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New functional ingredient from orange juice byproduct through a green extraction method

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Abstract

The aim of this work was to obtain, by water extraction, antioxidant dietary fiber from orange juice byproducts. Extractions were performed to orange pomace powder (OPP) varying temperature (55, 65, 75°C) and time (30, 45, 60 min). Two fractions (sediment and supernatant) were obtained from the water extractions and extractable polyphenolic content, antioxidant capacity, total carotenoid content (TCC), and bioaccessibility were determined. Results showed that at 75°C and 45 min the best combination of antioxidant capacity for both fractions is obtained. Under this condition, the sediment is a potential source of antioxidant dietary fiber. Supernatant is rich in bioaccessible polyphenols, associated to soluble fiber as majority component of dietary fiber. Moreover, the water extraction increased the bioaccessibility of polyphenols and carotenoids contained in the OPP. Therefore, both fractions are a potential source of functional food ingredients and orange juice byproducts were entirely revalued.

Practical applications

The present study suggests an extraction method for orange juice byproducts. Following the principles of green extraction, the technique proposed consists of a water extraction (free of solvents), does not produce waste and is of easy industrial application. As a result, two potential food ingredients with different functional and technological properties are obtained. Therefore, orange juice byproducts are completely revalued and a solution for the management of these byproducts is presented. In addition, the two functional ingredients obtained in this study are a step in the right direction for the development of new functional food formulations.

1 | INTRODUCTION

Citrus fruits, which includes orange (*Citrus sinensis*), are one of the most popular world fruit crops and are highly consumed worldwide as fresh produce or juice (Rafiq et al., 2016). During citrus industrial processing 50% of the fruit mass is discarded as waste or byproduct (peel, pulp, internal tissues, and seeds) (Sharma, Mahato, Cho, & Lee, 2017). In fact, 24,996.4 thousand tons of citrus were processed globally during the year 2015 (FAO, 2017), which implies 12,498.2 thousand tons of waste produced. These byproducts pose a complex waste disposal problem and additional economic burdens on production (Putnik et al., 2017). Citrus fruit byproduct may be best used as a substrate for the extraction of its fiber, and biologically active compounds such as phenolic acids, flavonoids, and carotenoids (Sharma et al., 2017).

Dietary fiber is of increasing nutritional and clinical interest owing to its beneficial effects on health and is being used as an ingredient in a large variety of foods (Oh, Bae, & Lee, 2014). Depending on the chemical, physical, and functional properties, dietary fiber can be classified into soluble and insoluble fiber. Soluble dietary fiber

(SDF) includes pectins, gums, inulin-type fructans, and some hemicelluloses whereas insoluble dietary fiber (IDF) includes lignin, cellulose, and some hemicelluloses. SDF is considered to have benefits on serum lipids, lowering the level of serum total cholesterol, while IDF is linked to laxation benefits (Quiles, Campbell, Struck, Rohm, & Hernando, 2016).

Citrus fruit byproducts are a potential source of antioxidant dietary fiber, which can be defined as a product containing significant amounts of natural antioxidants associated with the fiber matrix (Saura-Calixto, 1998). This material combines the physiological properties of both dietary fiber and phenolic compounds and promises to be a potential food ingredient useful in enhancing the bioactive and technological properties of products(Quirós-Sauceda et al., 2014).

Nevertheless, the lack of cost-effective extraction methods for these compounds with the required quality has caused low levels of citrus residues utilization (Putnik et al., 2017). In spite of the high-energy consumption and the large amount of solvents used, often the yield is low (Chemat, Vian, & Cravotto, 2012).

Previous studies of citrus byproducts have centered in extraction techniques which use organic solvents and/or new technologies which are difficult to carry on in an industrial scale (Li, Smith, & Hossain, 2006; Papoutsis et al., 2016; Zia-ur, 2006; Khan, Abertvian, Dangles, & Chemat, 2010; Luengo, Álvarez, & Raso, 2013; Toledo-Guillén, Higuera-Ciapara, De la Fuente, & García-Navarrete, 2010). However, current trends make focus on "green extraction" which is based on the discovery and design of extraction processes which will reduce energy consumption, allows use of alternative solvents (water or agro-solvents) and renewable natural products, and ensure a safe and high-quality extract/product (Chemat et al., 2012).

In recent years, both market and academic research have reported the rising awareness and interest of consumer in health and functional foods (Kaur & Singh, 2017), which are enriched with an ingredient (functional ingredient) able to provide or promote a beneficial action for human health (Quirós-Sauceda et al., 2014).

The in vivo effects of antioxidants depend on their bioaccessibility and bioavailability after ingestion. Only the compounds released from the food matrix and/or absorbed in the small intestine are potentially bioavailable and in conditions to exert their beneficial effects (Palafox-Carlos, Ayala-Zavala, & González-Aguilar, 2011). These can be studied from an in vitro digestive process.

The aim of this work was to obtain antioxidant dietary fiber from orange juice byproducts through water extraction. In this way, by a green extraction method of easy industrial application, there is focus in the development of new functional ingredients and revalorization of the orange juice byproducts.

2 | MATERIALS AND METHODS

2.1 | Orange pomace powder

Orange juice pomace was obtained as a byproduct from industrial juice production (Brown extractor method). Orange pomace was dried in a convection oven at 45°C ± 2°C until the moisture content was less than 10 g/100 g and ground in a laboratory mill (Retsch ZM 200) to obtain a powder particle size of less than 1 mm.

Proximate analysis, extractable polyphenol content (EPC), TCC, and in vitro digestion was done to the orange pomace powder (OPP).

2.2 | Experimental design

Different aqueous extractions were tried to obtain a product that could be considered antioxidant dietary fiber according to Saura-Calixto (1998)from OPP. Experiments were carried out varying two factors: temperature "T" and time "t," in three different levels (Table 1) according to a complete factorial design with the aim to determine the best water extraction treatment. OPP was diluted with water (1:7, m/v) in a centrifuge tube. Then, each tube was placed in a water bath at the extraction temperature (T), with agitation (100RPM), during the extraction time (t).

Each mixture was centrifuged (Thermo Scientific, Sorvall ST-8R) at 9500 RPM for 20 min at room temperature. After centrifugation, the supernatants were separated from the sediment. Finally, each supernatant and sediment was lyophilized (Biobase Bioindustry Shandong Co. Model BKFD 10 PT Capacity 3kg /24 hr). Each experiment was done by duplicate.

To select the best water extraction treatment, antioxidant capacity, EPC, and TCC were measured to the already lyophilized supernatants and sediments.

2.3 | Antioxidants extraction

An extraction of polyphenols from the lyophilized sediments and OPP was needed. The procedure used was proposed by Saura-Calixto (1998). First an extraction with methanol/water (50:50, v/v) at room temperature for 60 min was done. After that, the previous step was repeated with acetone/water (70:30, v/v) as the solvent extract. The supernatants obtained from both extractions were combined and made up to 100 ml with distilled water. The resulting extracts or fractions were used to determine antioxidant capacity (AC) and EPC, by the procedures described below.

2.4 | Antioxidant capacity

Antioxidant capacity was measured using the ABTS radical cation discoloration assay, as proposed by Re et al. (1999) with modifications. A stock solution (2.5Mm) of ABTS was prepared. 2.5 ml of it were mixed with 44 µl of potassium persulfate 140 mM and left standing in the dark for 16 hr for complete formation of ABTS

TABLE 1 Water extraction treatments

Time/Temperature	55°C	65°C	75°C
30 min	Treatment 1	Treatment 2	Treatment 3
45 min	Treatment 4	Treatment 5	Treatment 6
60 min	Treatment 7	Treatment 8	Treatment 9

radical cation. Trolox standard curve was prepared diluting Trolox in ethanol:water (50:50 v/v), from 0 to 2.0 mM final concentration.

The stock solution was diluted with ethanol:water (50:50) until absorbance reached 0.70 (±0.02) at 734 nm. 3 ml of this solution were added to 30 µl of extract or Trolox standard solution and left standing for 30 min in the dark. Absorbance was read at 734 nm in a Shimadzu 1800 UV–Visible spectrophotometer. Results were expressed in mmol of trolox per gram of lyophilized sample.

2.5 | Extractable polyphenol content

The total polyphenol content of lyophilized fractions was determined using Folin–Ciocalteu method (Singleton & Rossi, 1965) modified by Georgé, Brat, Alter, and Amiot (2005). A gallic acid standard curve from 10 to 70 mg/L was prepared. 2.5 ml of Folin–Ciocalteu reagent water dilution (1/10, v/v) was added to 0.5 ml of the different extracts and gallic acid standard solutions. Then, 2 ml of sodium carbonate (75 g/L) were added. Mixtures were left for 30 min in the dark. Absorbance at 760 nm was measured. Results were expressed in mg of GAE per gram of lyophilized or dried sample.

2.6 | Total carotenoid content

The determination of TCC were performed on OPP, lyophilized sediments, and lyophilized supernatants by extraction with methanol (1:50, m/v), after centrifugation at 2,500 RPM during 10 min. The supernatants were separated and their absorbance was measured at 653, 470, and 666 nm. TCC was calculated according to Lichtenthaler and Wellburn (1983).

2.7 | Proximate analyses

Proximate analyses were done to the OPP and to the best lyophilized fractions obtained. Protein and total dietary fiber content (TDF), IDF, SDF were determined using AOAC methods 984.13 and 985.29, respectively (AOAC, 2012). Fat was estimated following the ISO-1999-1999 procedure. Moisture content was determined by gravimetric analysis in a convection oven at 105°C until constant weight. Ash was determined in a muffle furnace following ISO-2002-2002. Total carbohydrates content was obtained by difference.

2.8 | In vitro digestion

To study potential bioaccessibility of the phenolic compounds, an in vitro digestion was carried out on the OPP and the best lyophilized fractions obtained, following the method proposed by Hollebeeck, Borlon, Schneider, Larondelle, and Rogez (2013), which consists of three stages: salivary, gastric, and intestinal. Incubations were performed in closed Erlenmeyer flasks (50 ml), in a shaking water bath at 37°C and 200 rpm. Volumes were adjusted with phosphate buffer saline (PBS) 10 mM, pH 6.9.

For the first step, incubation volume was 10.43 ml, with α-amylase (90 units/ml, 0.43 ml) during was 5 min. For the gastric step, pepsin was added and pH adjusted to 2.0 with HCl 1M. Volume was adjusted to 22.73 ml with PBS; reaction time was 90 min. Finally, pancreatin and bile were added and pH was adjusted to 7.0 with NaHCO3 0.1 M. Volume was completed to 30.09 ml and the sample was incubated for 150 min.

Once the third step had finished, enzymatic reactions were stopped in a water bath at 90°C for 10 min. Samples were centrifuged at 10,000 rpm for 10 min. After separating the supernatant from solid residue, samples were frozen at −80°C and lyophilized.

2.9 | Data analysis

Analyses were performed in triplicate, and all data reported as mean ± *SD*. One way analysis of variance (ANOVA) was performed on each assay, and differences between samples were determined by the Tukey test ($\alpha \leq 0.05$). Pearson's correlation coefficient was calculated for parameters studied in the experimental design. Analyses were performed using XLSTAT Version 2011 (Addinsoft 1995–2010, France).

3 | RESULTS AND DISCUSSION

3.1 | Proximate composition, EPC and TCC of OPP

Orange pomace was dried until it achieved 7.5 ± 0.2 g/100 g of moisture. Proximate composition is presented in Table 3.

Protein content (5.2 g/100 g dwb) resulted to be lower than values found by Nagarajaiah and Prakash, (2016) for orange pomace (8.45 g/100 g dwb). Nagarajaiah and Prakash (2016) indicated orange pomace had the highest protein content, while blue grapes and pineapple pomace had significantly lower protein contents.

Fruits are poor sources of fat: hence, their ether extractives ranged from 1.44 to 2.16 (g/100 g dwb) in fruit pomace (Nagarajaiah & Prakash, 2016). Nevertheless, OPP presented higher lipid content than the fresh fruit (0.91 g/100 g dwb, USDA, 2018) due to pomace high content of seeds rich in fatty acids.

OPP ash content was similar to the one stated by Nagarajaiah and Prakash (2016) for orange pomace (2.65 g/100 g dwb).

As expected, total dietary fiber content (TDF) in OPP was higher than in the fresh fruit (18.11 g/ 100 g dwb, USDA, 2018). OPP is formed principally by citrus peel. Since this byproduct represent the structural matrices, it is mostly comprised of celluloses, hemicelluloses, pectin, and related material (Nagarajaiah & Prakash, 2016; Rafiq et al., 2016).

The TDF of OPP was lower than the TDF of the orange peel powder (63.24 g/100 g dwb) registered by Wang et al. (2015). Such differences owe to OPP higher content of pulp (rich in sugars and pigments) which decreases fiber proportion. Wang et al. (2015) registered a lower content of SDF (13.62 g/100g dwb) probably because of the SDF dialysis purification done. Instead, 23.31 g/100 g dwb of SDF was obtained for citric residues obtained using the "in line" extraction system.

The value of total carbohydrates is lower than the fresh fruit (71.57 g/100 g dwb, USDA, 2018) because most of the nonstructural carbohydrates remain in the juice.

The extractable polyphenol content (EPC) of OPP was 41.7 ± 0.3 mg GAE/ g dwb. Fernández-López et al. (2009) obtained 40.67 ± 0.45 mg GAE/g dwb of extractable polyphenols for high dietary fiber powder from orange juice byproducts.

The TCC of OPP was 43 ± 1 gμ/g (47 ± 2 gμ/g dwb). Different values for this parameter have been registered: 29.87 ± 0.98μg/g (Canan et al., 2016) and 151.57μg/g dwb (Xu, Fraser, Wang, & Bramley, 2006). Such disparity might be caused by the methods used for analysis or by different cultivar and fruit growing conditions.

3.2 | Functional composition of water fractions

The results obtained for trolox equivalent antioxidant capacity (TEAC), EPC, and TCC for each extract are shown in Table 2.

The water extraction treatment temperature influenced the TEAC of both extracts: sediment and supernatant. A significant ($p < 0.05$) moderate Pearson correlation was found between TEAC and treatment temperature. In the case of the supernatant fractions, it was a negative correlation ($r = -0.55$): as the treatment temperature employed increased, the TEAC of supernatants decreased. However, a positive correlation ($r = 0.65$) was obtained for the sediments fractions.

The same correlation trend was found between EPC and treatment temperature. The Pearson correlation coefficient for these two parameters were −0.80 and 0.51 ($p < 0.05$) for supernatant and sediment fractions, respectively. A stronger correlation between EPC and temperature can be appreciate for supernatant than for sediments fractions.

According to these, a lower water extraction temperature favors the polyphenol extraction in the soluble extract. Previous studies suggested that the EPC of water fractions increased with an increase in the temperature of heat treatments (Jeong et al., 2004; Lou, Lin, Hsu, Chiu, & Ho, 2014). However, increasing temperature extraction above certain values may promote possible concurrent degradation of phenolic compounds which were previously mobilized at lower temperature or even the decomposition of residual phenolics remaining in the plant matrix (Mokrani & Madani, 2016).

Alternatively, the TCC presented a significant ($p < 0.05$) and moderate negative correlation with temperature for supernatant ($r = -0.45$) and sediment ($r = -0.32$). The content of carotenoid is decreased in a moisture-dependent manner upon heat and moisture treatment, implying that heating in the presence of moisture affected the carotenoid levels (Beta & Hwang, 2018).

In contrast, the time length of the water extraction treatment did not have strong influence in the TEAC, EPC, and TCC of the fractions as treatment temperature. There was no significant Pearson correlation found between treatment time and TEAC or EPC. Liu and Tsai (2012) found that TEAC value increased as the heating time increased. In the case of TCC, a significant, moderate, and positive correlation ($r = 0.57$) was determined between treatment time and TCC of the sediments. Hence, a slightly better extraction of carotenoids in the sediment fractions can be obtained as the duration of the water extraction treatment increases. Nevertheless, no significant correlation was found between treatment time and TCC of supernatants.

TEAC of OPP was better correlated with EPC than with TCC. The Pearson correlation coefficient between TEAC and EPC was 0.76 and 0.65 for the sediment and supernatant, respectively. In contrast, no significant Pearson correlation between TEAC and TCC was registered. Previous studies also found strong and moderate correlations between antioxidant capacity and polyphenolic content in fruit pomace (Jara-Palacios, Hernanz, Escudero-Gilete, & Heredia, 2014).

Extractable polyphenols and carotenoids exhibited a different distribution between fractions. A higher EPC was obtained in the supernatants, while a higher TCC was obtained in the sediments.

Up to our knowledge, there is no previous study about carotenoid extraction with water. Owing to their hydrophobic nature,

TABLE 2 Trolox Equivalent Antioxidant Capacity (TEAC), Extractable Polyphenol Content (EPC), and Total Carotenoid Content (TCC) of sediments and supernatants obtained from water extraction treatments

			Sediment			Supernatants		
TR	T (°C)	t (min)	TEAC (mmol TE/g)	EPC (mg GAE/g)	TCC (μg/g)	TEAC (mmol TE/g)	EPC (mg GAE/g)	TCC (μg/g)
1	55	30	0.076 ± 0.001^{a}	8.2 ± 0.2^{a}	$46.0 \pm 2^{a,b}$	$0.088 \pm 0.001^{c,b}$	14.2 ± 0.3^{c}	12.4 ± 0.3^{c}
2	65		0.091 ± 0.003^{c}	$9.0 \pm 0.1^{c,b}$	$45.7 \pm 0.5^{a,b}$	0.080 ± 0.002^{a}	9.8 ± 0.1^{a}	$11.9 \pm 0.3^{b,c}$
3	75		$0.088 \pm 0.001^{b,c}$	$8.5 \pm 0.1^{a,b}$	43.3 ± 0.4^{a}	$0.084 \pm 0.003^{a,b}$	$10.4 \pm 0.3^{a,b}$	$11.6 \pm 0.1^{b,c}$
4	55	45	$0.079 \pm 0.001^{a,b}$	8.2 ± 0.3^{a}	51 ± 2^{c}	0.093 ± 0.002^{c}	13.8 ± 0.2^{c}	$10.9 \pm 0.2^{a,b}$
5	65		0.094 ± 0.001^{c}	$8.6 \pm 0.1^{a,b}$	$46.0 \pm 0.8^{a,b}$	$0.084 \pm 0.002^{a,b}$	$10.1 \pm 0.2^{a,b}$	$12.2 \pm 0.4^{b,c}$
6	75		0.097 ± 0.003^{c}	$8.9 \pm 0.1^{c,b}$	$49 \pm 2^{c,b}$	$0.086 \pm 0.001^{a,b,c}$	10.5 ± 0.1^{b}	10.0 ± 0.4^{a}
7	55	60	$0.083 \pm 0.001^{a,b}$	8.1 ± 0.1^{a}	49.9 ± 0.7^{c}	0.092 ± 0.001^{c}	13.9 ± 0.4^{c}	$12.2 \pm 0.1^{b,c}$
8	65		0.095 ± 0.003^{c}	9.3 ± 0.2^{c}	$48.9 \pm 0.4^{c,b}$	$0.085 \pm 0.003^{a,b,c}$	$10.3 \pm 0.1^{a,b}$	12.5 ± 0.6^{c}
9	75		0.091 ± 0.003^{c}	$8.8 \pm 0.3^{c,b}$	$48.0 \pm 0.4^{c,b}$	$0.081 \pm 0.003^{a,b}$	10.5 ± 0.1^{b}	9.8 ± 0.1^{a}

Note. Means within a column which do not share a letter differ significantly ($p > 0.05$).

carotenoids are conventionally extracted using organic solvents (Saini & Keum, 2018). A water extraction method may not facilitate the recovery of carotenoids in the soluble phase.

The use of an organic solvent rather than water may have improved the extraction of carotenoids and polyphenols from OPP. However, the objective of proposing a green extraction method of bioactive compounds would not be accomplished. The present study makes focus in both fractions obtained (supernatant and sediment). In this way, it is possible to obtain two potential food ingredients with different technological applications and rich in bioactive compounds through a water extraction.

In order to choose the best water extraction treatment the TEAC of both fractions was considered. Water extraction treatment number six (75°C, 45 min) was the one which better combined the TEAC of both fractions. TEAC of the supernatant obtained at 75°C and 45 min is statistically equal to the highest TEAC registered for the fractions. By a water extraction treatment at 75°C during 45 min, the microbiological stability is guaranteed for both fractions.

3.3 | Proximate composition of sediment 75°C 45 min (LSE6) and supernatant 75°C 45 min (LSU6)

The moisture content LSE6 and LSU6 was 3.00 ± 0.03 g/100 g and 7.58 ± 0.07 g/100 g, respectively. Proximate composition is shown in Table 3.

The protein content obtained was lower for the fractions than in the OPP. Yi, Wang, Zhuang, Pan, and Huang (2014) also registered a reduction in the crude protein content after different treatments applied to citrus juice byproducts, including water bath treatment.

The higher lipid content of LSE6 with respect to LSU6 is in accord with the distribution of carotenoids between fractions explained above. As carotenoids are lipophilic, they concentrate in LSE6 which presents a higher lipid content.

LSU6 presented the lowest TDF, hence the water extraction treatment proposed in the present study was not as effective in the extraction of dietary fiber as in the extraction of polyphenols in the soluble phase. Nevertheless, the TDF of LSE6 is higher than 50% on a dry matter basis. According to these results and the ones presented in Table 2, it is possible to consider LSE6 as a potential source of antioxidant dietary fiber, with possible applications as a functional ingredient.

Apart from that, the main advantage of dietary fiber from citrus fruits, when compared to other alternative sources, such as cereals, is its higher proportion of SDF (Marín, Soler-Rivas, Benavente-García, Castillo, & Pérez-Alvarez, 2007).This is evident in the IDF/SDF ratio obtained for OPP, LSE6, and LSU6, which are 0.91, 1.26, and 0.16, respectively. Furthermore, IDF/SDF ratios ranging between 0.9 and 1.3 were found for pomegranate peel and it has been shown that ratios from 1 to 2.3 are the most advantageous for the beneficial physiological effects associated with dietary fiber consumption (Hasnaoui, Wathelet, & Jiménez-Araujo, 2014). Additionally, dietary fibers are not only desirable for their nutritional value but also for their functional and technological properties (Marín et al., 2007).

3.4 | In vitro digestion

Health benefits of bioactive compounds depend not only on the intake levels but also on their bioavailability. In vitro methods to simulate gastro-intestinal digestion allow to determine the bioaccessibility of these compounds, as a first step of bioavailability (Cilla, Bosch, Barberá, & Alegría, 2017). Thus, an in vitro digestion was carried out to OPP, LSE6, and LSU6 to estimate the bioaccessibility of polyphenols (Figure 1) and carotenoids (Figure 2) in the different matrices.

LSU6 was the matrix which presented the highest bioaccessibility of polyphenols and carotenoids. These results may be due to its lower content of dietary fiber compare to the dietary fiber content of LSE6 and OPP.

In order to be bioavailable, polyphenols bound to dietary fiber need to be hydrolyzed by enzymes in the upper area of the intestine (Palafox-Carlos et al., 2011). In fact, the generation of functional foods fortified with fiber and phenolic compounds could result in a loss of absorption of the antioxidants, because fiber may trap the antioxidant molecules, decreasing the proposed food functionality (Quirós-Sauceda et al., 2014).

Carotenoids bioaccessibility refers to the fraction of ingested carotenoids that is released from the food matrix and incorporated into micelles during digestion in the gastrointestinal tract (Palmero et al.,

TABLE 3 Proximate composition of orange pomace powder (OPP), sediment 75°C 45 min (LSE6), and supernatant 75°C 45 min (LSU6)

	OPP (g/100 g dwb)	LSE6 (g/100 g dwb)	LSU6 (g/100 g dwb)
Protein	5.2 ± 0.2^{b}	4.5 ± 0.1^{a}	4.0 ± 0.1^{a}
Lipids	1.2 ± 0.1^{b}	2.3 ± 0.1^{c}	0.56 ± 0.02^{a}
Ash	$2.8 \pm 0.1^{a,b}$	2.5 ± 0.1^{a}	3.1 ± 0.1^{b}
TDF[1]	43.0 ± 0.3^{b}	50.7 ± 0.9^{c}	10.20 ± 0.05^{a}
IDF[2]	20.5 ± 0.6^{b}	28.0 ± 0.7^{c}	1.39 ± 0.01^{a}
SDF[3]	22.5^{b}	22.7^{b}	8.81^{a}
Carbohydrates[4]	47.78^{b}	39.93^{a}	82.14^{c}

Note. Means within a row which do not share a letter differ significantly ($p > 0.05$).
[1]Total dietary fiber. [2]Insoluble dietary fiber. [3]Soluble dietary fiber obtained by difference between TDF and IDF. [4]All carbohydrates content obtained by difference.

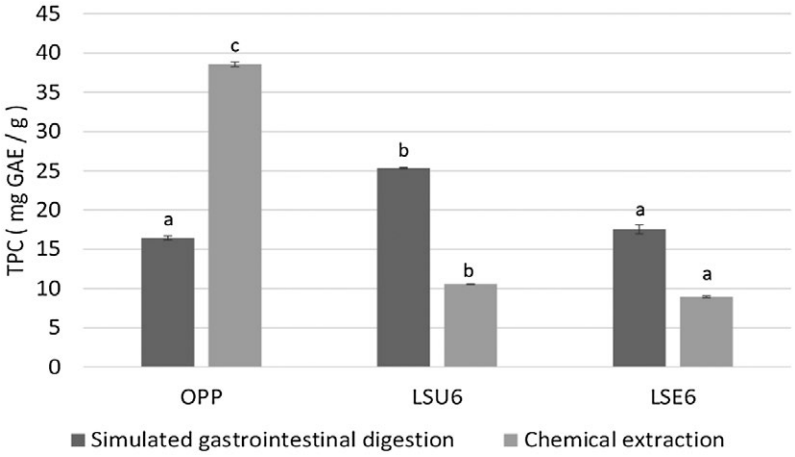

FIGURE 1 Extractable polyphenol content (EPC) of orange pomace powder (OPP), supernatant 75°C 45 min (LSU6), and sediment 75°C 45 min (LSE6) using chemical extraction and after simulated gastrointestinal digestion. Different letters for columns of the same series indicate significant differences ($p > 0.05$) for EPC

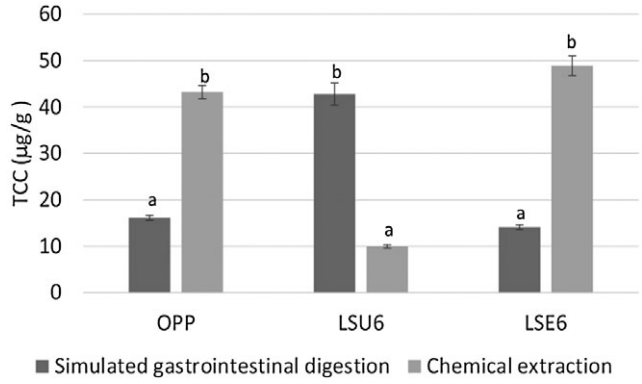

FIGURE 2 Total carotenoid content (TCC) of orange pomace powder (OPP), supernatant 75°C 45 min (LSU6), and sediment 75°C 45 min (LSE6) using chemical extraction and after simulated gastrointestinal digestion. Different letters for columns of the same series indicate significant differences ($p > 0.05$) for TCC

2013). Fiber may entrap the lipids and bile salt molecules, thereby avoiding micelle formation with carotenoids, which may block the passive absorption in the small intestine (Palafox-Carlos et al., 2011). Moreover, Palmero et al. (2013) found that the highest increase in carotenoid bioaccessibility was obtained after transferring carotenoids into an oil phase. So, the lower content of lipids in comparison to dietary fiber in OPP and LSE6 matrices may have hinder the carotenoid's bioaccessibility.

Apart from that, if the EPC and TCC obtained in the simulated gastrointestinal digestion (SGD) and in the chemical extraction (CE) are compared, different patterns are observed. In other words, the EPC was higher in SGD than in CE only for LSU6 and LSE6, while the TCC was higher in SGD than in CE only in LSU6. These may be due to the different morphological states of the matrices which may have facilitated the action of digestive enzymes or not.

In the case of EPC, Gouw, Jung, and Zhao (2017) also registered that total phenolic content from SGD was higher than that from CE, except for dried raspberry pomace. These higher values were probably due to more compounds released from the SGD, as they were subjected to simulated mastication, stomach digestion, and intestinal digestion. Mastication made the cells rupture that allowed the penetration of endogenous compounds, such as enzymes and acids, thus further breaking down the cell walls and releasing the dietary fiber components (Gouw et al., 2017).

OPP was the only matrix which its EPC of SGD was lower than its EPC of CE, what might be due to the lacking of the heat treatment applied during the water extraction. It has been well documented that heating can substantially change the texture of plant tissues, with the modifications being dependent on the composition and structure of the fiber components (Yi et al., 2014), facilitating the action of digestive enzymes. On the other hand, although heat processing increases the carotenoid's bioaccessibility by destroying the integrity of cell wall and membranes of organelles in which carotenoids are located (Cilla et al., 2017), LSE6 did not exhibit a higher carotenoid bioaccessibility than OPP, nor a better release of carotenoids after SGD compared to CE.

As a result, LSU6 was the only matrix which registered a higher TCC in its SGD extract than in the CE. This result may be due to the less amount of carotenoid's natural structural barriers in LSU6, as carotenoids have already been extracted into the soluble phase during the water assisted extraction. In fact, results obtained by Palmero et al. (2013) showed an inverse correlation between the levels of carotenoid bioencapsulation and the carotenoid in vitro bioaccessibility. Differences in cell wall composition and chromoplast substructure among the matrices are important factors determining carotenoid bioaccessibility (Palmero et al., 2013).

4 | CONCLUSION

A water extraction method was proposed, for its easy industrial application, to obtain two innovative food ingredients. These ingredients have different functional and technological applications for the food industry. One of them is soluble and rich in antioxidants, while the insoluble one is also rich in dietary fiber and carotenoids.

The supernatant presented the highest bioaccessibility for polyphenols and carotenoids. By water extraction, the bioaccessibility

of polyphenols and carotenoids present in OPP was increased. The sum of the bioaccessible polyphenols and carotenoids of both water fractions exceeds the bioaccessible polyphenols and carotenoids of OPP. In this way, the entire orange juice byproduct is revalued, without any subsequent waste production.

CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

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