Plant Foods Hum Nutr (2016) 71:294–300
DOI 10.1007/s11130-016-0559-0

ORIGINAL PAPER

Extractable and Non-Extractable Phenolics and Antioxidant Capacity of Mandarin Waste Dried at Different Temperatures

Francisco J. Esparza-Martínez[1] · **Rita Miranda-López**[1] · **Sara M. Mata-Sánchez**[2] · **Salvador H. Guzmán-Maldonado**[3]

Published online: 1 July 2016

Abstract The mandarin industry is generating more waste due to the increasing demand for juice. In this study, extractable and non-extractable phenolics as well as 2,2-diphenyl-1-picrylhydrazyl (DPPH), 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS), ferric reducing ability of plasma (FRAP), and oxygen radical absorbance capacity (ORAC) antioxidant activities in Satsuma mandarin waste dried at different temperatures were determined. The amounts of non-extractable total phenols, total flavonoids, and condensed tannins measured in mandarin waste dried at 120 °C were 39.4, 44.3, and 45.6 %, respectively, which were higher than those of fresh-mandarin waste. Dried mandarin waste is rich in extractable and non-extractable hesperidin (259.86 and 182.52 mg/g, respectively) and eriocitrin (85.12 and 197.24 mg/g, respectively), as well as non-extractable gallic acid (36.08 μg/g). The antioxidant capacities of extractable and non-extractable phenolics, from the highest to the lowest, were ABTS > ORAC > DPPH > FRAP and ORAC > ABTS > DPPH > FRAP, respectively. The information reported here may encourage mandarin industry operators to re-evaluate their by-products, extending the application of mandarin fruits and reducing waste.

✉ Salvador H. Guzmán-Maldonado
shoraciogm@gmail.com

[1] Posgrado de Ingeniería Bioquímica, Tecnológico de Celaya, Antonio García Cubas Pte. No. 600 esq. Av. Tecnológico, C.P. 38010 Celaya, Guanajuato, México

[2] Chemical Department, Brigham Young University, C100 BNSN, Provo, UT 84602, USA

[3] Laboratorio de Alimentos, Campo Experimental Bajío (INIFAP), Km. 6.5 Carretera Celaya-San Miguel de Allende, C. P. 38110 Celaya, Guanajuato, México

Keywords Mandarin waste · Phenolics · Antioxidant capacity · Drying temperature

Abbreviations

DF	Dietary fiber
EPP	Extractable polyphenols
MW	Mandarin waste
NEPP	Non-extractable polyphenols

Introduction

Citrus fruits are one of the most important horticultural crops in the world. Oranges, lemons, grapefruit and mandarins represent approximately 98 % of the entire industrialized crops. Thirty-four percent of citrus fruit is used to produce juice. The process generates an estimated 15×10^6 tons of waste *per* year, creating a serious environmental problem [1]. Citrus waste consists of the peels (albedo and flavedo), seeds, and pulp (including carpellary membranes) that remain after the juice extraction. Chemical characterization of juice and citrus waste has been mainly focused on oranges and lemons. However, mandarin has attracted attention in recent years due to the increasing demand for its juice and segments. Few groups have sought to determine the phenolic composition and antioxidant capacities of mandarin waste. These studies have focused on mandarin peels [2–5].

Dietary fiber (DF) and polyphenols are addressed separately as non-related compounds, probably because of substantial differences in their chemical and biological properties [6]. The literature data on food polyphenols address only extractable polyphenols (EPP), ignoring the significant fraction of polyphenols that remains in the residues, the so-called non-extractable polyphenols (NEPP) [6, 7]. On the other hand, the drying

temperature could affect EPP and NEPP content and antioxidant capacity of citrus peels [8–10]. For instance, heat treatment may release some low molecular weight phenolic compounds, increasing the antioxidant capacities [11]. The aim of the present study was to evaluate the effect of drying temperature on the extractable and non-extractable phenolic composition and antioxidant capacity of the whole mandarin waste. This information will be of great value to the industry when it turns its attention to such residues.

Materials and Methods

Materials Satsuma (*C. reticulata* Blanco) mandarin fruits (seedless variety) were harvested in November 2014 at El Marquez, Queretaro, Mexico and sanitized (aqueous HCl, 0.05 %) before processing. Mandarins were cut into two pieces and squeezed on a Hamilton Beach electric juice squeezer (Hamilton, China) to produce juice and mandarin waste (albedo, flavedo, pulp residues and carpellary membranes), known hereafter as MW. One batch of MW was freeze-dried (fresh-MW). Three other batches were dried at 60, 90 and 120 °C (MW-60, MW-90, and MW-120) in a tray dryer (UOP 8 Tray Dryer, Armfield, England) equipped with controls for temperature and airflow velocity. Dehydration lasted until a moisture content of ~4.5 % was achieved. The dried samples were ground, sieved (40 mesh) - and stored at −20 °C until analyses were performed.

Soluble and Insoluble Fiber Dietary fibers were assessed according to Manthey *et al.* [12].

Extractable Polyphenols Total phenols (TP) of wastes were determined by the Folin–Ciocalteu spectrophotometric method [13]. Total flavonoids (TF) and condensed tannins (CT) were determined according to Oomah *et al*. [14] and Deshpande *et al.* [15], respectively, in the methanolic extracts of MW. Simple phenolics were quantified through an HPLC-DAD [16]. The mandarin waste residue (MWR) was reserved for extraction of NEPP.

Non-Extractable Polyphenols The MWR was hydrolyzed with 10 mL of methanol/H_2SO_4 (90:10, *v*/v), which is required to release NEPP [7]. Phenolic compounds in the supernatant were determined using the same methods already described for extractable polyphenols.

Antioxidant Capacities ABTS, ORAC, DPPH, and FRAP assays were used to determine the antioxidant capacities of EPP and NEPP extracts [17–20]. Antioxidant capacities were reported as micromoles of Trolox equivalent *per* g sample dry weight (μM TE/g DW).

Statistical Analysis A completely randomized design was used. All data were reported as the means ± SD of three replicates *per* treatment ($n = 3$) for each chemical determination. Statistical analyses were performed using JMP.5.0.1 software (A Business Unit of SAS, 1989–2003, SAS Inst. Inc., NC, USA). Differences among treatment means were tested for significance by using analysis of variance (ANOVA) procedures and the Tukey test at a 0.05 level of significance.

Results and Discussion

Extractable and Non-Extractable TP, TF, and CT The amounts of analyzed extractable and non-extractable phenolics increased as the drying temperature increased (Table 1). For example, when the MW was dried at 120 °C (MW-120), increments of 18.8, 28.3, and 180 % in extractable TP, TF, and CT were detected, respectively, compared to those of fresh-MW. Additionally, the amounts of non-extractable TP, TF, and CT detected in MW-120 were 39.4, 44.3, and 45.6 % higher, respectively, compared to those of fresh-MW. Seok-Moon *et al.* [11] and Chen *et al.* [10] reported significant increments in extractable TP and TF when Satsuma mandarin and orange peels were dried at 100 and 150 °C. The formation of phenolic substances during the drying process may be due to the availability of precursors of phenolics by non-enzymatic inter-conversion between phenolic molecules [21].

The amount of extractable TP in fresh-MW (18.26 mg GAE/g DW) was lower than the range reported by Zhang *et al.* [2] for mandarin peel (31.09 to 51.14 mg/g), while TF content in this report (10.03 mg ER/g, DW) was in the range reported by Zhang *et al.* [2] (6.28 to 20.66 mg/g DW). The discrepancy in TP amounts probably reflects the difference in analysis between the two studies: the samples in Zhang *et al.*'s report were from wild species of fruit, and peels were the only mandarin structure analyzed. The samples in our study, however, included peel, carpellary membranes, and residues of pulp. The sum of non-extractable TP, TF, and CT of each sample was higher compared to the sum of extractable TP, TF, and CT of the corresponding sample (Table 1). For example, the amounts of non-extractable phenolics were from 51.9 % (in fresh-MW) to 64.5 % (in MW-120) higher than those of extractable phenolics.

Phenolic Acids, Flavonoids and Flavanones In this study, 17 extractable and non-extractable simple phenolics were identified, including 10 phenolic acids, three flavonoids, and four flavanones (Table 2). Of these compounds, hesperidin and eriocitrin were found to be the major flavonoids. For example, extractable and non-extractable hesperidin represent 78.8 and 35.7 %, respectively, of the sum of simple phenolics in fresh-MW, while extractable and non-extractable eriocitrin represent 11.9 and 38.6 %, respectively, of the

Table 1 Extractable and non-extractable polyphenols of mandarin waste

Treatment	Total phenols (mg GAE[a]/g DW[b])	Total flavonoids (mg RE[c]/g DW)	Condensed tannins (mg CE[d]/g DW)	Total
Extractable				
Fresh-MW[e]	18.26 ± 0.29 e	10.03 ± 0.46 d	0.44 ± 0.01 f	28.73
MW-60[f]	17.34 ± 1.01 e	10.19 ± 0.36 e	0.43 ± 0.01 f	27.96
MW-90[f]	19.48 ± 0.62 d	11.07 ± 0.56 d	0.69 ± 0.01 e	31.24
MW-120[f]	21.69 ± 0.91 d	12.87 ± 1.07 c	1.23 ± 0.04 d	35.79
Non-extractable				
Fresh-MW	45.15 ± 0.81 c	12.01 ± 0.15 c	2.59 ± 0.11 c	59.75
MW-60	47.47 ± 1.03 bc	11.96 ± 0.23 c	3.18 ± 0.28 b	62.61
MW-90	49.04 ± 2.15 b	15.68 ± 0.16 b	3.48 ± 0.08 b	68.20
MW-120	74.56 ± 0.73 a	21.56 ± 0.24 a	4.76 ± 0.12 a	100.88

Means in the same column with a common letter are not significantly different ($p < 0.05$, Tukey)

[a] Gallic acid equivalents

[b] Dry weight

[c] Rutin equivalent

[d] Catechin equivalent

[e] Mandarin waste

[f] Mandarin waste dried at 60, 90 and 120 °C

sum of simple phenolics in fresh-MW. Extractable and non-extractable hesperidin represent up to 60.7 and 46.2 %, respectively, of the sum of simple phenolics in dried MW (MW-60). Hesperidin contents reported here are by far higher than those reported by Xi *et al.* [3] (6.67–22.13 mg/g, DW) for mandarin pulp. Non-extractable gallic acid and catechin were surprisingly higher compared to levels detected in the extractable profile. Extractable and non-extractable simple phenolics identified in the present study are within the list of those detected in mandarin pulp [2, 3], peel [2] and fresh fruit [22].

Variation patterns of extractable and non-extractable phenolic acids content largely suggest that the drying temperature affects phenolic acids differentially. For example, six of the nine extractable phenolic acids were not detected in MW-120. Extractable syringic acid was not detected in fresh-MW and MW-60 samples, but it was detected in MW-90 and MW-120 samples. Only four non-extractable phenolic acids were identified, compared to the eight extractable phenolic acids detected. On the other hand, the extractable flavanones, eriocitrin and hesperidin, decreased by up to 50.4 % (MW-60) and 40.2 % (MW-120), respectively, in dried samples compared to fresh-MW.

A similar pattern was detected in non-extractable ellagic and gallic acids. Extractable naringin and naringenin gradually increased in MW-60 and MW-90 samples and eventually decreased in the MW-120 sample. This effect was also observed in non-extractable eriotricin, hesperidin, naringin, and naringenin (Table 2). Variation patterns on phenolic acids content in samples heated at different temperature were also reported by Xu *et al.* [8] in huyou [*Citrus sinensis* (L.) × *C. grandis* (L.)] fruit peel. Moreover, Chen *et al.* [10] reported that at 100 °C drying temperature, extractable naringin content was significantly higher than in samples dried at 50, 60, 70, 80, and 90 °C as well as in the fresh sample. Such results are similar to the present study. The lower contents of some non-extractable simple phenolics compared to the extractable in this study agree with results reported by Xi *et al.* [3]. As can be seen in Table 2, the sum of non-extractable simple phenolics was higher than the sum of extractable ones in dried samples. Xi *et al.* [3] reported that the extractable phenolic acids were lower than the non-extractable in pulp, which is contrary to our results. A reason for this difference is that those authors did not detect gallic acid in its study. It was previously discussed that non-extracted gallic acid content was by far higher compared to the rest of the extracted and non-extracted phenolic acids detected in the present work.

Antioxidant Capacities The DPPH assay has been widely used for the determination of primary antioxidant capacity. DPPH radical content could be decreased by reactions with antioxidant compounds that can donate hydrogen. The DPPH values for extractable and non-extractable phenolics ranged from 71.27 to 161.04 and from 333.43 to 351.55 (μM TE/g, DW), respectively (Table 3). DPPH levels of EPP increase as the temperature is increased. However, DPPH values of EPP in dried MW were lower compared to those of EPP in fresh-MW. Reports indicate that DPPH capacity in orange peels increases as temperature [10] and heating time increase [9].

NEPP of MW-120 showed a 5.2 % higher DPPH value compared to that of fresh-MW, MW-60 and MW-90. As seen

Table 2 Extractable and non-extractable phenolic acids, flavonoids and flavanones content of mandarin waste

Simple phenolic		Treatment		
	Fresh-MW[a]	MW-60[a]	MW-90[a]	MW-120[a]
Extractable phenolic acids (μg/g)				
Benzoic	4.19 ± 0.21 c	4.36 ± 0.28b c	4.75 ± 0.11 b	5.55 ± 0.09 a
Chlorogenic	1.59 ± 0.04 b	0.63 ± 0.02 d	1.87 ± 0.03 a	1.21 ± 0.01 c
Ferulic	2.33 ± 0.04 a	0.73 ± 0.01 b	2.34 ± 0.06 a	ND
Gallic	1.41 ± 0.07 b	1.39 ± 0.02 b	11.72 ± 0.24 a	ND
4-hydroxibenzoic	0.56 ± 0.02 b	0.62 ± 0.02 a	ND	ND
Protocatechuic	ND	ND	0.82 ± 0.02 a	ND
Sinapic	1.11 ± 0.05 a	0.47 ± 0.01 c	0.53 ± 0.01 b	ND
Syringic	ND	ND	2.34 ± 0.06 b	2.49 ± 0.03 a
Vanillic	0.33 ± 0.01 b	0.42 ± 0.02 a	ND	ND
Extractable flavonoids (μg/g)				
Catechin	0.84 ± 0.06 a	0.82 ± 0.01 a	0.94 ± 0.09 a	0.36 ± 0.01 b
Epicatechin	1.73 ± 0.12 a	0.51 ± 0.03 d	0.71 ± 0.02 c	0.98 ± 0.01 b
Ecatechin gallate	0.32 ± 0.01 d	0.48 ± 0.04 c	1.42 ± 0.01 b	1.58 ± 0.01 a
Extractable flavanones (mg/g)				
Eriocitrin	85.12 ± 1.09 a	42.23 ± 1.89 c	43.17 ± 1.99 c	52.23 ± 1.87 b
Hesperidin	259.86 ± 11.18 a	181.67 ± 9.68 b	177.93 ± 7.63 b	155.37 ± 5.97 c
Naringin	45.47 ± 0.87 c	57.07 ± 2.13 b	65.58 ± 3.21 a	54.87 ± 3.77 b
Naringenin	5.12 ± 0.09 b	7.86 ± 0.29 a	8.07 ± 0.33 a	5.41 ± 0.31 b
Total	409.98	299.26	321.48	279.05
Non-extractable phenolic acids (μg/g)				
Benzoic	1.05 ± 0.07 b	0.51 ± 0.01 d	0.76 ± 0.06 c	1.19 ± 0.04 a
Ellagic	5.12 ± 0.31 a	4.99 ± 0.23 a	4.85 ± 0.28 a	4.87 ± 0.29 a
Gallic	36.09 ± 2.36 a	28.85 ± 1.71 c	27.98 ± 2.21 c	32.51 ± 0.41 b
Protocatechuic	0.76 ± 0.05 c	1.09 ± 0.05 b	1.22 ± 0.09 ab	1.27 ± 0.08 a
Non-extractable flavonoids (μg/g)				
Catechin	26.96 ± 1.01 b	32.84 ± 2.39 a	30.96 ± 2.21 a	33.89 ± 1.31 a
Epicatechin gallate	0.49 ± 0.02 b	0.68 ± 0.03 a	0.45 ± 0.02 b	0.34 ± 0.02 c
Nonextractable flavanones (mg/g)				
Eriocitrin	197.24 ± 6.07 b	187.12 ± 1.09 b	241.38 ± 10.89 a	152.13 ± 8.97 c
Hesperidin	182.52 ± 4.98 b	202.53 ± 14.91 a	208.31 ± 12.73 a	161.72 ± 9.67 c
Naringin	54.69 ± 1.44 b	75.41 ± 3.33 a	76.85 ± 4.91 a	53.72 ± 2.89 b
Naringenin	6.45 ± 0.13 b	4.48 ± 0.19 c	8.17 ± 0.41 a	4.54 ± 0.28 c
Total	511.35	438.50	600.93	446.18

Means in the same row with a common letter are not significantly different ($p < 0.05$, Tukey)

[a] See Table 1

in Table 3, DPPH antioxidant capacity of NEPP was substantially higher compared to that of EPP. Levels of DPPH antioxidant capacity detected in this work for EPP were much higher compared to those reported in mandarin peels (25.14 to 50.46 μM TE/g DW) [2] and pulp (9.10 to 19.75 μM TE/g DW) [3]. Differences in measured antioxidant capacities between those studies and our work are perhaps due to the mandarin cultivars and structures analyzed.

The ABTS method is commonly used to study the antioxidant capacity of plants based on the capacity to scavenge the radical cation $ABTS^{+}$ generated in the system [23]. The ABTS values of EPP for fresh and dried MW ranged from 310.38 to 574.71 μM TE/g DW, while ABTS levels of NEPP were from 307.85 to 549.55 μM TE/g DW (Table 3). The higher ABTS value was found in the MW-120 sample, both for EPP and NEPP. ABTS levels of EPP increased as temperature increased. These results were not observed in NEPP because the ABTS capacity decreased at 60 and 90 °C and then increased at 120 °C. Levels of ABTS antioxidant capacity detected in this work for EPP were significantly higher

Table 3 Antioxidant capacities (μM Trolox equivalent/g DW) of extractable and non-extractable phenolics of mandarin waste

Treatment	DPPH[a]	ABTS[b]	FRAP[c]	ORAC[d]
Extractable				
Fresh-MW[e]	161.04 ± 0.19 c	342.21 ± 2.38 d	81.91 ± 0.53 a	261.83 ± 15.26 f
MW-60[e]	71.27 ± 3.14 f	310.38 ± 15.35 e	43.08 ± 0.59 c	295.88 ± 14.51 e
MW-90[e]	106.38 ± 2.55 e	447.07 ± 14.17 b	49.69 ± 0.79 b	240.67 ± 17.53 f
MW-120[e]	140.82 ± 2.72 d	574.71 ± 19.25 a	50.34 ± 0.63 b	196.02 ± 7.91 g
Nonextractable				
Fresh-MW	332.89 ± 7.72 b	427.31 ± 12.62 b	3.15 ± 0.01 g	639.76 ± 14.75 b
MW-60	333.71 ± 8.27 b	307.85 ± 19.36 e	4.63 ± 0.02 f	670.31 ± 5.05 a
MW-90	333.43 ± 9.32 b	364.67 ± 15.88 c	6.34 ± 0.09 e	619.14 ± 15.81 c
MW-120	351.55 ± 3.73 a	549.55 ± 19.54 a	10.84 ± 0.52 d	589.63 ± 13.78 d

Means in the same column with a common letter are not significantly different ($p < 0.05$, Tukey)

[a] 2,2-diphenyl-1-picrylhydrazyl

[b] 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid)

[c] Ferric reducing ability of plasma

[d] Oxygen radical absorbance capacity

[e] See Table 1

compared to those reported for mandarin peels (65.62 to 108.06 μM TE/g DW) [2] and pulp (19.79 to 34.66 μM TE/g DW) [3]. The change trend was similar to that of the DPPH scavenging effect. Xu *et al.* [8] showed that the $ABTS^{+}$ scavenging effect of the huyou peel extract increased with heating time and temperature.

The FRAP method is commonly applied to measure the capacity of the sample to reduce ferric complexes to the ferrous form. FRAP values of EPP were notably higher compared to FRAP for NEPP (Table 3). For example, FRAP values for EPP were from 7.8- to 26-fold higher compared to FRAP values for NEPP phenolics. On the other hand, as temperature increase FRAP values increased for both EPP and NEPP. Levels of FRAP antioxidant capacity detected for EPP of dried MW were similar to FRAP values for mandarin peel (40.47 to 46.98 μM TE/g DW) [2], but they were higher compared to those reported by Xi *et al.* [3] in mandarin pulp (19.28 to 34.62 μM TE/g DW). Xu *et al.* [8] reported a reduction of FRAP activity after high temperature treatment of citrus peel.

The ORAC assay is also used to determine the hydrogen atom transfer of biological samples to oxygen radicals. ORAC levels of EPP and NEPP were higher in MW-60 than in the fresh-MW extract, but they were lower in MW-90 and MW-120 extracts (Table 3). Similar to DPPH and ABTS, ORAC levels of NEPP were quite high compared to those of EPP. Levels of ORAC values of EPP presented in this study are in the range reported by Xi *et al.* [3] (126.9–407.27 μM TE/g DW) in mandarin pulp and lower than those reported by Zhang *et al.* [2] in mandarin peel (395.66–748.21 μM TE/g DW).

Soluble and Insoluble Fiber When soluble fiber content of fresh-MW was compared to those of MW-60, MW-90, and MW-120, a decrement of 4, 14, and 20 %, respectively, was detected (Table 4). No differences were identified for insoluble fiber among MW samples. Drying temperature did not affect the total dietary fiber of MW obtained from the Satsuma variety. Mandarin waste reported here showed higher total dietary fiber (43.01–44.09 g/100 g, DM) compared to that of orange (41.6 g/100 g, DM), lemon (39.2 g/100 g, DM), and mandarin (27.9 g/100 g, DM) peels [24]. On average, the total dietary fiber content of both fresh-MW and dried MW was 3.4- and 3.1-fold higher than the value reported by Gorinstein *et al.* [25] for orange and lemon peels, respectively. The higher fiber content of MW reported here compared to that by Gorinstein *et al.* [25] could be explained by the fact that, as was mentioned previously, the MW in this study also included the albedo, flavedo, pulp residues and carpellary membranes.

On the other hand, dry mango peels, grape peels, grape seeds, and pineapple shells have shown lipid oxidation and hypercholesterolemic properties [6]. Additionally, the

Table 4 Soluble, insoluble and total dietary fiber (%) of mandarin waste

Treatment	Soluble fiber	Insoluble fiber	Total
Fresh MW[a]	8.84 ± 0.09 a	34.73 ± 1.32 a	43.57 ± 1.42 a
MW-60[a]	8.48 ± 0.11 b	35.61 ± 2.45 a	44.09 ± 2.53 a
MW-90[a]	7.78 ± 0.35 c	35.76 ± 2.84 a	43.55 ± 2.81 a
MW-120[a]	7.09 ± 0.59 c	35.92 ± 3.23 a	43.01 ± 3.08 a

Means in the same column with a common letter are not significantly different ($p < 0.05$, Tukey)

[a] See Table 1

biological effect of antioxidant dietary fiber rich in EPP and NEPP has been abundantly reported in the literature. Therefore, biological effects of MW studies need to be considered, and they are pending for evaluation.

Conclusions

To our knowledge, this is the first report that addresses mandarin wastes including albedo, flavedo, carpellary membranes, and pulp residues that survive juice extraction. We report here the outstanding fiber and phenolic contents as well as the high antioxidant capacities of both fresh and dry MW. According to the results for antioxidant activities, it is very likely that some interactions between components may be taking place during heat treatment: some compounds are liberated and others are produced by interaction among components [10, 26]. Subsequently, changes in the structure of flavonoids during heat treatment may affect the antioxidant activities of extracts [10]. However, the details of the reactions need further investigation.

MW could be considered as mandarin antioxidant dietary fiber (M-ADF), considering the fiber contents, the antioxidant capacity, and the high levels of extractable and non-extractable eriocitrin, hesperidin, naringin, naringenin and gallic acid. These characteristics suggest many potential applications of M-ADF in the development of low-calorie foods, dietetic drinks, breakfast cereals, dairy products, and pastry bakery products rich in dietary fiber containing high amounts of associated bioactive compounds. The potential use of the agro-industrial by-products has been addressed only slightly by the scientific community [27, 28]. The information reported here may stimulate mandarin operators to re-evaluate their by-products formed from juice production, extending the application of mandarin fruits while reducing waste generation.

Acknowledgments Esparza-Martinez thanks CONACyT for his doctoral scholarship.

Compliance with Ethical Standards

Conflicts of Interest The authors declare that any of us have a conflict of interest.

Human and Animal Rights This article does not contain any studies with human or animal subjects.

References

1. Marin FR, Soler-Rivas C, Benavente-García O, Castillo J, Pérez-Alvarez JA (2007) By-products form different citrus processes as a source of customized functional fibres. Food Chem 100(2):736–741
2. Zhang Y, Sun Y, Xi W, Shen Y, Qiao L, Zhong L, Ye X, Zhou Z (2014) Phenolic composition and antioxidant capacities of Chinese wild mandarin (*Citrus reticulate* Blanco) fruits. Food Chem 145: 674–680
3. Xi W, Zhang Y, Sun Y, Shen Y, Ye X, Zhou Z (2014) Phenolic composition of Chinese wild mandarin (*C. reticulate* Blanco.) pulps and antioxidant properties. Ind Crop Prod 52:466–474
4. Manassero CA, Girotti JR, Mijailovsky S, García de Bravo M, Polo M (2013) *In vitro* comparative analysis of antiproliferative activity of essential oil from mandarin peel and its principal component limonene. Nat Prod Res 27(16):1475–1478
5. Genovese S, Fiorito S, Locatelli M, Carlucci G, Epifano F (2014) Analysis of biologically active oxyprenylated ferulic acid derivatives in citrus fruits. Plant Foods Hum Nutr 69(3):255–260. doi:10.1007/s11130-014-0427-8
6. Saura-Calixto F (2011) Dietary fiber as a carrier of dietary antioxidants: an essential physiological function. J Agric Food Chem 59(1):43–49
7. Arranz S, Silván JM, Saura-Calixto F (2010) Nonextractable polyphenols, usually ignored, are the major part of dietary polyphenols: a study on the Spanish diet. Mol Nutr Food Res 54:1646–1658
8. Xu G, Ye X, Chen J, Liu D (2007) Effect of heat treatment on the phenolic compounds and antioxidant capacity of citrus peel extract. J Agric Food Chem 55(2):330–335
9. Ho SC, Lin CC (2008) Investigation of heat treating conditions for enhancing the anti-inflammatory activity of citrus fruit (*Citrus reticulata*) peels. J Agric Food Chem 56:7976–7982
10. Chen M-L, Yang D-L, Liu S-C (2011) Effects of drying temperature on the flavonoid, phenolic acid and antioxidative capacities of the methanol extract of citrus fruit (*Citrus sinensis* (L.) Osbeck) peels. Int J Food Sci Technol 46:1179–1185
11. Seok-Moon J, So-Young K, Dong-Ryul K, Seong-Chun J, Nam KC, Ahn DU, Seung-Cheol L (2004) Effect of heat treatment on the antioxidant activity of extracts from citrus peels. J Agric Food Chem 52:3389–3393
12. Manthey FA, Hareland GA, Huseby DJ (1999) Soluble and insoluble dietary fiber content and composition of oat. Cereal Chem 76(3):417–420
13. Singleton VL, Orthofer R, Lamuela-Raventos RM (1999) Analysis of total phenols and other oxidation substrates and antioxidants by means of the Folin–Ciocalteu reagent. Methods Enzymol 299:152–178
14. Oomah BD, Cardador-Martínez A, Loarca-Piña G (2005) Phenolics and antioxidative activities in common beans (*Phaseolus vulgaris* L). J Sci Food Agric 85:935–942
15. Deshpande SS, Cheryan M, Salunkhe DK (1986) Tannin analysis of food products. CRC Crit Rev Food Sci Nutr 24(4):401–444
16. Ramamurthy MS, Maiti B, Thomas P, Nair PM (1992) High performance liquid chromatography determination of phenolic acids in potato tubers (*Solanum tuberosum*) during wound healing. J Agric Food Chem 40(4):569–572
17. Re R, Pellegrini N, Proteggente A, Pannala A, Yang M, Rice-Evans C (1999) Antioxidant activity applying an improved ABTS radical cation decolorization assay. Free Radic Biol Med 26(9–10):1231–1237
18. Ou B, Hampsh-Woodill M, Prior R (2001) Development and validation of an improved oxygen radical absorbance capacity assay using fluorescein as a fluorescent probe. J Agric Food Chem 49(10):4619–4626
19. Brand-Williams W, Cuvelier ME, Berset C (1995) Use of a free radical method to evaluate antioxidant activity. LWT-Food Sci Technol 28(1):25–30
20. Iris FF, Benzie IFF, Strain JJ (1996) The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": the FRAP assay. Anal Biochem 239(1):70–76
21. Que F, Mao L, Fang X, Wu T (2008) Comparison of hot air-drying and freeze-drying on the physicochemical properties and

antioxidant activities of pumpkin (*Cucurbita moschata* Duch.) flours. Int J Food Sci Technol 43(7):1195–1201

22. Lu A, Zhang C, Bucheli P, Wei D (2006) Citrus flavonoids in fruit and traditional Chinese medicinal food ingredients. Plant Foods Hum Nutr 61:57–65. doi:10.1007/s11130-006-0014-8
23. Kim DO, Lee KW, Lee HJ, Lee CY (2002) Vitamin C equivalent antioxidant capacity (VCEAC) of phenolic phytochemicals. J Agric Food Chem 50(13):3713–3717
24. Ghanem N, Mihoubi D, Kechaou N, Mihoubi NB (2012) Microwave dehydration of three citrus peel cultivars: effect on water and oil retention capacities, color, shrinkage and total phenols content. Ind Crop Prod 40:167–177
25. Gorinstein S, Martin-Belloso O, Park Y-S, Haruenkit R, Lojek A, Cız M, Caspi A, Libman I, Trakhtenberg S (2001) Comparison of some biochemical characteristics of different citrus fruits. Food Chem 74:309–315
26. Liu SC, Yang DJ, Jin SY, Hsu CY, Chen SL (2008) Kinetics of color development, pH decreasing, and anti-oxidantive activity reduction of Maillard reaction in galactose/glycine model systems. Food Chem 108(2):533–541
27. Salgado JM, Rodrigues BS, Donado-Pestana CM, dos Santos Dias CT, Morzelle MC (2011) Cupuassu (*Theobroma grandiflorum*) peel as potential source of dietary fiber and phytochemicals in whole-bread preparations. Plant Foods Hum Nutr 66(4):384–390. doi:10.1007/s11130-011-0254-0
28. McKee LH, Latner TA (2000) Underutilized sources of dietary fiber: a review. Plant Foods Hum Nutr 55:285–304