	5.2 ± 0.2 ^{d}	4.14 ± 0.03 ^{e}
16	3.67 ± 0.09 ^{b}	4.19 ± 0.07 ^{a}	1.93 ± 0.13 ^{e}	3.02± 0.12 ^{c}	2.59 ± 0.08 ^{d}	2.38 ± 0.15 ^{d}	2.11 ± 0.12 ^{e}	0.79 ± 0.07 ^{f}
17	12.7 ± 0.4 ^{d}	16.9 ± 0.4 ^{b}	14.03 ± 0.09 ^{c}	18.6 ± 0.5 ^{a}	17.22 ±0.12 ^{b}	16.7 ± 0.2 ^{b}	6.9 + ±0.5 ^{e}	6.79 ± 0.13 ^{e}
18	45.2 ± 2.4 ^{a}	44.0 ± 1.2 ^{ab}	49.5 ± 0.6 ^{b}	44 ± 2 ^{ab}	43.3 ± 1.4 ^{ab}	43.9 ± 0.8 ^{ab}	27.9 ± 1.6 ^{c}	27.6 ± 1.5 ^{c}
Total phenolic acids	14.7 ± 0.3 ^{a}	10.4 ± 0.2 ^{cd}	10.2 ± 0.2 ^{d}	7.87 ± 0.09 ^{e}	10.76 ± 0.02 ^{c}	8.24 ± 0.12 ^{e}	11.75 ±0.03 ^{b}	6.59 ± 0.02 ^{f}
Total flavonoids	856 ± 6 ^{b}	993.4 ± 0.9 ^{a}	773 ± 4 ^{e}	812 ± 9 ^{c}	796 ± 8 ^{d}	818 ± 2 ^{c}	502 ± 6 ^{f}	492 ± 4 ^{f}
Total phenolic compounds	871 ± 7 ^{b}	1003.8 ± 0.6 ^{a}	784 ± 4 ^{e}	820 ± 9 ^{cd}	806 ± 8 ^{d}	826 ±2 ^{c}	514 ± 6 ^{f}	499 ± 4 ^{g}

tr: Traces. Different letters within the same row indicate significant differences (p<0.05)

Table 5. *In vitro* antioxidant activity presented as EC_{50} values (mg/mL) obtained for the different studied samples. The results are presented as mean ± SD

		Reducing Power	Lipid peroxidation inhibition	
	DPPH scavenging activity	Ferricyanide/Prussian blue assay	β-Carotene bleaching inhibition	TBARS formation Inhibition
Freeze dried grapefruit powder				
GG	7.11 ± 0.13 [c]	2.26 ± 0.03 [d]	9.1 ± 0.3 [e]	2.75 ± 0.15 [d]
LG	5.61 ± 0.07 [f]	1.67 ± 0.01 [e]	2.7 ± 0.4 [f]	1.65 ± 0.03 [g]
FD_1	7.21 ± 0.11 [c]	3.1 ± 0.3 [a]	16.1 ± 0.4 [b]	1.86 ± 0.06 [f]
FD_2	6.1 ± 0.2 [e]	2.44 ± 0.04 [c]	9.5 ± 0.4 [e]	2.04 ± 0.03 [e]
FD_3	6.4 ± 0.3 [d]	2.72 ± 0.02 [b]	14.1 ± 0.5 [d]	3.0 ± 0.3 [c]
FD_4	6.3 ± 0.5 [de]	2.51 ± 0.07 [c]	14.6 ± 0.3 [c]	4.0 ± 0.2 [b]
Spray dried grapefruit powder				
SD_1	8.61 ± 0.10 [a]	2.73 ± 0.01 [b]	19.2 ± 0.7 [a]	2.13 ± 0.04 [e]
SD_2	7.6 ± 0.3 [b]	2.50 ± 0.07 [c]	19.0 ± 0.7 [a]	4.17 ± 0.06 [a]

Different letters within the same column indicate significant differences ($p<0.05$)

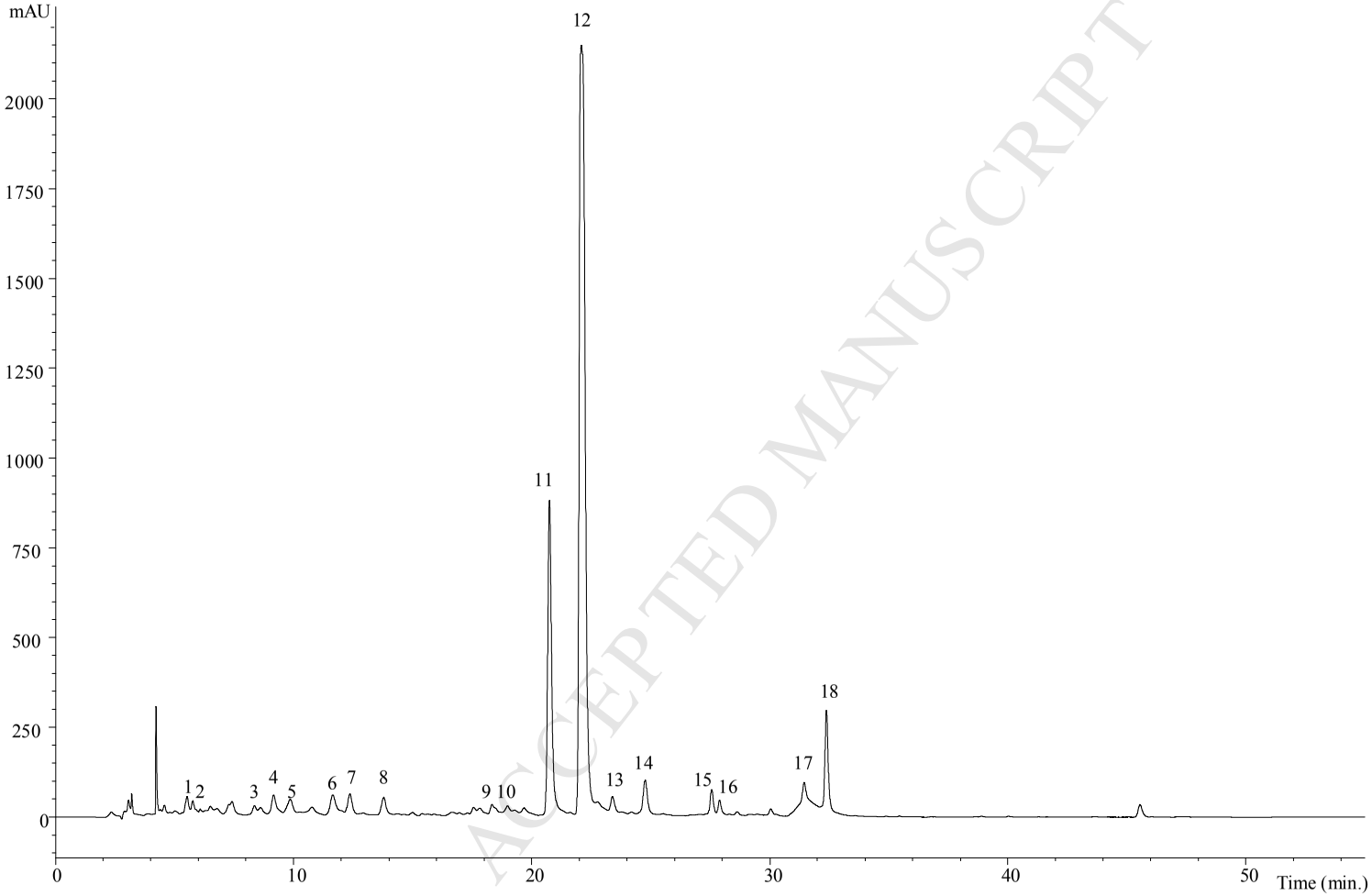

Figure 1. HPLC profile of phenolic compounds in the freeze-dried liquidized grapefruit sample (LG), recorded at 280 nm.

Highlights

- Freeze drying showed better retention of bioactive compounds than spray drying

- Arabic gum and bamboo fiber protect bioactive compounds against degradation

- The most abundant grapefruit flavonoids are flavanones

- Total phenolic compounds showed a high correlation with antioxidant activity