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Influence of osmotic dehydration pre-treatment and combined drying method on physico-chemical and sensory properties of pomegranate arils, cultivar *Mollar de Elche*

Marina Cano-Lamadrid [a], Krzysztof Lech [b], Anna Michalska [d], Malwina Wasilewska [b], Adam Figiel [b], Aneta Wojdyło [c], Ángel A. Carbonell-Barrachina [a,*]

[a] Universidad Miguel Hernández de Elche (UMH), Escuela Politécnica Superior de Orihuela, Department of Agro-Food Technology, Ctra. Beniel, km 3.2, 03312 Orihuela, Alicante, Spain
[b] Institute of Agricultural Engineering, Wrocław University of Environmental and Life Sciences, 37a Chełmońskiego Street, 51-630 Wrocław, Poland
[c] Department of Fruit, Vegetable and Plant Nutraceutical Technology, Wrocław University of Environmental and Life Sciences, 37 Chełmońskiego Street, 51-630 Wrocław, Poland
[d] Institute of Animal Reproduction and Food Research of the Polish Academy of Sciences, Division of Food Science, Olsztyn, Poland

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A B S T R A C T

"*Mollar de Elche*" is the most popular Spanish pomegranate cultivar (intense sweetness and easy-to-chew arils); however, arils have pale pink colour and flat sensory profile. "*Mollar the Elche*" arils first underwent an osmotic dehydration pre-treatment (OD) with concentrated juices: (i) chokeberry, (ii) apple, and/or (iii) pomegranate cultivar "*Wonderful*", to improve their antioxidant capacity, colour, and sensory profile complexity, and later the arils were dried by a combined method (convective pre-drying + vacuum microwave finish drying). The use of OD provided dried arils with characteristic sweetness, and improved colour and aromatic complexity. The recommended OD methods were those using (i) pomegranate, and (ii) pomegranate with chokeberry juices; they improved the total anthocyanin content (mean of 368 mg kg^{-1}), red colour (a^* coordinate 15.6), and antioxidant capacity (e.g. ABTS mean of 5.7 mmol Trolox 100 g^{-1}). However, further research is still needed because freeze-dried arils had the highest anthocyanin content.

1. Introduction

Epidemiological studies concluded that high consumption of fruits and vegetables reduces the risk of chronic diseases (EUFIC, 2012). Among fruits, pomegranate (*Punica granatum* L.) and pomegranate-based products have been specifically associated with inhibition of prostate, breast, and lung cancer (Orgil, Spector, Holland, Mahajna, & Amir, 2016), reduction of dyslipidaemia, and cardiovascular issues (Haghighian et al., 2016), antioxidant stress effect (Orgil et al., 2016), and anti-diabetic properties. Pomegranate owns its health-related properties to the unique composition of biologically active components, mainly polyphenols from the fruit peel (exterior rind) (Calín-Sánchez et al., 2015).

Pomegranates are usually available on the market as fresh fruits or as beverages, basically juices, concentrates or wine (Jaiswal, DerMarderosian, & Porter, 2010). In smaller amounts, they are available as an additive to jams, jellies, and are used for candy production (Tezcan, Gültekin-Özgüven, Diken, Özçelik, & Erim, 2009). To prolong the arils shelf-life, different drying processes have been applied; however, they had a significant impact on the final products quality (Kingsly & Singh, 2007). Dried pomegranate arils are a great source of vitamins and minerals and are rich in biologically active components (Alaei & Amiri Chayjan, 2015). The main purpose for production of dried pomegranate arils is consumption of the arils as a nibbling snack.

Among Spanish pomegranates, the most popular cultivar is "*Mollar de Elche*", which production is safeguarded by a Protected Designation of Origin (DOP) since 2016 [R (UE) 2016/83]. This cultivar is recognized worldwide, due to its high sweetness intensity and arils with soft woody portion. It has also disadvantages, such as pale pink colour that significantly decreases the quality of pomegranate-based products, especially the juice after the heat treatment. Besides, its sensory profile is too flat with a predominating sweetness and with very weak fruity notes (Vázquez-Araújo et al., 2014).

There are agronomic practices that can be used to improve the flavour of some fruits. Regulated deficit irrigation led to pomegranate fruits, cv. "*Mollar de Elche*" with a more complex sensory pro-

* Corresponding author.
E-mail address: angel.carbonell@umh.es (Á.A. Carbonell-Barrachina).

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file and enriched chemical composition (high punicalagin content); these fruits are called "*hydroSOStainable*" (Galindo et al., 2014).

The appearance of commercial dried arils is mostly not acceptable for consumers, due to intense browning. A previous study (Calín-Sánchez et al., 2013) showed that high quality dried arils could be prepared using appropriate drying methods. It was indicated that the application of fruit or berry juices (e.g. chokeberry) during the osmotic dehydration (OD) step could improve the functionality, and colour of the dried products, by transferring bioactive and organoleptic-active compounds from the osmotic solution into the dehydrated pomegranate arils (Calín-Sánchez et al., 2013).

Considering all the above, the aim of this study was to evaluate the physico-chemical and sensory properties of the dried arils prepared using first osmotic dehydration (OD), with selected fruit juice concentrates, and later a combined drying technique [convective pre-drying (CPD) and vacuum-microwave finish drying (VMFD)] for dehydration of pomegranate arils cultivar "*Mollar de Elche*". The drying kinetics, quality parameters (anthocyanin content, antioxidant capacity, colour, rehydration ratio), and descriptive sensory profile were studied.

2. Materials and methods

2.1. Material

Pomegranates [cultivars "*Mollar de Elche*" (used for preparation of dried arils) and "*Wonderful*" (used for preparation of osmotic solution)] were cultivated in a farm located in Murcia (Spain) under regulated deficit irrigation (RDI) (Galindo et al., 2014). Pomegranate fruits (~100 kg) were hand-harvested in mid-September 2015 at a commercial maturity stage (~15 °Brix), and immediately posted to Poland. The pomegranate fruits were submitted to short term storage (less than 2 weeks) at 5 °C at an approximate relative humidity (RH) of 90%, which are the optimal storage conditions for pomegranates (Elyatem & Kader, 1984).

Each fruit from *Punica granatum* L. "*Mollar de Elche*" was cut at the equatorial zone, and arils were manually separated. Arils were immediately used after its preparation. The main physico-chemical parameters of the fresh arils were: pH 7.8, titratable acidity (TA) 2.9 g L^{-1}, total soluble solids (TSS) 15.2 °Brix, maturity index [TSS(°Brix)/TA (g *per* 100 mL)] 53.4, and moisture content 81.5%.

2.2. Drying processes

Freeze drying (FD) was carried out in freeze dryer OE-950 (Hungary) at a reduced pressure of 65 Pa for 24 h. The temperature within the drying chamber was −60 °C, while the heating plate was at ~30 °C. The freeze-dried arils, without osmotic treatment, were considered as the control sample. Freeze-drying conditions for pomegranate arils were previously optimized by Calín-Sánchez et al. (2013).

The process of combined drying (CPD-VMFD) consisted of convective pre-drying (CPD) at temperature 60 °C for 2 h with an air velocity of 0.6 m s^{-1} performed in the drier designed and built at the Institute of Agriculture Engineering (Wrocław, Poland), and followed by vacuum microwave finish drying (VMFD) with microwave power reduced from the initial 360 W–120 W to avoid overheating of the dried material. A similar combined procedure, CPD-VMFD, was also used during chokeberry drying (Calín-Sánchez et al., 2015).

The VMFD process of the CPD dried samples was performed in a SM 200 dryer (Plazmatronika, Wrocław, Poland) connected to a vacuum system consisting of a vacuum pump BL 30 P (Tepro, Koszalin, Poland), a vacuum gauge MP 211 (Elvac, Bobolice, Poland), and a compensation reservoir of 0.15 m^3. A control sample of "*Mollar de Elche*" pomegranate arils dried by CPD-VMFD, with no osmotic pre-treatment, was prepared using the above equipment for 240 min at 60 °C.

2.2.1. Osmotic dehydration pre-treatment

Fresh "*Mollar de Elche*" arils were osmotically dehydrated at 45 °C for 90 min in different combinations and ratios of concentrated juices (40°Brix):

1. OD(POM): 100% pomegranate *Punica granatum* L., cv. "*Wonderful*";
2. OD(POM + Ch): 50% pomegranate "*Wonderful*" and 50% chokeberry (*Aronia melanocarpa* L.);
3. OD(POM + A): 50% pomegranate "*Wonderful*" and 50% apple (*Pyrus malus* L.);
4. OD(A + Ch): 50% apple and 50% chokeberry; and
5. OD(AP + Ch): 75% apple and 25% chokeberry.

Chokeberry and apple concentrated juices (65 and 67 °Brix, and 22.2 and 5.0 g citric acid kg^{-1}, respectively) were commercial products (Rauch Polska Sp z o.o., Płońsk, Poland). However, pomegranate concentrate was prepared under laboratory conditions by pressing pomegranate fruits, cv. "*Wonderful*" cut in halves, using a domestic citrus juicer (Braun, model CJ 3053, Barcelona, Spain) followed an evaporation step conducted in a vacuum evaporator (Rotavapor R 151, Donserv, Warsaw, Poland). The main quality parameters of this juice were: pH 3.5, total acidity 16.9 g L^{-1}, total soluble solids 16.8 °Brix, and maturity index 10.1. Juices, after concentration to 40 °Brix, were immediately frozen until use. There were three main reasons for selecting these three fruit juices as osmotic solutions: (i) the three juices will improve the sensory quality of the dried pomegranate arils, and especially their appearance; (ii) chokeberry and apple juices are very abundant in Poland, and new applications must be sought; and, (iii) the pomegranate juice, cultivar "*Wonderful*" will led to a final product which is 100%. The values of dynamic viscosity for the osmotic solutions 1–5, determined using viscometer SV-10 (A&D Co., Tokyo, Japan), were 5.13 ± 0.10, 4.13 ± 0.15, 4.76 ± 0.13, 4.20 ± 0.1 and 4.39 ± 0.12 mPa, respectively.

The ratio of osmotic solution to pomegranate arils was maintained at 200 mL to 100 g of fresh matter and the mixture was manually agitated every 5 min (Lech et al., 2015).

In previous studies using pomegranate arils, cultivar *Kandhari*, Mundada, Singh, and Maske (2010) used slightly different working conditions, 55 °Brix, 40 °C, 100 min, and fruit to solution ratio of 1:4 (w/w). However, the parameters of the osmotic dehydration step (time, temperature, and seed ratio) used in the current experiment were chosen on the basis of the results obtained in previous studies on the dehydration of similar matrices, such as chokeberries (Calín-Sánchez et al., 2015), beetroots (Lech et al., 2015), and sour cherries (Nowicka, Wojdyło, Lech, & Figiel, 2015b). These experimental conditions provided optimum mass exchange, with significant enrichment of the dried products but without significant changes in the chemical composition of the osmotic solutions which will limit their potential reuse in several osmotic steps.

After the OD treatment, the samples were removed from the osmotic solutions using a tea strainer, and were left in the strainer for ~20 min; then, the excess of osmotic solution was gently removed from their outer surface with absorbent paper.

2.3. Modelling of drying kinetics

The pomegranate arils drying kinetics was evaluated on the basis of the mass loss of the arils. During CPD, the samples were weighted every 5 min for the initial 20 min and the time intervals between the mass measurements were successively extended with the drying time. During VMFD samples were weighted at time 3, and every 8 min thereafter. The drying kinetics represents decreasing of moisture ratio *MR* in time of drying. *MR* is defined according to Eq. (1):

$$MR = \frac{M(t)}{M_0} \quad (1)$$

Preliminary tests conducted in this study proved that the best fitting was obtained for the modified Page model (as given by Eq. (2)); consequently, only this model was used in this study: where *A*, *n* and *k* are constants.

$$MR = A \cdot e^{-k \cdot t^n} \quad (2)$$

The good-fitting of a specific model to the experimental data was evaluated using: (i) coefficient of determination (R^2) and (ii) Root Mean Square Error (*RMSE*). The model fit is better, if the value of R^2 is closer to 1.0 and the RMSE value is closer to 0.

2.4. Extraction and LC-PDA/MS analysis of anthocyanins

The pomegranate extract of polyphenols was prepared as described previously (Wojdyło, Nowicka, Carbonell-Barrachina, & Hernández, 2016). Identification and quantification of polyphenols in all samples were carried out using an ACQUITY Ultra Performance LC™ system equipped with PDA (photodiode detector; UPLC™) with binary solvent manager (Waters Corporation, Milford, USA) series coupled with the mass detector G2 QTof Micro mass spectrometer (Waters, Manchester, UK) equipped with an electrospray ionization (ESI) source operating in positive mode. Separations of polyphenols were carried out using an Aquity BEH C18 column (1.7 μm, 2.1 × 100 mm, Waters Corporation, Milford, USA) at 30 °C using conditions previously reported by Wojdyło et al. (2016). The PDA spectra were measured over the wavelength range of 200–600 nm in steps of 2 nm. The runs were monitored at 520 nm for anthocyanins. Retention times and spectra were compared with those of pure standards. Calibration curves at concentrations ranging from 0.05 to 5.00 mg mL^{-1} ($R^2 \leq 0.9998$) were made using cyanidin-3-glucoside, cyanidin-3,5-diglucoside, pelargonidin-3-glucoside from Extrasynthese (Lyon, France). The results were expressed as mg *per* kg dry matter (dm).

2.5. Antioxidant capacity (TEAC ABTS$^+$ and FRAP)

Approximately 1 g of dried pomegranate arils in 10 mL of 30% aqueous methanol (v/v) was sonicated for 15 min. After being kept for 24 h at 4 °C in the dark, the extracts were centrifuged (1500g, 10 min, 4 °C). The antioxidant capacity of the extracts was examined using the Trolox Equivalent Antioxidant Capacity test (TEAC ABTS$^+$) according to Re et al. (1999). The ferric reducing ability was determined by FRAP assay (Benzie & Strain, 1999). Results were presented as mmol Trolox 100 g^{-1} dry weight, dw (±standard deviation).

2.6. Colour measurement

Colour coordinates L^*, a^* and b^* were evaluated using a Minolta Chroma Meter CR-200 Reflectance System (Osaka, Japan), and colour difference (ΔE) was calculated by Eq. (3):

$$\Delta E = [(L - L^*)^2 + (a - a^*)^2 + (b - b^*)^2]^{0.5} \quad (3)$$

The ΔE indicates the degree of total colour change in comparison to the colour of fresh pomegranate arils, cv. "*Mollar de Elche*" (L^* = 33.13, a^* = 18.40, and b^* = 13.12). Low ΔE values represent high similarity to the ideal colour of fresh arils, and a good performance of the drying method (Dak, Sagar, & Jha, 2014). This analysis was conducted in 5 replications.

2.7. Moisture content, water loss and solid gain and rehydration ratio

Moisture content (*MC*), water loss (*WL*) and solid gain (*SG*) were determined according to Nowicka, Wojdylo, Lech, and Figiel (2015a) and Bchir, Besbes, Karoui, Attia, et al. (2012). Rehydration of pre-osmotic dehydrated dried pomegranate arils was performed using 15 mL of distilled water during 1 h at room temperature. The rehydration ratio (RR) was calculated as using Eq. (4).

$$RR = m_1 m_0^{-1} \quad (4)$$

where, m_1 is the mass of the rehydrated arils (g), and m_0 is the mass of the dried arils (g). These analyses were conducted in 5 replications.

2.8. Conductivity

The conductivity of the rehydration liquid (distilled water conductivity = 6 μS cm^{-1}) was measured with the EC-Meter GLP 31 (Crison Instruments S.A., Barcelona, Spain) to evaluate the loss of electrolytes at the end of rehydration step.

2.9. Descriptive sensory evaluation

Eight highly trained panellists, (aged 25–50 years; 4 females and 4 males) from the department of Agro-Food Technology (UMH, Orihuela, Spain) participated in the study (Meilgaard, Civille, & Carr, 2007). The panel was selected and trained following the ISO standard 8586-1 (1993), and it is specialized in descriptive sensory evaluation of fruits and vegetables, including pomegranate products (e.g. Szychowski et al., 2015). For the current study, the panellists received two orientation sessions of 60 min, on fresh and dried pomegranate arils. The following attributes were chosen on the basis of the lexicon by Vázquez-Araújo et al. (2014): (*appearance*) colour and uniformity; (*basic tastes and chemical feelings*) sweetness, sourness, bitterness and astringency; (*flavour*) pomegranate ID, chokeberry ID, apple ID, fruity, caramel, citric, off-flavours, woody and burnt; and, (*texture*) crispiness, adhesiveness, and solubility in saliva. The panel used a numerical scale for quantifying the intensity of the pomegranate products attributes where 0 represents none and 10 extremely strong with 0.5 increments.

Samples (~4 g of dehydrated arils) were served monadically in a randomized order and coded using 3 digit numbers. Unsalted crackers and distillate water were provided to panellists to clean their palates between samples.

2.10. Statistical analysis

All experiments and analyses were run, at least, in triplicate, and data reported are presented as the mean ± standard deviation. All data were subjected to analysis of variance (ANOVA) test and later to Tukey's multiple range test to determine significant differences among treatments at $p < 0.05$. The statistical analyses were done using Statgraphics Plus 5.0 software (Manugistics, Inc., Rockville, MD, USA). Table Curve 2D Windows v. 2.03 enabled mathematical modelling with the best determination coefficient.

3. Results and discussion

3.1. Moisture content (MC), water loss (WL), solid gain (SG), and drying kinetics

The osmotic dehydration in different fruit solutions reduced *MC* of "*Mollar de Elche*" arils from an initial 81.5 ± 0.7% down to an average value of 72.1 ± 0.4% (Table 1). The *WL* was not affected by the nature of the osmotic solution; it reached a mean of 30.2 ± 0.6%, and ranged from 28.9% and 30.7% (Table 1). Similarly to previous studies (Lech et al., 2015), a mean increase of 19.2 ± 1.6% in *SG* was observed after osmotic pre-treatment, with the OD treatment using pomegranate juice cultivar "*Wonderful*" leading to the highest *SG* increase (23.3%). The *SG* reached the lowest value when chokeberry was used in the osmotic solution; this observation was connected with the high particle size of this juice and the accumulation of solids near the surface, causing compaction of the surface layers and increased mass transfer resistance for both water and solids (Bchir, Besbes, Karoui, Paquot, et al., 2012; Kulling & Rawel, 2008). A significant ($p < 0.05$) positive correlation ($R^2 = 0.789$) was observed between dynamic viscosity and *SG*; the higher the viscosity, the higher the adhesive forces maintaining adsorbed solids on the surface and inner capillaries of the pomegranate arils (Juszczak, Witczak, & Galkowska, 2009).

The modified Page model has been already successfully used to describe the drying kinetics of pomegranate arils cultivar *Hicaz* (Horuz & Maskan, 2015; Kingsly & Singh, 2007) and cultivar *Kandhari* (Mundada, Hathan, & Maske, 2011). In the current study, the mean square error (*MSE*) ranged between 4.2×10^{-3} and 7.0×10^{-3} for CPD and 5.0×10^{-4} and 1.9×10^{-3} for VMFD, with the coefficient of determination (R^2) being above 0.9822 (Table 2). These values (high R^2 and low *MSE*) proved the good agreement between the thin layer modelling equation and the experimental data.

The *MR* at the very beginning of CPD was influenced by the composition of the juice used for the osmotic dehydration (Fig. 2a). The constant *A* ranged from 0.556 and 1.00 (Table 2), with lowest *A* values being indicative of high *WL*. Similarly, the VMFD step was affected by the *WL* during CPD and, thus, the moisture content of the samples after CPD. The values of *A* indicated the *MR* of samples dehydrated by combination of OD-CPD at the initial time of the VMFD step (Fig. 2b). The constant *A* and the drying time followed a positive relationship, with lower *A* values leading to shorter drying times due to an increased *WL*. The OD and CPD steps had fixed times, 90 and 240 min, respectively (Table 2). Thus, the final drying time depended basically on the VMFD time. The longest VMFD time was found for arils that were not OD, as they contained higher moisture content to be removed as compared to samples that lost some water during OD. The constant *A* took the lowest value during VMFD (Table 2) when only pomegranate concentrated juice was used, influencing the final time of dehydration and making it the shortest one. In this case, i.e. POM, POM + Ch and POM + A, the final drying time was 2.4 times shorter than in the control CD-VMFD sample (Fig. 2b).

In this way, the composition of the osmotic solution affected the final drying time of samples, and the application of pomegranate juice in OD solutions reduced the final drying time of arils in contrast to apple juice which hindered the process of CD-VMFD. It is worth noting, that enriching pomegranate arils with solids originated from pomegranates increased the drying rate during CD-VMFD by increasing water diffusivity, which on other hand was decreased by gaining of solids from apple concentrate. These trends can be explained in terms of physicochemical mechanisms

Table 1
Moisture content (*MC*, g/100 g fresh weight, fw), water lost (*WL*, g/100 g fw), solid gain (*SG*, g/100 g fw), rehydration ratio (RR), and rehydration solution conductivity (∑) of pomegranate arils before and after osmotic dehydration (OD) in different fruit juices.

Samples[†]	Before OD	After OD			Rehydration	
	MC	*MC*	*WL*[±]	*SG*[±±]	*RR*	∑
	(g *per* 100 g fw)					(μS cm^{-1})
FD	–	–	–	–	1.90 c	263 b
CPD-VMFD	–	–	–	–	3.14 a	200 e
OD(POM)-CPD-VMFD	81.5	71.4	30.2 a	23.3 a	2.95 ab	284 a
OD(POM + Ch)-CPD-VMFD	81.5	73.1	28.9 a	15.3 c	3.00 a	250 c
OD(POM + A)-CPD-VMFD	81.5	71.0	32.1 a	22.2 a	1.07 d	229 de
OD(A + Ch)-CPD-VMFD	81.5	72.4	29.3 a	18.9 b	2.80 b	245 d
OD(AP + Ch)-CPD-VMFD	81.5	72.4	30.7 a	16.4 c	2.85 b	244 d

[†] Mean values followed by the same letter, within the same column, were not significantly different (p < 0.05), according to HSD Tukey's least significant difference test.

Table 2
Values of the parameters *A*, *k*, and *n* of the functions describing drying kinetics of "*Mollar de Elche*" pomegranate arils dried by combined method consisted of convective pre-drying (CPD) and microwave finish vacuum drying (VMFD).

Drying method	Osmotic treatment[†]	Drying kinetics $MR = A \cdot e^{-k \cdot \tau^n}$					Partial time (min)	Total time (min)
		A	*K*	*n*	MSE	R^2		
CPD	None	1.000 a[‡]	0.012 c	0.949 b	0.0092	0.9990	240 a	–
	POM	0.573 c	0.110 b	1.060 a	0.0069	0.9990	240 a	–
	POM + Ch	0.617 b	0.009 c	1.080 a	0.0042	0.9996	240 a	–
	POM + A	0.556 c	0.009 c	1.090 a	0.0045	0.9994	240 a	–
	A + Ch	0.602 b	0.179 a	0.850 c	0.0062	0.9985	240 a	–
	AP + Ch	0.604 b	0.019 c	0.880 c	0.0070	0.9983	240 a	–
VMFD	Without OD	0.124 a	0.107 c	0.640 a	0.0019	0.9963	83 a	323[¶] a
	OD-POM	0.026 d	0.049 d	0.681 a	0.0005	0.9822	35 c	365 c
	OD(POM + Ch)	0.029 d	0.345 a	0.198 d	0.0006	0.9822	35 c	365 c
	OD(POM + A)	0.030 d	0.218 b	0.338 c	0.0006	0.9834	35 c	365 c
	OD(A + Ch)	0.096 b	0.238 b	0.519 b	0.0016	0.9956	51 b	381 b
	OD(AP + Ch)	0.069 c	0.129 c	0.602 ab	0.0012	0.9945	59 b	389 b

[†] POM, Ch, A, and AP stand for pomegranate, chokeberry, apple (at 50% in mixture with 50% chokeberry), and apple (at 75% in mixture with 25% chokeberry), respectively.
[‡] Mean values followed by the same letter, within the same column and drying method, were not significantly different (p < 0.05), according to HSD Tukey's least significant difference test.
[¶] The OD step lasted 90 min for all treatments.

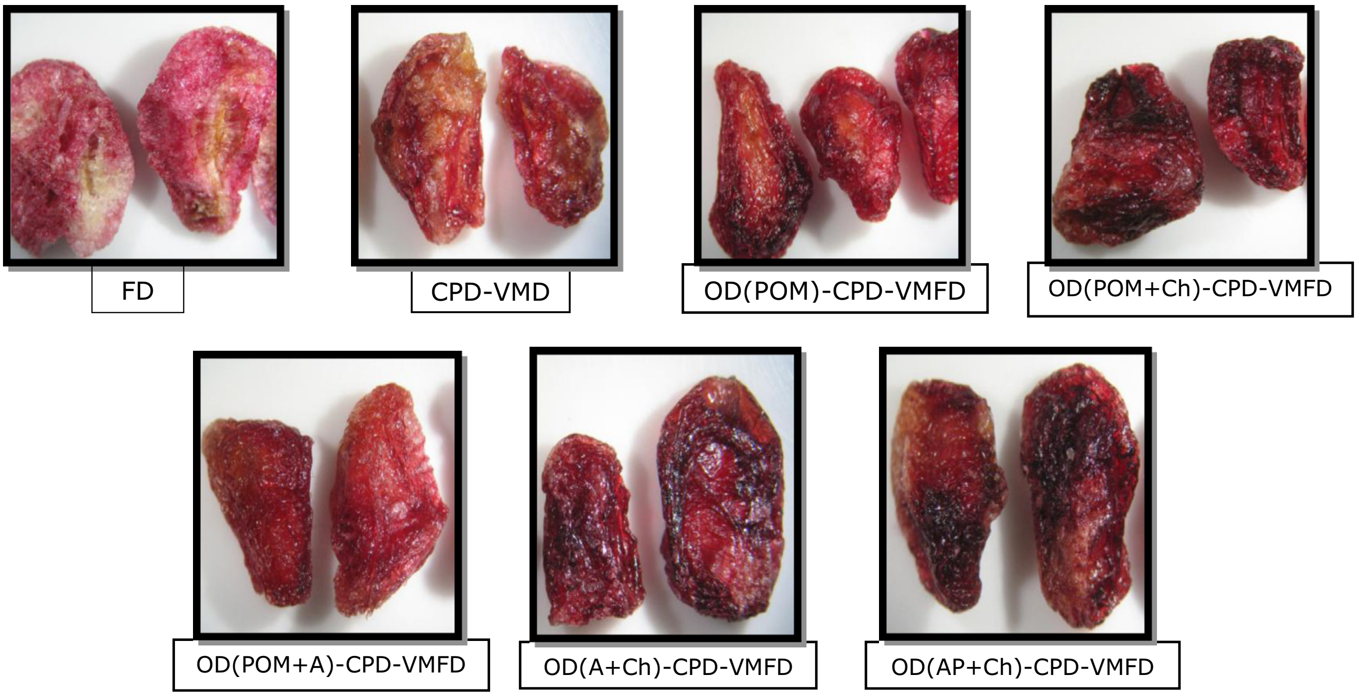


Fig. 1. "*Mollar de Elche*" pomegranate arils dried by freeze drying (FD), combined drying (CPD-VMFD), and osmotic dehydration (OD) before combined drying method (CPD-VMFD).

of water binding and changes in the cellular structure of pomegranate arils caused by solids gain. The sugars present in apple juice hinder water transport to a higher extend by clogging the pores of the dried material (Lech et al., 2015) and increasing mass transfer resistance for water (Bchir, Besbes, Karoui, Paquot, et al., 2012), which contributed to the rise in internal pressure under microwave heating associated with less intensive water evaporation confirmed by the highest maximal temperature during VMFD (Fig. 2c). The cooling effect resulting from intensive water evaporation (Figiel, 2010) decreased the maximal temperature of pomegranate arils (Fig. 2c) which were characterised by the highest moisture content at the very beginning of VMFD (Fig. 2b). The course of temperature of pomegranate arils pre-treated in different osmotic solutions shown in Fig. 2c resulted from the thermal balance between the energy generated by water dipoles inside the microwaved material and energy necessary for water evaporation to the ambient of lower temperature (Figiel, 2010).

Dried pomegranate arils might be consumed as ready-to-eat snacks (Kingsly, Singh, Manikantan, & Jain, 2006) or might be rehydrated before consumption. The *RR* (rehydration ratio) values ranged between 1.07 up to 3.14 (Table 1) and were similar to those previously found in other dried fruits (Megías-Pérez, Gamboa-Santos, Soria, Villamiel, & Montilla, 2014). The *RR* values were significantly affected by drying method and composition of the osmotic solution used for arils pre-treatment. The lowest *RR* (1.07) was found in OD(POM + A) arils, which was also characterised by the highest *WL*. Bchir, Besbes, Karoui, Paquot, et al. (2012) reported that the cells of OD pomegranate arils appeared shrunk and distorted due to solubilisation of polysaccharides that compose the cell walls, the intense WL, and the pre-concentration of sucrose on the surface of the tissue during the OD. Contrary to the previous research (Megías-Pérez et al., 2014), FD products had relatively low *RR* values (1.90) as it might result from the destruction of the outer layer structure of arils during freezing and freeze drying processes. As a result, the porous structure of outer layer allows the water for better incorporation into tissue during the rehydration process.

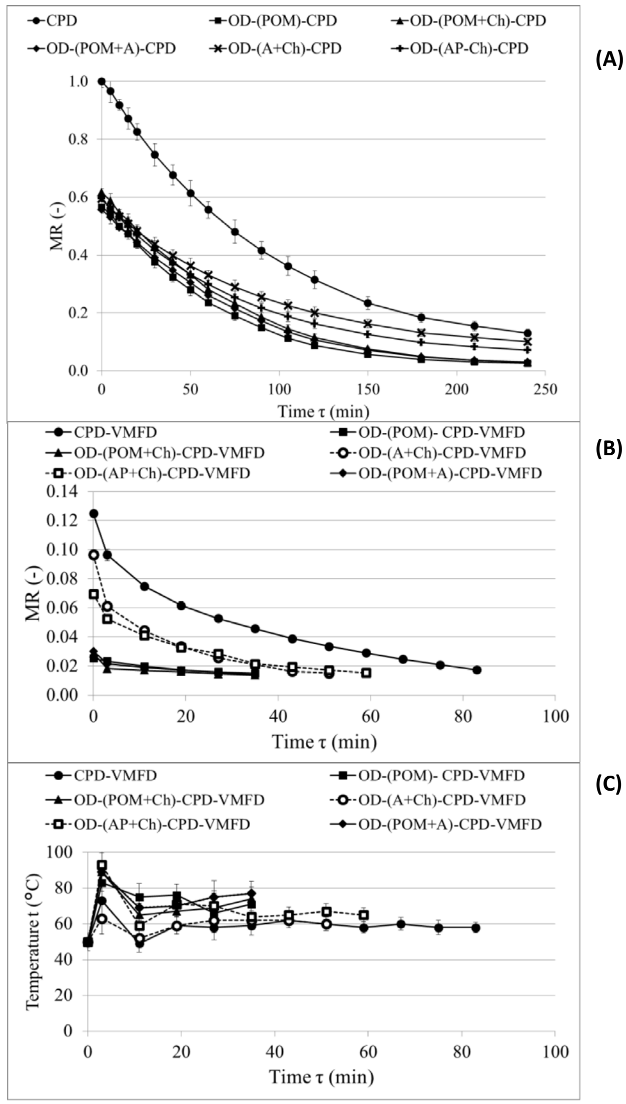
Finally, the electrical conductivity ($\sum$) is a measure of the soluble solids and electrolytes in the medium, which was indicative of the release of intercellular ions from sample tissue, due to an induced damage or creation of a porous structure in the material during drying. The $\sum$ values ranged between 200 ± 1 and 284 ± 2 μS cm^{-1} (FD sample, with porous structure), and were significantly higher than that of the water used for rehydration, 6 μS cm^{-1} (Table 1). The OD pre-treatment increased the $\sum$ values as compared to the CPD-VMFD sample, because the solids retained during the OD were not irreversibly bonded to the aril matrix.

3.2. Anthocyanins

Initially, the anthocyanin profiles of the juices used for the osmotic dehydration consisted of the following compounds:

- Pomegranate juice: A1–A6 with contents being 79.2, 178, 72.2, 283, 75.3, and 5.9 mg 100 mL^{-1} of juice, respectively (total of 694 mg 100 mL^{-1}).
- Chokeberry juice: A4 and A7 with contents being 28.2 and 1032 mg 100 mL^{-1}, respectively.
- Apple juice: A4 and A7 with 10.4 and 76.9 mg 100 mL^{-1}, respectively.

A total of 6 anthocyanins typical of pomegranate products were identified: delpinidin-3,5-diglucoside (A1), cyanidin-3,5-diglucoside (A2), delphinidin-3-glucoside (A3), cyanidin-3-glucoside (A4), pelargonidin-3-glucoside (A5), cyanidin-pentoside (A6), and another 4 anthocyanins not-typical for pomegranate products, basically coming from the chokeberry juice used in the osmotic dehydration step were also found, and are quantified together as A7 (Table 3); the mass spectral characteristics and positive ions in LC–MS QTof of all anthocyanins are summarized in Table 1S



Legend: *POM, Ch, A, and AP stand for pomegranate, chokeberry, apple (at 50% in mixture with 50% chokeberry), and apple (at 75% in mixture with 25% chokeberry), respectively.*

Fig. 2. Drying kinetics of "*Mollar de Elche*" pomegranate arils during the two steps of the combined drying method, CPD-VMFD: convective drying CPD (A); and, vacuum-microwave drying, VMFD (B); and, temperature profile during the VMFD step (C).

(Supplementary material). These 4 chokeberry anthocyanins were identified as cyanidin-3-*O* -galactoside, -glucoside, -arabinoside, and -xyloside. All above mentioned compounds (A1–A5) were previously reported in various pomegranate products (Jaiswal et al., 2010; Mena, Martí, & García-Viguera, 2014; Trigueros, Wojdyło, & Sendra, 2014), except cyanidin-pentoside.

Among all samples analysed, FD led to the highest retention of total anthocyanins (Table 3), followed by CPD-VMFD. In comparison, a greater degradation of those constituents (even down to 61%) was noted when cabinet and sun drying was applied for dehydration of pomegranate arils (Jaiswal et al., 2010). In general, the application of an osmotic dehydration step led to a reduced anthocyanin content due to a migration of these compounds to the osmotic solutions, as previously reported by other researchers (Bchir, Besbes, Karoui, Attia, et al., 2012; Bchir, Besbes, Karoui, Paquot, et al., 2012). Among the OD treatments, the combination of "*Wonderful*" pomegranate and chokeberry juices (50%–50%; Pom + Ch) resulted in the highest content of total anthocyanins (441 mg kg^{-1} dm) in dried arils and pomegranate juice (295 mg kg^{-1}). The high anthocyanin content of the first treatment could be due to high contents of anthocyanins A7 from chokeberry. The use of apple juice with its high sugar content restricted the migration of A7 anthocyanins from the chokeberry juice to the dried arils.

The most abundant anthocyanin in all samples analysed was cyanidin-3,5-diglucoside (A2). On the other hand, chokeberry juice is a great source of polyphenolic compounds, with anthocyanins comprising between 25 and 50% of all polyphenol content (Oszmiański & Lachowicz, 2016). These constituents are natural food colouring agents that may significantly improve the colour of foods; these is the case of the samples OD(POM-Ch)-CPD-VMFD and OD(A-Ch)-CPD-VMFD. Previously, 7 anthocyanins (4 cyanidin glycosides: 3-galactoside, 3-glucoside, 3-arabinoside and 3-xyloside) were identified in chokeberry (Oszmiański & Lachowicz, 2016) among which six of them were

Table 3
Effect of drying conditions on anthocyanin content, antioxidant capacity and the colour of osmotic dehydrated pomegranate arils as affected by the drying method.

Sample†	Anthocyanins‡								Antioxidant capacity		Colour			
	A1	A2	A3	A4	A5	A6	A7	Total A	ABTS+	FRAP	L^*	a^*	b^*	ΔE
	(mg kg−1 dm)								(mmol Trolox 100 g−1)					
FD	113 a¶	219 a	100 a	155 a	58.7 b	0.0 d	0.0 e	646 a	4.7 b	3.4 b	34.5 a	16.3 a	13.0 a	2.5 c
CPD-VMFD	41.8 bc	127.3 b	49.8 b	62.3 cd	77.3 a	0.0 d	0.0 e	358 c	3.6 d	2.7 d	24.3 b	15.2 ab	11.2 a	9.6 b
OD(POM)-CPD-VMFD	48.8 b	102.2 c	49.0 b	73.6 bc	20.3 de	1.2 d	0.0 e	295 c	6.1 a	4.4 a	23.4 b	14.6 ab	9.6 b	13.4 b
OD(POM + Ch)-CPD-VMFD	19.5 de	94.3 cd	37.7 bc	92.0 b	36.3 cd	13.5 a	147 a	441 b	5.3 b	4.0 a	21.5 c	16.6 a	6.8 bc	21.8 a
OD(POM + A)-CPD-VMFD	31.1 cd	79.9 d	30.0 cd	47.0 de	16.0 e	0.0 d	8.5 d	213 d	4.4 bc	3.4 bc	16.0 d	9.7 c	2.8 d	11.1 b
OD(A + Ch)-CPD-VMFD	10.4 e	51.0 e	33.3 bc	31.1 e	38.7 c	6.5 b	63.2 b	234 d	3.9 cd	3.1 c	13.9 e	7.8 d	2.8 d	24.3 a
OD(AP + Ch)-CPD-VMFD	11.6 e	56.2 e	14.0 d	27.9 e	23.3 cde	4.5 bc	39.4 c	177 d	3.3 d	2.7 d	18.0 cd	11.1 b	4.2 c	19.0 a

† POM, Ch, A, and AP stand for pomegranate, chokeberry, apple (at 50% in mixture with 50% chokeberry), and apple (at 75% in mixture with 25% chokeberry), respectively.
‡ A1: delphinidin-3,5-diglucoside; A2: cyanidin-3,5-diglucoside; A3: delphinidin-3-glucoside; A4: cyanidin-3-glucoside; A5: pelargonidin-3-glucoside; A6: cyanidin-pentoside; A7: non-pomegranate-typical anthocyanins.
¶ Mean values followed by the same letter, within the same column, were not significantly different ($p < 0.05$), according to HSD Tukey's least significant difference test.

detected in arils osmotically dehydrated in chokeberry juice, whereas none of them was identified in other pomegranate products. The OD-treatment including a "*Wonderful*" juice showed higher quantity of cyanidin-3-glucoside (A4) than CPD-VMFD (drying control without OD). These results agreed with previous studies showing that the amount of cyanidin-3-glucoside in "*Wonderful*" pomegranate variety juice was higher than in "*Mollar de Elche*" (Mena et al., 2014).

3.3. Antioxidant capacity (TEAC ABTS+ and FRAP)

The ABTS+ antioxidant capacity was affected by the osmotic dehydration pre-treatments as well as the drying methods (Table 3). The highest ABTS+ values were found in the samples osmotically dehydrated in pomegranate juice and in pomegranate and chokeberry juice, and were 1.6- and 1.2-times higher than the values of FD and CPD-VMFD control samples, respectively. On the other hand, the lowest ABTS+ value was found in samples osmotically treated with apple juice.

Similarly, the FRAP values of the same two treatments were 1.6- and 1.2-times higher than those of the control samples. Again, the lowest FRAP values were found in samples treated with apple juice.

The experimental results obtained demonstrated that an initial osmotic dehydration step with pomegranate and pomegranate/-chokeberry concentrated juices can increase the antioxidant capacity of dried pomegranate arils. These results did not agree with results published in other fruits, such as dried sour cherry, in which the osmotic dehydration step led to lower antioxidant activities of dried products (Nowicka et al., 2015a). This decrease in the antioxidant capacity of dried sour cherries was justified by short drying time and polymerisation reactions (Yilmaz & Toledo, 2005), and could be also due leaching of natural solutes into the osmotic solutions (Bchir, Besbes, Karoui, Paquot, et al., 2012).

There were not significant correlations among the total anthocyanin content and the ABTS+ or FRAP values; however, a positive and significant ($p < 0.001$) correlation was found between ABTS+ and FRAP, ($R^2 = 0.977$). Thus, other compounds (not only anthocyanins) were responsible for the antioxidant capacity of dried pomegranate arils; it is well established that hydrolyzable tannins (punicalagins and punicalins) and phenolic acids (e.g. ellagic acid) are the key compounds in the antioxidant capacity of pomegranate fruits (Calín-Sánchez et al., 2013; Gil, Tomás-Barberán, Hess-Pierce, Holcroft, & Kader, 2000)

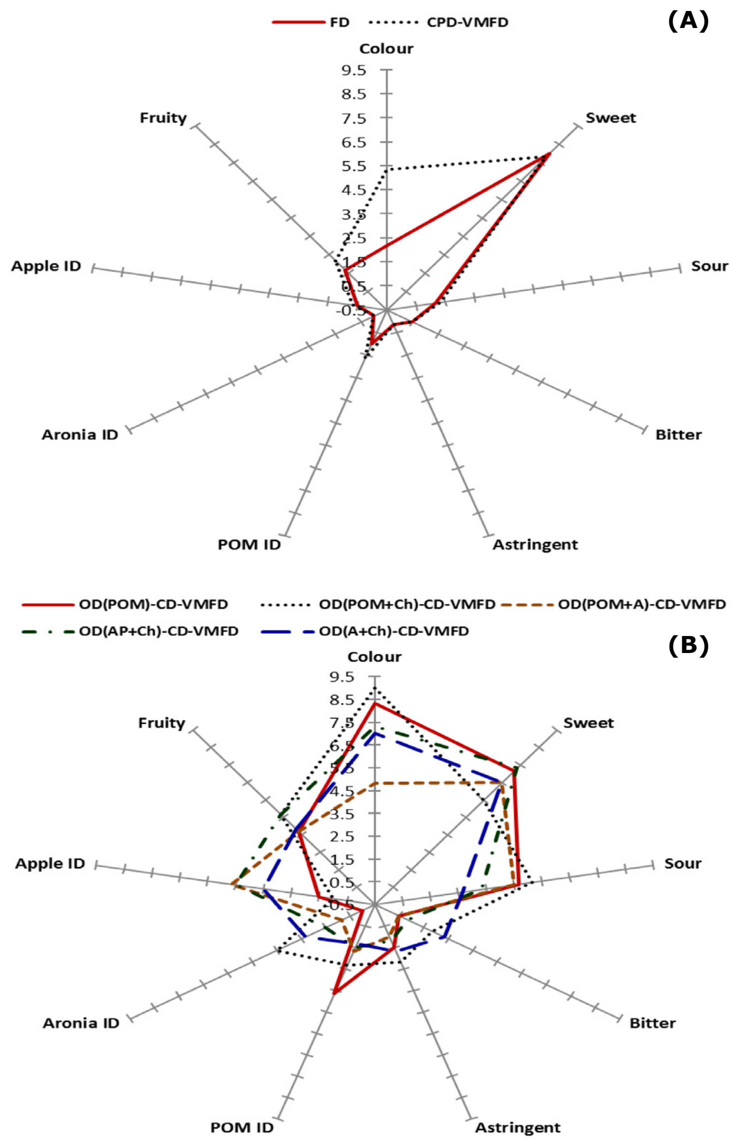
3.4. Colour

The dried pomegranate arils available in the market now are usually brownish and not attractive to consumers, due to degradation of arils anthocyanins (Maskan, Kaya, & Maskan, 2002).

The values of coordinate L^*, lightness, considerably decreased in all samples when compared to the freeze dried (FD) products (Table 3). Among the samples analysed, the darkest products (lowest L^* values) were found in the OD(POM + A), OD(A + Ch), and OD(AP + Ch) samples.

The level of red pigments, as described by the coordinate a^*, was affected by osmotic dehydration. The highest a^* values were found in the samples osmotically treated with pomegranate–chokeberry juices and pomegranate juice; their a^* values were statistically equivalent to those of the control samples (FD and CPD-VMFD). The high values of these two treatments (pomegranate-chokeberry and pomegranate juices) led to an equivalent red colour intensity to that previously reported in dried-pomegranate arils, cultivar "*Hicaz*" (<14.0) (Horuz & Maskan, 2015), which have a very intense red colour, which is lacking in the arils of the cultivar used in the current study "*Mollar de Elche*".

control (A) and osmotic-dehydrated (B) samples.



Legend: *POM, Ch, A, and AP stand for pomegranate, chokeberry, apple (at 50% in mixture with 50% chokeberry), and apple (at 75% in mixture with 25% chokeberry), respectively.*

Fig. 3. Descriptive sensory analysis of dried "*Mollar de Elche*" pomegranate arils [control (A) and osmotic-dehydrated (B) samples].

The application of an osmotic dehydration step resulted in a significant decrease in the b^* values (Table 3), as compared to control samples. Osmotic dehydration in "*Wonderful*" pomegranate juice affected the b^* values to a lesser extent when compared to the rest of the analysed osmotic solutions.

The colour difference (ΔE) of samples ranged from 2.5 to 24.3 (Table 3). The lowest ΔE values were found in the control samples, followed by samples osmotically treated with pomegranate juice.

Pearson's correlation coefficient showed that the total anthocyanin content was positively ($p < 0.05$) correlated with L^*, a^*, and b^* coordinates (R^2 = 0.81, 0.59, and 0.62, respectively).

As a summary of this section, it can be stated that application of "*Wonderful*" pomegranate juice resulted in the best colour of the osmotically dried arils of "*Mollar de Elche*", and that anthocyanins played an important role in the final colour of dried arils.

3.5. Descriptive analysis

The main objective of this study was to optimize the colour of the samples, because it is main attribute driving consumer acceptance. The values of colour (the higher the value, the more intense reddish was the colour) obtained by the trained panel were: 2.2 (FD) < 4.8 [OD(POM-A)-CPD-VMFD] < 5.3 (CPD-VMFD) < 7.0 [OD(AP-Ch)-CPD-VMFD] < 7.3 [OD(A-Ch)-CPD-VMFD] < 8.3 [OD(POM)-CPD-VMFD] < 9.0 [OD(POM-Ch)-CPD-VMFD] (Fig. 3). The goodness of the appearance of the samples of the last two treatments can be seen in Fig. 1.

It is noteworthy that none of the samples had measurable off-flavours notes, supporting the high quality of the products.

Control samples (FD and CPD-VMFD) presented a flat (no complex) sensory profile, with their predominant attribute being sweetness (Fig. 3A). The profile changed considerably when "*Wonderful*" pomegranate juice was used during the osmotic dehydration [OD(POM)-CPD-VMFD]; this step enriched the sensory complexity of the "*Mollar de Elche*" dried arils by increasing the intensities of sourness (4.7), fruity notes (3.7) and pomegranate flavour (3.7). The use of chokeberry juice significantly decreased sweetness, and increased astringency, bitterness, sourness, and chokeberry flavour. The use of apple juice led to samples with low colour intensity and low pomegranate flavour but high intensity of apple; the apple flavour masked that of pomegranate.

Besides, three texture attributes were evaluated: crispiness, adhesiveness, and solubility in the saliva. There were no significantly differences among the dried samples. Samples were defined by low crispiness (<1 in scale 0–10) and low solubility (<1.5). Regarding the adhesiveness, the OD pre-treatment significantly increased them due to higher contents of soluble solids. Control samples (FD and CPD-VMFD) had values ~1.6, while the OD-samples had values ranging between 2.4 and 3.8. This experimental finding may be justified because samples with a pre-treatment had more quantity of sugar (OD with juice concentrated = 40 °Brix), and it seems reasonable that a positive relationship between high sugar content and high adhesiveness intensity may exists.

4. Conclusions

Osmotic dehydration using "*Wonderful*" pomegranate and chokeberry concentrated juices improved the quality of dried "*Mollar de Elche*" pomegranate arils in terms of rehydration rate, antioxidant capacity, colour, and sensory profile; however still further research is needed to fully optimize this combined drying treatment because the freeze-dried sample still had higher anthocyanin content and better instrumental colour parameters. On the basis of the results obtained connected with colour, antioxidant capacity and anthocyanin contents, the combination of pomegranate and chokeberry juices resulted in the highest intensity of the key flavour notes; the second best results were obtained by using just "*Wonderful*" pomegranate juice. All treatments were acceptable in terms of sensory parameters (for example, had no off-flavours) but with different characteristic flavour notes. Improving the quality of dried pomegranate arils must increase the popularity of this product, even in groups with reduced fruit consumption, such as teenagers and children, leading to higher consumer acceptance, consequently higher product demand, and finally higher benefits for the farmers and industry. The energy consumption and cost of the drying treatments will be evaluated in future studies, as done by Calín-Sánchez et al. (2014).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.foodchem.2017.04.033.

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