Upcycling food ingredients from orange by-products by hot air-microwave drying. Impact on energy consumption.

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Abstract

Currently industrial citrus by-products represent a relevant environmental issue. The main aim of this work was the chemical characterization of the different bioactive compounds obtained after hot air-microwave drying (HAD+MW) of orange by-products, and their further conversion into three upcycled ingredients with health-related benefits: aqueous extract, ethanolic extract and dietary fibre. Total phenolics, antioxidant capacity, individual phenolic acids, flavonoids, limonin and carotenoids were monitored during blanching and colour extraction steps by analysing fresh by-products and process co-products: an aqueous extract rich in polyphenols and an ethanolic extract rich in carotenoids. After drying, the resulting fibre was characterized in terms of chemical composition, soluble and insoluble dietary fibre content and particle size. Technological properties and colour were compared to those of commercial citrus fibre. Energy and time consumption were compared with conventional hot air drying (HAD). Most polyphenols (50-65 %) and limonin (70 %) were extracted during the blanching step. 86 % of carotenoids were removed by soaking in ethanol. The orange fibre obtained had 71.9 g DF/ 100 g and antioxidant properties (205 mg TE/ Kg_{dm}). Whiteness, water retention capacity and oil retention capacity were similar to commercial citrus fibre. HAD+MW reduced drying time and energy consumption by up to 50 % compared to HAD.

Keywords: upcycled ingredients; orange by-product; microwave drying; dietary fibre; bioactive compounds; citrus polyphenols; limonin

32 **NOTATION**

33 **Abbreviations**

34	MC	moisture content
35	a_w	water activity
36	MW	microwave drying
37	HAD	hot air drying
38	TPC	total phenolic content
39	AOC	antioxidant capacity
40	WRC	water retention capacity
41	DF	dietary fibre
42	GAE	Gallic acid equivalent
43	TE	Trolox equivalents
44	TDF	total dietary fibre
45	IDF	insoluble dietary fibre
46	SDF	soluble dietary fibre
47	dm	dry matter
48	T_x	temperature at experimental conditions “x” (°C)
49	φ	relative humidity (-)
50	M	mass per time in wet basis (kg s^{-1})
51	M'	mass per time in dry basis (kg s^{-1})
52	x	mass fraction (kg kg^{-1})
53	X	absolute moisture ($\text{kg water kg dry air}^{-1}$)
54	t	time (s)
55	h	specific enthalpy (J kg^{-1})
56	p_s	water saturation pressure (kPa)
57	C_p	heat capacity at constant pressure ($\text{W g}^{-1} \text{K}^{-1}$)

58	ΔH	molar enthalpy (J mol^{-1})
59	ρ	density (kg m^{-3})
60	v	velocity (m s^{-1})
61	S	section (m^2)
62	E	energy (kW or kWh)
63	W	microwave energy (W g^{-1})
64	P	absolute pressure (Pa)
65	T	absolute temperature (K)
66	R	ideal gases universal constant ($\text{J mol}^{-1} \text{K}^{-1}$)

67 **Subscripts and superscripts:**

68	amb	ambient
69	D air	drying air
70	D	drying
71	0	initial time
72	v	vapour
73	w	water

74 **Supplementary material:**

75 SC1: Dataset deposited at Zenodo. DOI 10.5281/zenodo.5653562: Energy calculations,
76 tables, figures and manuscript files.

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Introduction

Food industry generates millions of tons per year of plant processing by-products, particularly in the fruit processing sector. The exploitation of these by-products is limited because their inadequate biological stability.

The most common and easiest route for exploitation citrus processing by-products is to use them in animal feeding (Bampidis & Robinson, 2006). For more than twenty years, the use of processing technologies to fractionate and recover potentially high value components such as dietary fibre (DF) (Aravantinos-Zafiris, Oreopoulou, Tzia, & Thomopoulos, 1994; Harris & Smith, 2006; Larrauri, 1999), carotenoids and natural antioxidants (Kris-Etherton et al., 2002; Lu & Foo, 2000; Moure et al., 2001) has been investigated. Studies have been conducted to demonstrate the physical, chemical and functional properties of dietary fibre (DF) derived from fruits such as oranges and lemons (Fernández-López et al., 2009; Figuerola, Hurtado, Estevez, Chiffelle, & Asenjo, 2005; Marin, Soler-Rivas, Benavente-Garcia, Castillo, & Perez-Alvarez, 2007; Russo et al., 2014), passion fruit (Hernández-Santos et al., 2015) and pitaya peels (Zhuang, Zhang, & Sun, 2012) highlighting their antioxidants properties.

Sensory properties such as colour, flavour and above all, bitterness of citrus by-products has limited their use in food application. Some polyphenols (flavonoids) and terpenoids such as limonin are responsible for this bitterness. Flavanones are usually present in diglycoside form, conferring the typical taste to citrus fruits (Macheix, 1990). The colour is mainly given by carotenoids and to a lesser extent by flavonoids (Braddock, 1999).

Different process for obtaining upcycled dietary fibre from orange by-products have been described by some authors (Andrade, Jong, & Henriques, 2014; Attavanich, 2003; de Moraes Crizel, Jablonski, de Oliveira Rios, Rech, & Flôres, 2013), but these methods use conventional hot air drying. In previous work carried out by Fava et al. (2013) the use of hot air-microwave technology was tested as a procedure for stabilizing citrus by-products and for further recovery of bioactive compounds and dietary fibre from the dried by-product. Results from this work showed that the use of microwaves did not only minimize processing time and microbial load, but also showed potential for obtaining upcycled dietary fibre with neutral colour and optimum water retention capacity from the stabilized by-product.

Drying costs are one of the main costs in valorisation processes of high moisture by-products and represents a key factor in the economic feasibility of the process (Zhang, Tang, Mujumdar, & Wang, 2006). High temperatures or long drying times in conventional air drying may cause serious damages to product flavour, colour and nutrients, reducing bulk density and rehydration capacity of the dried product (Chen, Yang, & Liu, 2011; Femenia et al., 2009). Microwaves have been used for food drying in order to avoid quality losses and to achieve fast and effective thermal processing (Nieto Calvache et al., 2015; Prothon et al., 2001). The use of microwaves in some processes such as drying results in a substantially reduced processing time leading to increased production capacity, as well as improved quality and shelf life of final products (Zhang et al., 2006).

Apart from drying, there are two important pre-treatments that are used for improving final product quality: blanching and extraction of undesired compounds, mainly responsible for colour and bitterness. Blanching is necessary to eliminate soluble sugars and to inactivate the polyphenol oxidases to avoid possible browning of the fibre during the drying step (Morales-Blancas, Chandia, & Cisneros-Zevallos, 2002; Peerajit, Chiewchan, & Devahastin, 2012; Wuttipalakorn, Srichumpuang, & Chiewchan, 2009). Different methods have been studied to reduce bitterness based on the elimination of limonin content, which is the molecule responsible for bitterness in citrus fruits. Some of these methods are: use of limonoate dehydrogenase to prevent development of limonin (Hasegawa, Brewster, & Maier, 1973), use of adsorbents such as β -cyclodextrin for removal of limonin (Konno, Misaki, Toda, Wada, & Yasumatsu, 1982) or use of ethanol (Attavanich, 2003; Ubando-Rivera, Navarro-Ocana, & Valdivia-Lopez, 2005).

High fibre ingredients exhibit many properties that influence the physiological functions of foods. However, preparation steps such as drying may modify these properties. Understanding the properties of high fibre ingredients is essential to developing new food products (Nelson, 2001). Particle size is of major importance, determining both fibre technological functionality and fibre role in the digestive tract (transit time, fermentation, faecal excretion). The shape and the size of fibres depends on degree of processing and may vary during transit in the intestine tract as result of digestion process. Water retention capacity (WRC), swelling capacity (SC) and oil retention capacity (ORC) suggest some possibilities about the use of upcycled fibres as ingredients in food products: eg., dietary fibre with high ORC allow the stabilization of high fat food products and emulsions.

Dietary fibres with high WRC and SC can be used as functional ingredients to avoid syneresis and modify the viscosity and texture of some formulated low fat foods (Elleuch et al., 2011).

The aim of this work was (i) to chemically characterize the liquid extracts obtained after hot air-microwave drying of orange by-products (ii) to monitor the distribution of individual polyphenols, carotenoids and limonin during blanching and ethanol soaking and (iii) to study the physico-chemical and technological properties of the upcycled dietary fibre obtained and (iv) to compare energy and time consumption of hot air-microwave drying compared to conventional hot air drying.

Material and Methods

Plant material

Oranges (*Citrus sinensis* (L.) Osbeck, var Washington Navel) were processed for juice extraction by a Spanish citrus juice industry (Grupo Leche Pascual, Aranda de Duero, Spain) and the residue was supplied as samples of orange by-products for this study, including flavedo, albedo, segments, juice sacs and seeds. For polyphenol analysis only, samples were lyophilized in a freeze-dryer (Labconco, freezone 4.5, Kansas, USA) for 24 h, powdered using a blender (IKA, Germany) and stored at 4 °C in the dark until use.

Procedure and sample preparation

This process was developed at the pilot plant of AZTI (Spain). The methodology consisted of five main steps (Figure 1): Mincing, blanching, decolouring, drying and milling.

Figure 1: Flow diagrama of the process developed

Orange by-products were minced to 0.5-1 cm particle size using a cutter (Stephan UMC-5, Stephan, Germany) and blanched in water (vol 1:4) at 65 °C for 5 min. The maximum operational temperature was considered 65 °C considering that blanching causes adverse losses in the antioxidant capacity over 70 °C (Abu-Ghannam & Jaiswal, 2015). Afterwards, samples were centrifuged at 1000 x g for 5 min using a high-performance centrifugal machinery (Comteifa, Barcelona, Spain). The aqueous extract resulting from blanching was collected and analysed for polyphenols characterization.

Approximately, 500 g batches of blanched by-products were then soaked in food grade 96 % ethanol (two cycles, 1:5 w/vol) under constant agitation, using a mixing/overhead stirrer RW 28 basic with propeller stirrer 4-bladed (IKA, Germany), during 1 hour for removal of compounds responsible for colour and bitterness. Finally, samples were

centrifuged at 1000 g for 5 min to remove excess ethanol. Ethanol was collected for its characterization.

Decoloured samples (300 g batches) were then introduced in a pilot scale 2450 MHz electromagnetic microwave equipment (MMP20T, Sairem S.A., Miribel, France). Combined drying chamber dimensions were 0.66 m x 0.66 m x 0.83 m, air velocity was 7 m/s, air temperature was 35 °C (relative humidity = 6.5 %), ambient temperature was 15 °C and relative humidity was 60 %. For the energy consumption calculations, HAD+1.5 W/g was studied (the applied MW power was referred to the initial weight). Based on results from previous work (Fava et al., 2013), the drying process was performed during 90 min. This was the time needed to reach 0.01 kgw/kgT of final moisture in orange by-product. After drying, samples were milled using an ultracentrifuge mill (ZM 100, Retsch, Germany) with a sieve of 200 µm. At this stage, powdered samples were packed in plastic bags for characterization. The procedure described above was carried out in duplicate from the same batch of fresh material in order to avoid variability due to cultivar, seasonality and time of harvest.

Characterization methods

Chemicals and reagents

Trolox [(+/-)-6-hydroxy-2,5,7,8-tetramethyl-chroman-2-carboxylic acid], Folin Ciocalteu reagent, DPPH [2,2-diphenyl-1-picrylhydrazyl] and Phenolic standards (gallic acid, chlorogenic acid, caffeic acid, p-coumaric acid, ferulic acid, eriocitrin, hesperidin, naringin, quercetin-3-O-glucoside and limonin) were obtained from Sigma Aldrich, (St. Louis, USA). Sodium carbonate, methanol, formic acid, acetone, n-hexane and ethanol (Panreac Quimica S.A.U., Barcelona, Spain) and Mili-Q water were also used. All chemical and reagents used in this study were of analytical grade.

Proximate analysis

Samples were analysed according to the ISO recommended standards: Moisture by 1442:1997, (ISO, 1997), ash by ISO 936:1998 (ISO, 1998), protein content by ISO 937:1978 (ISO, 1978) and fat content by (ISO, 1973). Total sugars were analysed by Luff–Schoorl method for reducing sugars (Lees, 1968).

The total protein content was determined by the Kjeldahl method using the Digestion Unit K-435 and a distillation unit B-324 (Buchi Labortechnik AG, Switzerland). A correction factor of 6.25 was used. Crude fat was extracted with petroleum ether using a Soxtec HT

1043 extraction unit (Foss Tecator, Denmark). The ash content was performed in a muffle furnace (Carbolite CWF 1100, Carbolite, UK) set to 550 °C. The moisture content was determined at 105 °C (Memmert, Germany), for approximately 18 h until constant weight. The carbohydrate content was determined by the difference.

For the fibre ingredient obtained, total, soluble, and insoluble dietary fibre was determined by the AOAC enzymatic-gravimetric method, 991.43 (1991). Neutral detergent fibre and acid detergent fibre was analyzed by the gravimetric method AOAC 973.18 (1977). Cellulose content was calculated as the difference between acid detergent fibre and acid detergent lignin. Finally, hemicellulose content was determined according to NF V 18-122 (AFNOR, 1997).

The total caloric value was calculated by adding up the calories provided by the energy-containing nutrients and considering 4 kcal/g for protein, 9 kcal/g for fat, 4 kcal/g for carbohydrate and 2 Kcal/g for fibre ("Regulation EU No 1169/2011", 2011).

Polyphenol extraction

Polyphenol extraction from orange peels was carried out adapting the procedure described by Khan, Abert-Vian, Fabiano-Tixier, Dangles, and Chemat (2010). Dried powdered samples (1 g) were placed in a 50 mL falcon tube and 20 mL of extraction solution consisting of MeOH:H₂O:Formic acid (75:24.9:0.1; v/v/v) was added. Then, it was sonicated in an ultrasound bath (Ultrasounds-H Bath model, P-Selecta) at 30 °C, 25 MHz for 30 min, centrifuged at 2660 g for 10 min, and taken to a final volume of 25 mL with MQ water. Extracts were filtered through a 0.22 µm PVDF filter and stored at 4 °C in the dark until use for the determination of total phenolics, phenolic acids and flavonoids by chromatography and for the antioxidant assay.

Total phenolics content

Total phenol content (TPC) was measured by Folin-Ciocalteu assay, using gallic acid as standard (2-20 mg/L) adapted from Singleton, Orthofer, and Lamuela-Raventos (1999) to plate reader spectrophotometer (Varioskan, Thermo Scientific, USA). The reaction mixture was composed of 140 µL of orange extract obtained (previously diluted 1:50) or gallic acid standards, 30 µL of Folin–Ciocalteu reagent and 140 µL of 20 % sodium carbonate in 96-well plates. The reaction was mixed and allowed 1 h in dark. The absorbance was measured at 750 nm and TPC was expressed as mg of gallic acid equivalent per kg of dried matter (mg GAE/kg dm).

Individual polyphenols and limonin analysis by chromatography

Standard stock solutions were prepared in methanol, except for hesperidin that was dissolved with water: dimethylformamide (80:20, v/v) and stored at -20 °C in darkness. Agilent 1100 Series liquid chromatograph (Agilent, California, USA) equipped with a degasser, a binary pump, an autosampler, a column oven and a diode array detector (DAD) coupled to a mass detector Ion Trap XCT PLUS with an electrospray interface was used for the analysis. Chromatographic separation was performed at 30 °C on a Gemini C18 analytical column (150 mm x 2.1 mm i.d, 5 µm, Phenomenex, Torrance, Canada) with a C18 column guard cartridge. A gradient program for general polyphenol analysis was employed. The mobile phase consisted of water/0.1 % formic acid (Solvent A) and methanol/0.1 % formic acid (Solvent B). The elution conditions applied were: 0-2 min, 10 % B isocratic; 2-35 min, linear gradient 10-35 % B; 35-55 min, linear gradient 35-60 % B; 55-58 min, 60 % B isocratic; 58-59 min, linear gradient 60-100 % B, 59-62 min, 100 % B isocratic; and finally, 62-63 min, linear gradient 100-10 % for reconditioning the column. Citrus flavanones eriocitrin, hesperidin and naringin were analysed separately under the same conditions but following shorter elution chromatogram: 0-12 min, linear gradient 33-40 % B; 12-13 min, linear gradient 40-100 % B; 13-15 min, 100 % B isocratic; and finally, 15-16 min, linear gradient 100-33 % for reconditioning the column. For limonin analysis, the mobile phase consisted of water/ 0.1 % formic acid (Solvent A) and acetonitrile/0.1 % formic acid (Solvent B). The elution conditions applied were: 0-5 min, linear gradient 20-35 % B; 5-18 min 35 % B isocratic; 18-19 min, 35-60 % B linear gradient; 19-22 min, 60 % B isocratic, 22-23 min, 60-100 % B linear gradient; 23-25 min 100% B isocratic and finally 25-26 min linear gradient 100-10 % for reconditioning the column. The flow rate was 0.4 mL/min and 10 µl of sample was injected. Chromatograms were monitored at 210, 254, 280 and 320 nm. Mass spectral analysis was performed in positive (for polyphenols) and negative (for limonin) ionization mode. Nitrogen was used as drying gas at a constant flow rate of 8 l/min and the temperature was set at 350 °C. The nebulizer gas pressure was set at 40 psi and the capillary voltage at 3500 V. Polyphenols and limonin were analysed by direct injection of the orange extracts, previously diluted (1:2) in water and filtered through a 0.22 µm PVDF filter. Identification was made possible by comparison of retention times, UV-visible spectra and MS/MS data with those of the standards. Quantification was

performed by reporting the measured integration areas in the calibration equation of the corresponding standards.

Antioxidant capacity

The antioxidant capacity (AOC) was measured by DPPH method using Trolox as standard (0-225 mg/L) adapted from (Brand-Williams, Cuvelier, & Berset, 1995) to a plate reader spectrophotometer (Varioskan, Thermo Scientific, USA). Briefly, 20 µl of orange extract (without dilution) or Trolox standards were added to 280 µl of 25 mg/l DPPH solution (dissolved in methanol). The absorbance of the remaining DPPH was measured after 30 min at 515 nm. The final results were expressed as mg of Trolox equivalents (TE) per kg of orange dry matter (mg TE/kg_{dm})

Total carotenoids

Total carotenoids were determined spectrophotometrically by adapting the method described by (Sinclair, 1984).

For carotenoids extraction from solids samples, 0.1 g of sample was placed in a 15 mL falcon tube and 10 mL of acetone was added, then, sonicated in an ultrasound bath (Ultrasounds-H Bath model, P-Selecta) at ambient temperature for 30 min, centrifuged at 2660 g for 15 min. The supernatant was recovered and filtered through a 0.22 µm PTFE filter. For aqueous samples a liquid-liquid extraction with n-hexane was performed. Sample (2.4 mL) were mixed with n-hexane (2 mL) and mixed for 5 minutes in a vortex mixer. The n-hexane layer was recovered and filtered through a 0.22 µm PTFE filter. The absorbance at 450 nm was measured using a spectrophotometer (Varioskan, Thermo Scientific, USA). Concentration values were calculated through the mass extinction coefficient of beta-carotene in acetone (2542 mL/g cm). Values were expressed in ppm βCeq. (ppm of β-Carotene equivalent).

Particle size distribution

Analysis of the particle size distribution was carried out using a laser diffractometer Mastersizer 2000 (Malvern Instruments Ltd, Worcestershire, UK). The Mie theory was applied by considering a refractive index of 1.5 and absorption of 0.01. Samples were diluted in de-ionised water at 2000 rpm until an obscuration rate of 15 % was obtained. D_{3,2} (surface weighted mean diameter) and D_{4,3} (volume weighted mean diameter), D₁₀ (the size which 10% of the particles are below), D₅₀ (median size) and D₉₀ (the size which 90% of the particles are below) were obtained.

In order to study the technological properties of the new fibre obtained, commercial citrus fibre (Herbacel AQ Plus Citrus Fibre, Herbstreith & Fox) was used as a reference for water and oil retention capacities, swelling capacity and colour.

Water and oil retention capacity

For water retention capacity (WRC) and oil retention capacity (ORC), sample ($0.5 \text{ g} \pm 0.0001 \text{ g}$) was hydrated in 20 mL of distilled water or sunflower oil, respectively, in a 50 mL falcon tube and left overnight to ensure the fibre was fully hydrated. Then, the tubes were centrifuged at $1000 \times g$ for 10 min (adapted from Robertson et al., 2000). The supernatant was decanted and the tubes were carefully inverted to drain residual unbound water or oil from the sample. For WRC, the remaining pellet was dried overnight in an oven at 105°C and weighed to consider possible solid matter losses in the draining step. WRC and ORC were calculated as the amount of water or oil retained by the pellet (g/g_{dm}).

Swelling capacity

Swelling capacity (SC), defined as the ratio of the volume occupied when the sample is immersed in excess of water after equilibration to the sample weight, was measured by the method of (Raghavendra, Rastogi, Raghavarao, & Tharanathan, 2004). To 0.2 g of dry sample placed in a graduated test tube; around 10 mL of water was added to hydrate the sample for 18 h; then the final volume attained by fiber was measured and expressed as volume/g of original sample (dm).

Colour

Colour was measured following CIELab scale. L^* , a^* and b^* parameters were measured using a Minolta CM 3500d colorimeter (Osaka, Japan), where the L^* value gave information on the luminance, and the a^* and b^* values gave information about the colour of the sample (a : from green to red, and b : from dark blue to yellow). Whiteness was determined using the following formula: $\text{whiteness} = 100 - \sqrt{[(100 - L^*)^2 + a^{*2} + b^{*2}]}$, according to (Park, 1995).

Energy consumption

For the energy consumption calculations, two drying experiments were carried out in triplicate comparing conventional hot air drying at 55°C (HAD) with hot air drying at 35°C coupled with microwaves (HAD+MW). HAD was carried out on the same equipment

as described above without applying microwave energy. Drier dimensions were 0.66 m x 0.66 m x 0.83 m, air velocity was 7 m/s, ambient temperature was 15 °C and relative humidity was 60 %. Mass variation was measured at different times by a precision balance Mettler Toledo AB304-S (± 0.001 g).

Statistical analysis

Statistical analysis of variance (ANOVA) was performed by Statgraphics Centurion XVI 16.1 to estimate the differences among the fibre obtained by the new process and the commercial fibre. Evaluations were based on a $p \leq 0.05$ significance level.

Results and discussion

The proximate chemical composition of the fresh by-product is shown in Table 1.

Table 1: Fresh orange by-product and orange fiber composition in % wet matter.

Parameters	Fresh by-product (%)
Water	81.9 ± 1.9
Total sugars	4 ± 2
Protein	1.0 ± 0.5
Fat	<0.1
Ash	0.5 ± 0.1
TDF	6.4 ± 1.8

^a Values represent mean of 3 replicates $\pm$ SE

Average moisture content prior to drying procedure was 81.9 ± 1.9 %. It can be seen that after water, the main component was dietary fibre (6.4 ± 1.8 %) followed by sugars (3.8 ± 2.2 %), therefore the efficient removal of water and sugar from by-products is important for their exploitation into valuable ingredients such as dietary fibre. Furthermore, the removal of colour and bitter compounds from the fibre ingredient is crucial for its application in food matrices without affecting the sensory properties of the final product.

Total phenolic content, antioxidant capacity, phenolic acids and flavonoids in fresh orange by-products are listed in Table 2. TPC found in raw material was 8520 mg GAE/kg_{dm}. If results are expressed in wet matter (1713.4 mg GAE/kg they can be compared to those obtained by (Khan et al., 2010) who reported 2750 mg GAE/ kg after 30 min of ultrasound extraction from orange peel' extracts. Polyphenols identified and quantified in orange by-products were chlorogenic acid, caffeic acid, *p*-coumaric acid, ferulic acid, eriocitrin, quercetin, quercetin-rutinoside (rutin), quercetin-glucoside,

narirutin and hesperidin. Ferulic acid was the most abundant phenolic acid (42 ± 2.5 mg/kg_{dm}). These results are in accordance with data reported by Russo et al. (2014) who detected 46 ± 0.5 mg ferulic acid/ kg lemon by-products.

Table 2: Total polyphenol content (TPC), antioxidant content (AOC), carotenoids, limonin, phenolic acids and flavonoids in fresh orange by-products. Results are expressed as mg/kg of dried matter of raw material (fresh orange peel)^a. TPC is expressed in mg GAE/kg_{dm} and AOC in mg TE/kg_{dm}.

Analyte	Concentration (mg/kg _{dm})
TPC	8520 ± 17
AOC	1568 ± 10
Carotenoids	80 ± 6
Limonin	75.8 ± 9.2
<i>Phenolic acids</i>	
<i>p</i> -Cumarinic acid	10 ± 4
Ferulic acid	42 ± 2
Caffeic acid	12.0 ± 1.3
Chlorogenic acid	1.4 ± 0.8
<i>Flavonoids</i>	
Eriocitrin	17 ± 2
Quercetin-rutinoside (rutin)	7.5 ± 1.2
Quercetin-glucoside	3.0 ± 0.4
Quercetin	27 ± 8
Naringin	ND
Narirutin	287 ± 10
Hesperidin	866 ± 11

ND: not detected. ^a Values represent mean of 3 replicates ± SE

Hesperidin was the main flavonoid identified (866 ± 11 mg/kg_{dm}). Taking into account the variability due to variety, cultivar, harvest month, etc., and expressing the results in fresh weight (190 ± 2 mg/kg), they are consistent with those found by Ramful, Bahorun, Bourdon, Tarnus, and Aruoma (2010). These authors examined hesperidine in different citrus fruits and reported a range between 83 ± 0.06 and 234 ± 1.73 mg/g fresh weight. Nonetheless, no naringin was detected, instead, its isomer narirutin was identified and quantified as naringin (287 mg/kg_{dm}). Naringin and narirutin are flavanone glycosides of

naringenin. They differ in the disaccharide residue: neohesperidose (rhamnosyl- α -1,2-glucose) for naringin and rutinoside (rhamnosyl- α -1,6-glucose) for narirutin (Tripoli, Guardia, Giammanco, Majo, & Giammanco, 2007). Neohesperidosides have a bitter taste, while rutinosides have a bland taste. Hesperidin has also a rutinoside residue, thus, both main flavonoids found in orange peels have not a bitter taste. Therefore, bitter taste was mainly due to the presence of limonin.

Preliminary experiments for the optimization of the blanching and ethanol soaking conditions were published in Fava et al. (2013). These results showed that blanching by water immersion at 65° C and colour extraction by ethanol soaking in two consecutive cycles of 1:5 (w/vol) were more effective for compounds extraction.

During the blanching step main polyphenols, flavones glycosides, hesperidin and narirutin were removed. This could be due to the fact that blanching enhances polyphenol extraction by eliminating soluble sugars, making polyphenols flavonoids, usually linked to bigger molecules such as carbohydrates and proteins, more available. The ethanol soaking step was carried out for removal of compounds responsible of colour and bitternes (carotenoids and limonin, respectively).

As a result of the process developed, two main co-products were obtained: water from blanching (aqueous extract) and ethanol from decolouring (ethanolic extract). The concentrations of TPC, AOC, carotenoids, limonin, hesperidin and narirutin in both co-products are presented in Table 3.

Table 3: Distribution of total carotenoids, limonin, hesperidin and narirutin in process co-products (water and ethanolic extracts) and in orange fiber. AOC is expressed as mg TE/kg.

mg/kg ^a	Aqueous extract	Ethanolic extract
TP	1635.3 $\pm$ 12.3	599 $\pm$ 8
Hesperidin	1442.1 $\pm$ 9.1	212 $\pm$ 12
Narirutin	377.5 $\pm$ 11.3	63 $\pm$ 4
AOC	403.2 $\pm$ 9.7	105 $\pm$ 5
Limonin	34.4 $\pm$ 2.4	12.2 $\pm$ 0.7
Carotenoids	0.6 $\pm$ 0.0	40.4 $\pm$ 5

^a Results are expressed as mg/kg of initial fresh orange peel. Values represent mean of 3 replicates $\pm$ SE

Regarding TPC, for each kg of fresh orange by-products, 1.63 g GAE were obtained in the aqueous extract and 0.59 g GAE in the ethanolic extract. For each kg of fresh orange by-products, AOC was 0.4 g TE in the aqueous extract and 0.1 g TE in the ethanolic extract. Blanching water contained 51 % and 65 % of the recovered hesperidin and narirutin, respectively. Approximately 70 % of limonin was extracted during the blanching step and the rest was extracted by ethanol soaking (24 %). The aqueous extract was richer in total phenolics, antioxidants, limonin, hesperidin and narirutin than the ethanolic extract, which was richer in carotenoids. About 86 % of carotenoids were extracted with ethanol and very little were extracted during blanching (1%) due to its hydrophobic nature. Also, regarding the decolouring step, whiteness value of the fibre obtained (80.0 ± 2.3) was similar to the commercial fibre (82.9 ± 1.4). No significant differences ($p \leq 0.05$) were found for whiteness between these samples, therefore it can be concluded that the decolouring step was effective.

The orange fibre obtained did not have a significant amount of bitter compounds as the flavonoids present (hesperidin and narirutin) have no bitter taste and the limonin content was significantly reduced. Furthermore, the AOC of the fresh by-product was 1568.0 ± 10.2 mg TE/ kg_{dm}, at the end of the process the AOC of the fibre obtained was 205.1 ± 0.4 mg TE/ kg_{dm} which adds more value to this ingredient as it can be used also for its antioxidant properties.

The proximate composition of the orange fibre obtained is shown in Table 4. Results are similar to the TDF reported (Lundberg, Pan, White, Chau, & Hotchkiss, 2014) for citrus fibre obtained from by-products of orange juice production. In that study authors reported a fibre ingredient with 73.4 % of TDF, and composed of cellulose (15.9 ± 0.0), hemicellulose (10.0 ± 0.1), soluble fibres such as pectin (42.2 ± 1.1) and other type of fibres (5.1). The cellulose content of the fibre obtained by HAD+MW drying (15.7 ± 5.3) is very similar to the one reported by Lundberg et al. (2014). However, no hemicellulose was detected and the soluble fibre (24.5 ± 1.4) was also lower. This might be due to the differences among cultivars as the insoluble fibre components are mostly the elements that give the structure for the cell wall and the connective tissue of the plants. The ratio SDF:IDF was 1:2. It is generally accepted that the technical functionality of dietary fibre in foods is reliant on the ratio of soluble to insoluble dietary fibre. When dietary fibre is used as food ingredient it is suggested that the ratio of soluble to insoluble dietary fibre be 1:2. This ratio is more reflected in fibre from fruits and vegetables, while fibre from

cereals typically contain higher levels of insoluble dietary fibre as cellulose and hemicellulose (Figuerola et al., 2005).

Table 4: Proximate analysis of the fiber obtained from orange by-products by applying forced air-microwave drying.

g/ 100 g fiber	Fiber _{MW}
Moisture	10.5 ± 1.7
Protein	6.3 ± 0.3
Lipids	2.5 ± 0.1
Ash	3.5 ± 0.2
Carbohydrates	4.5
Sugar	0.8 ± 0.1
TDF	71.9 ± 1.6
SDF	24.5 ± 1.4
IDF	48 ± 3
cellulose	16 ± 5
hemicellulose	< 0.1
lignin	25 ± 5
Others	6.2
SDF:IDF	1:2

TDF (Total Dietary Fiber); IDF (Insoluble Dietary Fiber); SDF (Soluble Dietary Fiber). Data represent means and standard deviation of experiments performed in triplicate (n=9)

In the present study fibre size was determined in water and ethanol since some fibres swell in water and their particle size increase, besides alcoholic suspensions eliminate possible artifacts due to non-fibre components. The particle size distribution of the fibre obtained is presented in Table 5.

Table 5: Particle size distribution of the orange fiber obtained from orange by-products by applying forced air-microwave drying.

Particle size (µm)	Solvent	
	Ethanol	Water
D _{4,3}	48.5 ± 2.4	115.3 ± 1.6
D _{3,2}	17.4 ± 1.8	32.8 ± 2.3

D ₁₀	7.0 ± 1.3	12.9 ± 1.2
D ₅₀	34.2 ± 1.5	79.5 ± 1.13
D ₉₀	112.9 ± 8.7	274.1 ± 4.5

D_{4,3}, D_{3,2}, D₁₀, D₅₀ and D₉₀ represent volume weighted mean diameter, surface weighted mean diameter and 10 %, 50 % and 90 % of all particles finer than this size, respectively. Data represent means and standard deviation of experiments performed in duplicate.

A dramatic increase of all diameters was detected when comparing sizes in ethanol and in water, surely due to swelling in water. D_{4,3}, D₅₀ and D₉₀ increased their size x 2.5 while D_{3,2} and D₁₀ increased twice their size. The volume-weighted average diameter (D_{4,3}) is sensitive to the presence of large particles, whereas the surface-weighted average diameter (D_{3,2}) is more sensitive to the presence of small particles, therefore, larger particles swelled more than smaller particles. In the citrus fibre obtained, more than 90 % of particle sizes were over 112.9 ± 8.7 µm when suspended in ethanol, while their size increased to 274.1 ± 4.5 µm when immersed in water (Figure 2).

Figure 2: Particle size distribution in water (—) and in ethanol (---) of citrus fibre obtained by forced air-microwave drying

In order to assess the technological functionality of the orange fibre obtained, water, oil retention and swelling capacities were analysed and compared to the commercial fibre (Table 6).

Table 6: Comparison of the technological properties (SC, WRC, ORC) and colour parameters of the orange fiber obtained by forced air-microwave drying (Fiber_{MW}) and a commercial citrus fiber (COM).

Technological properties	Fiber _{MW}	COM
SC (mL/ g _{dm})	16.4 ± 1.8 ^a	21.5 ± 1.5 ^b
WRC (g water/ g _{dm})	11.5 ± 0.1 ^a	13.4 ± 0.3 ^b
ORC (g oil/ g _{dm})	1.5 ± 0.3 ^a	1.5 ± 0.2 ^a
L*	90.4 ± 0.0 ^a	89.3 ± 1.8 ^a
a*	0.9 ± 0.0 ^a	-0.2 ± 0.1 ^b
b*	17.5 ± 1.3 ^a	13.4 ± 0.3 ^b

^{a,b}Values. Swelling capacity (SC), Water Retention Capacity (WRC), Oil Retention Capacity (ORC). Data represent means and standard deviation of experiments performed in duplicate.

WRC of the fibre obtained by HAD+MW drying (11.6 ± 1.8 g water/ g_{dm}) was slightly lower than the commercial one (13.4 ± 0.3 g water/ g_{dm}). However, this results are significantly higher than 8 g/g_{dm} reported by Braddock (1999) in orange skin and similar to 11 g water/ g_{dm} reported by Robertson et al. (2000) in citrus pulp. There were no significant differences among fibres with regards to ORC, whereas for SC, commercial fibre swelled 21.5 ± 1.5 mL/ g_{dm} and the citrus fibre obtained in this study swelled 16.4 ± 1.8 mL/ g_{dm}. Nevertheless, swelling and water retention capacity are highly related to particle size distribution which is determined during milling. Lundberg et al. (2014) reported for the same fibre small increases in WRC as particle size decreased, suggesting that grinding may affect matrix structure and surface area of particles, therefore, differences among the fibre ingredients studied might be due to differences on the obtention process that can be further optimized. Results discussed above already showed the potential swelling capacity of the fibre obtained. Bosch-Sierra et al. (2019) carried out a randomised control nutritional intervention with a new orange juice fortified with fibre coming from the same fruit. The juices was obtained by ultra-high pressure homogenization (UHPH). The fibre-enriched orange juice decreased postprandial serum glucose and circulating insulin levels.

Previous studies carried out by Talens, Arboleya, Castro-Giraldez, & Fito, 2017; Talens, Castro-Giraldez, & Fito, 2018, demonstrated that HAD+MW methods not only resulted

in increased moisture reduction but also induced microstructural changes that generated higher sorption capacity.

Process yield was 75 g of fibre / kg of fresh by-products. After 90 min of HAD+MW drying the moisture content was reduced to 10.5 ± 1.7 %. Comparing to conventional drying methods such as HAD at 35 °C, the time needed to reach the target moisture content was doubled (Table 7). MW absorption provokes internal water heating and evaporation, greatly increasing the internal pressure and concentration gradients and thus the effective water diffusion. As a consequence, shorter processing time may be required and higher product quality may be achieved (Maskan, 2000).

Table 7: Time (min) needed to reach different moisture levels (x_w) of orange by-products dried by hot air at 55 °C (HAD) and forced air combined with microwave (MW).

x_w (g _w / 100 g orange by-product)	$t_{process}$ (min)	
	HAD	MW
10	180	90
20	165	81
30	145	71
40	125	63
50	110	55

Energy consumption was estimated as explained in Figure 3. The total energy required for the hot air-microwave drying process described in this work was estimated as the sum of the energy required by hot air (E_{HAD}) and the energy required by microwaves (E_{MW}) per time. E_{HAD} was calculated applying thermodynamics of humid air as explained in (Talens et al., 2017).

DRYING CONDITIONS AND EXPERIMENTAL DATA

T_{amb} φ_{amb} T_D M_0 x_{w_0} $t_{process}$

Figure 3: Drying conditions and experimental data for calculating energy consumption of two different drying processes applied to orange by-products: hit air drying (55 °C) and forced air combined with microwave drying

Figure 4 shows the energy and time required to achieve different moisture contents comparing HAD and HAD+MW. It is clear that energy consumption and time increase continuously with decreasing moisture content (Talens et al., 2017; Talens, Castro-Giraldez, & Fito, 2016; Talens et al., 2018). At the beginning of the treatments, when high water activity produces higher dissipation of microwave energy into heat energy, this is expended in water evaporation, maintaining, in the air, the level of the internal energy (Talens et al., 2016). At the final stages of drying (between 40 and 120 min) water transport is governed by mechanical energy.

Figure 4: Time (●,▲) and energy consumption (○,△) required for drying at different moisture content (x_w) using hot air drying (HAD) and forced air drying coupled with microwaves (MW)

It is possible to observe how HAD+MW drying not only reduced process time by up to 50 % but also reduced energy consumption by up to 26 %. These results are in accordance with Motevali, Minaei, and Khoshtagaza (2011). Motevali et al. (2011) who evaluated energy consumption of different drying methods for pomegranate. These authors showed that the use of microwave for drying decreased drying time and energy consumption in comparison with convection drying. Also, Alibas (2007) found that the optimum energy consumption was obtained when microwave and air-drying was applied simultaneously in pumpkin slices.

Conclusions

Orange by-products were dried by hot air coupled with microwave drying. An aqueous and an ethanolic bioactive extracts were obtained and characterized as rich sources of flavanones, flavanone glycosides and carotenoids typical of citrus plants. Most of these compounds were extracted during the blanching step except for carotenoids which were extracted during decolouring. A new dietary fibre ingredient with antioxidant properties has been developed. Particle size distribution showed an interesting swelling capacity

when fibre was rehydrated. Colour and technological properties of the ingredient obtained were of similar quality than commercial citrus fibre. HAD+MW drying reduced process time (50%) and energy consumption (26%) when compared to hot air drying. Therefore, the process developed represented a new sustainable alternative for citrus by-products valorisation and transformation into value-added ingredients.

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