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DIFFERENT GREEN EXTRACTION TECHNOLOGIES FOR SOLUBLE DIETARY FIBRE EXTRACTION FROM ORANGE BY-PRODUCT

Running title: SOLUBLE DIETARY FIBRE FROM ORANGE BY-PRODUCT

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Abstract

The aim of this work was to extract soluble dietary fibre from orange by-product, by testing four different green (non-contaminant, solvent free) extraction technologies: hot water, extrusion + hot water, jet cooker and jet cooker + hot water. Starting from orange pomace, the treatments were assayed and sample was separated in soluble and insoluble fractions. The processing and analysis of the soluble fraction was continued, through subsequent spray drying until obtaining a soluble fibre enriched powder. Powders were analysed: dietary fibre, sugar profile and bioactive characterization (total polyphenol content, antioxidant capacity). Through the application of these green technologies, it was possible to obtain a functional ingredient with soluble dietary fibre from orange by-product. Extrusion + hot water was the treatment that yielded the highest amount of soluble dietary fibre, the lowest content of glucose, sucrose and fructose, and the highest polyphenol content and antioxidant capacity.

Keywords: Green technologies, soluble fibre, orange by-product, antioxidants, extrusion, jet cooker.

1. Introduction

According to FAO, in 2019 approximately 76.3 million tons of oranges were harvested worldwide (FAO, 2021), and almost 17 million tons were destined for industrialization. From these fruits, around 50% w/w (wet weight) is waste, composed of seeds, peels and internal tissue (Wilkins et al., 2007), which means nearly 8.5 million tons were produced. This waste is normally discarded as a residue (landfill, composting), used as animal feed, or as a substrate for pectin extraction (Rafiq et al., 2018; Negro et al., 2017; Sharma et al., 2016). However, these approaches neglect the potential health benefits of this orange by product, mainly due to dietary fibre and bioactive compounds.

Regarding dietary fibre, its definition has been controversial throughout the years, since it cannot be defined as one specific component. However, all definitions include carbohydrate polymers and oligomers that reach the large intestine without being digested. Depending on its water solubility, it can be classified as soluble and insoluble. More recently, several groups have started to include low molecular

weight fibre, which is composed of short chain oligomers (degree of polymerisation 3-9).

In oranges, the proportion of soluble fibre is higher than in other sources of dietary fibre (such as cereals). There is evidence that fibre consumption lowers the prevalence of several diseases (Jones, 2014, Chutkan et al., 2012). Its consumption prevents several non-transmittable diseases, such as lower blood lipid and glucose levels and some types of cancer. Soluble fibre has been found to play a major role in these health benefits (Wang et al., 2015). Unfortunately, there is a worldwide shortage of fibre consumption, therefore a strategy to reverse this situation is to incorporate fibre in food preparation.

Extracting fibre and especially soluble fibre from the orange peel or orange by-product, would allow to obtain a functional ingredient to be used in the supplementation of other foods, in order to close the so-called “fibre gap” (Jones, 2014).

In this context, it is interesting to focus on the characteristics of the extraction processes to be used according to today's consumer trends. Consumers are now becoming more health conscious and demand “clean label” products. Hence, the ingredients to be used should be obtained from natural sources (Elleuch et al., 2011). Moreover, there is an increasing interest in environmental care, demanding for processes that are less contaminant and use less solvents. The commonly named “green” technologies fulfil these demands, as they are solvent-free and non-contaminant. One of the principles of these technologies is the use of water as an alternative solvent (Chemat et al., 2012). Moreover, several solvent free processes have been tried to extract soluble fibre, including ultrasound, extrusion, steam explosion, among others (Arora et al., 2020; Renard, 2018).

Therefore, the aim of this study was to try different green technologies, such as hot water, extrusion and jet cooking, to obtain a new ingredient, with a high content of soluble dietary fibre, from orange pomace.

2. Materials and methods

2.1. Orange pomace

Orange pomace was obtained as a by-product of industrial juice production, supplied by Novacore (Uruguay).

2.2. Treatments for fibre extraction

Four different treatments for fibre extraction (hot water, extrusion + hot water, jet cooking and jet cooking + hot water) were used (Figure 1). They were performed in duplicate, in pilot scale equipment.

2.2.1. Sample preparation

Except for extrusion + hot water extraction, all procedures were performed on wet orange pomace. It was ground in an industrial cutter to particle size of less than 1.0 mm. The resulting product was stored frozen at -18°C on closed polyethylene bags until use.

For extrusion + hot water extraction, orange pomace was dried in a convection oven at 50°C for 72 hours. It was ground in a laboratory mill (Retsch ZM 200) to obtain a particle size of less than 1.0 mm. Powder was stored on polyethylene bags at room temperature until use.

2.2.2. Hot water (HW)

Hot water extraction was performed based on Gutiérrez Barrutia et al. (2019), with modifications. Orange pomace was mixed with water (1:2, m/V) in a mechanically agitated (31 rpm) boiler. Mixture was indirectly heated with steam, and kept at 75°C for 45 minutes. The mixture was cooled and filtered with a cheesecloth. Supernatant and sediment were kept separated.

2.2.3. Extrusion + hot water (EHW)

Extrusion was performed according to Huang & Ma (2016), with modifications. Orange pomace powder's humidity was adjusted to 15% and left overnight at room temperature to reach equilibrium. The slurry was then passed through a single screw extruder (Brabender E330) with barrel temperature 129°C and screw speed of 230 rpm. After grinding the extruded sample to a size of less than 1.0 mm in a laboratory mill (Retsch ZM 200), it was mixed with water (1:16.6, m/v) in a mechanically agitated (31 rpm) boiler. Slurry was indirectly heated with steam, and kept at 75°C for 45 minutes.

2.2.4. Jet cooker (JC)

Jet cooker equipment is composed of a pump, a pipe, a steam injection valve and an exit valve. In this continuous process the sample is pumped through the pipe in

which steam is injected. Procedure was performed according to Felker et al. (2018), with modifications.

Wet orange pomace was mixed with cool water (ratio 1:2.5 respectively m/V). The obtained slurry was passed through the jet cooker, with a residence time in the heating element between 3 and 5 seconds. It was operated using a temperature of 85°C, steam pressure 2.8 bar. Exit pressure was 1.0 bar. After cooling the slurry, it was filtered with a cheesecloth and supernatant and sediment were separated.

2.2.5. Jet cooker + hot water (JCHW)

After going through the jet cooker and before being filtered, the sample passed through the hot water extraction procedure.

2.3. Spray - drying of the soluble fraction

Supernatants from HW, JC, and JCHW, and slurry from EHW were centrifuged (Sigma 6-16 KS) at 9500 rpm for 40 minutes at 4°C. Supernatant and sediment were frozen separately.

Supernatants from all treatments were concentrated under vacuum at 75°C. After reaching 15 ± 2 ˚Bx, they were dried on a Buchi B290 spray dryer. Whey protein isolate (WPI - Provon 292, Glanbia Nutritionals Inc.) was used as an encapsulating agent (8% on total solids basis). Conditions for drying were: 130°C inlet air temperature, atomization air flow rate 414 L/h, liquid feed pump rate 9 mL/min, main drying air flow rate 35 m^3/h. Outlet temperature was 70 ± 4˚C (Edrisi Sormoli & Langrish, 2016).

Powders were stored in laminated pouches at room temperature.

2.4. Proximate analysis of raw material

Orange pomace composition was analysed. Protein was determined using AOAC 984.13 method (AOAC, 2012). Ash was determined following procedure ISO-2002-2002. Fat was estimated following the ISO-1999-1999 procedure. Carbohydrate content was calculated by difference. Insoluble and soluble dietary fibre were determined by AOAC 991.43 method (AOAC, 2012), using the TDF kit from Megazyme.

2.5. Powder characterization

Powder obtained from the four treatments assayed were characterized according to next determinations.

2.5.1. Dietary fibre quantification

Since only supernatants were spray dried, dietary fibre is expected to be from soluble type. Spray dried powders' fibre content (high molecular and low molecular weight dietary fibre, HMWDF and LMWDF, respectively) were analysed by the enzymatic-gravimetric-liquid chromatography method AOAC 2009.01 (AOAC, 2012). This method separates the conventionally measured dietary fibre (insoluble, and soluble dietary fibre, which precipitates with ethanol: water 4:1), which is high molecular weight dietary fibre (HMWDF), from the nonprecipitable soluble dietary fibre of degree of polymerization ≥ 3, which is considered low molecular weight dietary fibre (LMWDF). Megazyme K-ITDF kit was used. D-Glucose from Sigma Aldrich was used as the calibration standard for HPLC. D-sorbitol was used as internal standard, provided by Megazyme kit. A Waters Sugar-Pak® 6.5 x 300 mm column was used. Manual deionization was performed using Ambersep® 200 H and Amberlite® FPA OH^- resins, from Megazyme.

2.5.2. Carbohydrate analysis

Sugar analysis was performed by high performance liquid chromatography, following AOAC 977.20. Fructose, glucose, sucrose, xylose and arabinose were quantified. Standards were of HPLC grade, from Sigma-Aldrich.

2.5.3. Bioactive characterization

2.5.3.1. Extraction for polyphenol content and antioxidant capacity

Polyphenols and antioxidants were extracted with ethanol. Briefly, 50 mg of the sample were mixed with 1.0 mL of 96% ethanol and centrifuged at 4°C for 10 minutes at 10000 rpm. 0.9 mL of the supernatant were removed and stored, and the same amount of 96% ethanol was added. Samples were centrifuged again and supernatants were mixed. Volume was adjusted to 10 mL with ethanol. Extractions were performed in triplicate and all extracts were kept at -20°C until use in glass bottles protected from light.

2.5.3.2. Total polyphenol content

Total polyphenol content was determined by Folin Ciocalteu method. Briefly, 1.0 mL of extract (or standard dilution) was mixed with 6 mL of distilled water and 0.5 mL of Folin-Ciocalteu reagent (ITW Reagents, Spain). Tubes were vortexed and left in the

dark for 3 minutes, when 1 mL of 20% Na_2CO_3 solution and 1.5 mL of water were added. Tubes were once again vortexed and samples were left in the dark at room temperature for ninety minutes. Absorbance was measured at 765 nm in plastic cuvettes. Gallic acid (ITW Reagents, Spain) was used for calibration purposes. Total polyphenol content was expressed as mg of gallic acid equivalents per g of dry matter.

2.5.3.3. Antioxidant capacity

FRAP

Ferric reducing ability of plasma (FRAP) assay was performed following Pulido et al. (2000) procedure, with minor modifications. Briefly, 30 µL of water and 30 µL of sample (ethanol for blank) were mixed with 900 µL of freshly prepared FRAP reagent, and left at 37°C for 30 minutes in the dark. Absorbance was measured at 595 nm after that time. Trolox (Santa Cruz Biotechnology, USA) was used for calibration purposes.

DPPH

Antioxidant activity of the ingredients was also evaluated by DPPH assay, following Shah et al. (2016) method, with modifications. 1 mL of extract was mixed with 0.004% (w/V) freshly prepared DPPH reagent (Sigma Aldrich, USA) in ethanol and left in the dark for 30 minutes at room temperature. Control was prepared with DPPH and solvent (ethanol). Absorbance was read at 517 nm. Trolox (Santa Cruz Biotechnology, USA) was used for calibration purposes.

2.6. Statistical analysis

Analyses were performed in duplicate. Results are expressed as mean ± SD. One way analysis of variance (ANOVA) was performed. Differences between samples were determined by the LSD Fisher test with p-value 0,05. Aiming to study correlation between fibre content, antioxidant capacity, polyphenol count and sugar content, a Pearson correlation test was performed, with p-value 0.01.
All statistics analysis were performed using XLSTAT 2021.2.1 software (Addinsoft 2021, New York, NY, USA).

3. Results and discussion

3.1. Orange pomace composition

Protein content was 3.94 ± 0.09 g/100 g dwb, ash content was 2.61 ± 0.08 g/100 g dwb, and fat content was found 0.73 ± 0.37 g/100 g dwb. Carbohydrates' content was 49.93 g/100 g dwb.

All contents were lower than those reported by Gutiérrez Barrutia et al. (2019) These differences could be due to the ripening stage of the oranges at the moment of harvest, as well as to different varieties of orange in the pool sample.

As Garcia-Amezquita et al. (2018), did not find significant differences on dietary fibre content in orange peels between traditional methodology (soluble and insoluble fibre contents - AOAC 991.43) and integrated methodology (AOAC 2011.25), orange pomace was analysed according to traditional methodology. Total dietary fibre content was 42.76 ± 0.77 g/100 g dwb, from which 26.41 ± 2.91 g/100 g dwb correspond to insoluble dietary fibre and 16.35 ± 2.14 g/100 g dwb to soluble dietary fibre. These results agree with those reported by Gutiérrez Barrutia et al. (2019)

3.2. Effect of treatments in fibre content

Results of high molecular weight (HMWDF), low molecular weight (LMWDF) and total dietary fibre (TDF) are shown on Table 1.

The treatment that yielded the higher amount of total soluble fibre was extrusion + hot water (EHW). This result is in agreement with those reported by bibliography (Huang & Ma, 2016; Redgwell et al., 2011). Extrusion significantly increases the amount of soluble fibre in the sample, by fostering the conversion of insoluble to soluble fibre. This conversion may be due to the mechanical transformation during extrusion that increases the soluble fibre content (Arora et al., 2020). Previous studies (data not shown) showed that the extrusion pre-treatment of the orange pomace powder did not change total dietary fibre, but increased soluble fibre and decreased insoluble fibre.

According to Maphosa & Jideani (2016), soaking modifies the composition and availability of fibres. Also, heat changes the ratio soluble: insoluble fibre. In the case of hot water extraction treatment (HW), results were similar to those reported by Gutiérrez Barrutia et al. (2019), who obtained a fibre content of 10.20 ± 0.05 g/100 g sample dwb in the supernatant of the extraction performed by lab scale.

The use of a jet cooker (JC) did not significantly ($p>0.05$) improve fibre extraction compared to hot water (HW). To this day, no bibliography was found on jet cooker usage for fibre extraction in fruits. Work has been done studying the effect of jet cooking, especially in legumes and cereals (de la Rosa-Millán et al., 2020; Felker et al., 2018). Jet cooking may result in changes in the ratio soluble: insoluble fibre fractions, because of the shear forces that are generated due to the hydrothermal process. These shear forces disrupt the internal structure of the cell walls, increasing soluble fibre (de la Rosa-Millán et al., 2020). Felker et al. (2018) found significant increase in the soluble fibre content when treating pinto beans with the jet cooker. de la Rosa-Millán et al. (2020), also found an increase in soluble dietary fibre when jet cooking chickpea flour. Both authors found that total dietary fibre content did not vary significantly. Several factors may explain the difference between these cases and the orange pomace one. Firstly, they measured the amount of soluble fibre in the whole ingredient, while in our study an extraction was performed and only the supernatant was analysed. Thus, the sediment may still have soluble fibre that was not extracted. Adding the hot water extraction after the jet cooking step (JCHW) significantly ($p<0.05$) improved high molecular weight fibre extraction when comparing to both treatments on their own. This may indicate that keeping the sample at a high temperature would improve the extraction of the linkages that may have been debilitated in the jet cooking process.

No significant differences ($p>0.05$) were found on the amount of low molecular weight dietary fibre extracted by hot water, jet cooking and the combination of both. However, extrusion improved extraction significantly ($p<0.05$) when compared to jet cooking and hot water, but not compared to the combination of both. Although the inclusion of these compounds in the amount of fibre is controversial, they are able to be fermented by the microorganisms in the large intestine, promoting growth and activity of bacteria in the digestive tract (Mudgil, 2017).

3.3. Sugar content

As dietary fibre is composed of saccharides, the content of some sugars (fructose, glucose, sucrose, xylose and arabinose) was assayed in order to check the degree of breakage of fibre. Neither arabinose nor xylose were detected (Figure 2).

The absence of the latter two compounds suggests that rhamnogalacturonans were not degraded in the processes.

Extrusion + hot water (EHW) treatment yielded the least amount of sugars. Lower fructose and sucrose levels were observed by Schmid et al. (2020) in apple pomace when comparing extrusion with the apple pomace itself; they found that these sugars were degraded under thermomechanical stresses.

Jet cooking (JC) and hot water (HW) showed the highest content in all the sugars analysed, with no significant differences ($p>0.05$). Jet cooking + hot water extraction had significantly lower contents ($p<0.05$) than JC and HW in all the sugars analysed. Since temperature fosters Maillard reactions and caramelization, and as JCHW is longer under higher temperatures, sugars could have undergone these reactions and be diminished (Zhang et al., 2019).

Although no bibliography was found regarding jet cooking effect on sucrose, glucose and fructose, Felker et al. (2018) studied the effect of this treatment on raffinose family oligosaccharides, finding an increase on them. This suggests that the treatment of the samples with steam may foment the release of mono and disaccharides from oligosaccharides, making them more extractable.

3.4. Bioactive characterization

Polyphenols and antioxidant compounds are described to be bound to dietary fibre (Rafiq et al., 2018; Tejada-Ortigoza et al., 2015, Marín et al., 2007), and orange pomace is known to have good antioxidant capacity (Gutiérrez Barrutia et al., 2019). Total polyphenols content and antioxidant capacity are shown on Table 2.

Extrusion + hot water (EHW) yielded the powder with the highest polyphenol content and antioxidant capacity, both in FRAP and DPPH analyses. DPPH results were lower than those reported by Hernández-Carranza et al. (2016).

Unlike what was observed by Gutierrez Barrutia et al. (2019), the negative correlation of temperature effect on supernatant antioxidant capacity is not seen in our work, as antioxidant capacity is highest in the treatment with the highest temperature (EHW). This could be due to extrusion breaking fibres and releasing the antioxidant components that cannot not be extracted when they are bounded to the fibre. Also, extrusion's higher temperatures may result in the formation of

Maillard compounds that may have antioxidant capacity, such as reductones and melanoidins (Hafsa et al., 2021; Nooshkam et al., 2019).

Hot water and jet cooking + hot water had no significant differences ($p>0.05$) between them in antioxidant capacity, both DPPH and FRAP. Jet cooking (JC) showed the lowest antioxidant ($p<0.05$) capacity. JC is a continuous process, with a short exposure time. As the main difference between jet cooking and jet cooking + hot water extraction is the time the sample was exposed to temperature, it could indicate that a longer time is needed to release the antioxidants and polyphenols from the matrix, or that these types of compounds are being produced by longer exposures to temperature, as indicated by Liu & Tsai (2012).

For all treatments, lower total polyphenol contents were found when compared to those reported by Gutierrez Barrutia et al. (2019), using water extraction at lab scale. Values for total polyphenol content of orange peels found in bibliography are extremely variable, with values ranging from 0.0139 mg GAE/g to 11.09 mg GAE/g in orange peels (Montero-Calderon et al., 2019).

Considering antioxidants and polyphenols are compounds fruits have to protect against environmental stress (Lado et al., 2018), these differences may be due to climate differences and ripening stages of the fruits.

3.5. Relation between sugar content, fibre content, antioxidant capacity and total polyphenol content

In order to establish correlations between sugar, total fibre contents, antioxidant capacity and total polyphenol content, Pearson correlation's test was performed. Results are shown on table 3.

A high correlation was found on high molecular weight dietary fibre content and polyphenols content ($r = 0.990$, $p<0.01$). Also, total sugar content was negatively correlated with total fibre content ($r = -0.993$, $p<0.01$). This would indicate that when treatments are low in fibre extraction, they exhibit a higher content of glucose, fructose and sucrose. This could imply that the extraction treatments may break dietary fibre linkages more than desired, liberating sugars instead of oligosaccharides chains.

Moreover, a high correlation was found between DPPH and TPC, and FRAP and TPC ($r = 0.997$, $r = 0.995$, respectively, p-value<0.01). These results may indicate

that the antioxidant capacity of the samples is strongly related to the presence of polyphenols.

4. Conclusions

Four different treatments were assayed on orange juice production by-product to obtain a soluble fibre enriched ingredient. Extrusion was the most effective treatment to extract soluble dietary fibre from orange pomace, having also the lowest sugar content. Regarding antioxidant capacity and total polyphenol content, extrusion yielded the highest values, while jet cooking treatment resulted in the lowest. Adding a hot water step after jet cooking increased the antioxidant capacity. This could be related to the formation of Maillard products, as reductones, in the most aggressive treatments (extrusion and jet cooking + hot water). Further investigation is required to elucidate the effect of jet cooking on dietary fibres.

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Data availability statement: The authors confirm that the data supporting the findings of this study are available within the article.

Ethical guidelines: Ethics approval was not required for this research.

References

AOAC. (2012). *Official methods of analysis of AOAC International (19th ed.).* Maryland, USA: Gaithersburg. AOAC International. (2014). *AOAC 2009.01*.

Arora, B., Yoon, A., Sriram, M., Singha, P., & Rizvi, S. S. H. (2020). Reactive extrusion: A review of the physicochemical changes in food systems. *Innovative Food Science and Emerging Technologies*, *64*, 102429. https://doi.org/10.1016/j.ifset.2020.102429

Chemat, F., Vian, M. A., & Cravotto, G. (2012). Green extraction of natural products: Concept and principles. *International Journal of Molecular Sciences*, *13*(7), 8615–8627. https://doi.org/10.3390/ijms13078615

Chutkan, R., Fahey, G., Wright, W. L., & Mcrorie, J. (2012). Viscous versus nonviscous

soluble fiber supplements: Mechanisms and evidence for fiber-specific health benefits. *Journal of the American Academy of Nurse Practitioners*, *24*(8), 476–487. https://doi.org/10.1111/j.1745-7599.2012.00758.x

de la Rosa-Millán, J., Orona-Padilla, J. L., Flores-Moreno, V. M., & Serna-Saldívar, S. O. (2020). Effect of jet-cooking and hydrolyses with amylases on the physicochemical and in vitro digestion performance of whole chickpea flours. *International Journal of Food Science and Technology*, *55*(2), 690–701. https://doi.org/10.1111/ijfs.14338

Edrisi Sormoli, M., & Langrish, T. A. G. (2016). Spray drying bioactive orange-peel extracts produced by Soxhlet extraction: Use of WPI, antioxidant activity and moisture sorption isotherms. *LWT - Food Science and Technology*, *72*, 1–8. https://doi.org/10.1016/j.lwt.2016.04.033

Elleuch, M., Bedigian, D., Roiseux, O., Besbes, S., Blecker, C., & Attia, H. (2011). Dietary fibre and fibre-rich by-products of food processing: Characterisation, technological functionality and commercial applications: A review. *Food Chemistry*, *124*(2), 411–421. https://doi.org/10.1016/j.foodchem.2010.06.077

FAO. (2021). Citrus Fruit Statistical Compendium 2020. Rome. Retrieved from https://www.fao.org/3/cb6492en/cb6492en.pdf.

Felker, F. C., Kenar, J. A., Byars, J. A., Singh, M., & Liu, S. X. (2018). Comparison of properties of raw pulse flours with those of jet-cooked, drum-dried flours. *LWT - Food Science and Technology*, *96*, 648–656. https://doi.org/10.1016/j.lwt.2018.06.022

Felker and collaborators studied the jet cooking process on pulse flours. They found that the jet cooking decreased the amount of insoluble fibre and increased the soluble type. Moreover, they studied the effect of the technology in raffinose family oligosaccharides, finding that the process increased their extractability.

Garcia-Amezquita, L. E., Tejada-Ortigoza, V., Heredia-Olea, E., Serna-Saldívar, S. O., & Welti-Chanes, J. (2018). Differences in the dietary fiber content of fruits and their by-products quantified by conventional and integrated AOAC official methodologies. *Journal of Food Composition and Analysis*, *67*, 77–85. https://doi.org/10.1016/j.jfca.2018.01.004

The authors tried conventional and HPLC approachs for fibre quantification in several fruits and their peels, and got to the conclusion that in the case of orange peel no significant differences were found between the two processes.

Gutiérrez Barrutia, M. B., Curutchet, A., Arcia, P., & Cozzano, S. (2019). New functional ingredient from orange juice byproduct through a green extraction method. *Journal of Food Processing and Preservation*, *43*(5), 1–8. https://doi.org/10.1111/jfpp.13934

The authors carried out hot water extraction at lab-scale to obtain antioxidant dietary fibre. They assayed different times and temperatures and how they affected antioxidant capacity.

Hafsa, J., Smach, M. A., Mrid, R. Ben, Sobeh, M., Majdoub, H., & Yasri, A. (2021). Functional properties of chitosan derivatives obtained through Maillard reaction: A novel promising food preservative. *Food Chemistry*, *349*, 129072. https://doi.org/10.1016/j.foodchem.2021.129072

Hernández-Carranza, P., Ávila-Sosa, R., Guerrero-Beltrán, J. A., Navarro-Cruz, A. R., Corona-Jiménez, E., & Ochoa-Velasco, C. E. (2016). Optimization of Antioxidant Compounds Extraction from Fruit By-Products: Apple Pomace, Orange and Banana Peel. *Journal of Food Processing and Preservation*, *40*(1), 103–115. https://doi.org/10.1111/jfpp.12588

Huang, Y. L., & Ma, Y. S. (2016). The effect of extrusion processing on the physiochemical properties of extruded orange pomace. *Food Chemistry*, *192*, 363–369. https://doi.org/10.1016/j.foodchem.2015.07.039

The authors studied the effect of extrusion on orange pomace composition. They optimized the process by response surface methodolgy, obtaining the optimal conditions for soluble dietary fibre extraction from orange pomace. Moreover, they studied the effect of extrusion on monosaccharide content.

ISO-5984-2002 International Organization for Standardization. (2002). Animal feeding stuffs — Determination of crude ash. Geneva: Switzerland.

ISO-6492-1999 International Organization for Standardization. (1999). Animal feeding stuffs — Determination of fat content. Geneva: Switzerland

Jones, J. M. (2014). CODEX-aligned dietary fiber definitions help to bridge the ‘fiber gap.’ *Nutrition Journal*, *13*(34). https://doi.org/10.1186/1475-2891-13-34

Lado, J., Gambetta, G., & Zacarias, L. (2018). Key determinants of citrus fruit quality: Metabolites and main changes during maturation. *Scientia Horticulturae*, *233*, 238–248. https://doi.org/10.1016/j.scienta.2018.01.055

Liu, S. C., & Tsai, C. W. (2012). Effects of heating time on the antioxidative capacities of

citrus fruit (Citrus sinensis (L.) Osbeck) by-products. *Food Science and Technology Research*, *18*(4), 505–513. https://doi.org/10.3136/fstr.18.505

Maphosa, Y., & Jideani, V. A. (2016). Dietary fiber extraction for human nutrition—A review. *Food Reviews International*, *32*(1), 98–115. https://doi.org/10.1080/87559129.2015.1057840

Marín, F. R., Soler-Rivas, C., Benavente-García, O., Castillo, J., & Pérez-Alvarez, J. A. (2007). By-products from different citrus processes as a source of customized functional fibres. *Food Chemistry*, *100*(2), 736–741. https://doi.org/10.1016/j.foodchem.2005.04.040

Montero-Calderon, A., Cortes, C., Zulueta, A., Frigola, A., & Esteve, M. J. (2019). Green solvents and Ultrasound-Assisted Extraction of bioactive orange (Citrus sinensis) peel compounds. *Scientific Reports*, *9*(1), 1–8. https://doi.org/10.1038/s41598-019-52717-1

Mudgil, D. (2017). The Interaction Between Insoluble and Soluble Fiber. In *Dietary Fiber for the Prevention of Cardiovascular Disease: Fiber's Interaction between Gut Micoflora, Sugar Metabolism, Weight Control and Cardiovascular Health*. Elsevier Inc. https://doi.org/10.1016/B978-0-12-805130-6.00003-3

Negro, V., Ruggeri, B., Fino, D., & Tonini, D. (2017). Life cycle assessment of orange peel waste management. *Resources, Conservation and Recycling*, *127*, 148–158. https://doi.org/10.1016/j.resconrec.2017.08.014

Nooshkam, M., Varidi, M., & Bashash, M. (2019). The Maillard reaction products as food-born antioxidant and antibrowning agents in model and real food systems. *Food Chemistry*, *275*, 644–660. https://doi.org/10.1016/j.foodchem.2018.09.083

Pulido, R., Bravo, L., & Saura-Calixto, F. (2000). Antioxidant activity of dietary polyphenols as determined by a modified ferric reducing/antioxidant power assay. *Journal of Agricultural and Food Chemistry*, *48*(8), 3396–3402. https://doi.org/10.1021/jf9913458

Rafiq, S., Kaul, R., Sofi, S. A., Bashir, N., Nazir, F., & Ahmad Nayik, G. (2018). Citrus peel as a source of functional ingredient: A review. *Journal of the Saudi Society of Agricultural Sciences*, *17*(4), 351–358. https://doi.org/10.1016/j.jssas.2016.07.006

Redgwell, R. J., Curti, D., Robin, F., Donato, L., & Pineau, N. (2011). Extrusion-induced changes to the chemical profile and viscosity generating properties of citrus fiber.

Journal of Agricultural and Food Chemistry, *59*(15), 8272–8279. https://doi.org/10.1021/jf201845b

Renard, C. M. G. C. (2018). Extraction of bioactives from fruit and vegetables: State of the art and perspectives. *LWT - Food Science and Technology*, *93*, 390–395. https://doi.org/10.1016/j.lwt.2018.03.063

Schmid, V., Trabert, A., Schäfer, J., Bunzel, M., Karbstein, H. P., & Emin, M. A. (2020). Modification of apple pomace by extrusion processing: Studies on the composition, polymer structures, and functional properties. *Foods*, *9*(10), 8–10. https://doi.org/10.3390/foods9101385

Shah, B. R., Zhang, C., Li, Y., & Li, B. (2016). Bioaccessibility and antioxidant activity of curcumin after encapsulated by nano and Pickering emulsion based on chitosan-tripolyphosphate nanoparticles. *Food Research International*, *89*, 399–407. https://doi.org/10.1016/j.foodres.2016.08.022

Sharma, S. K., Bansal, S., Mangal, M., Dixit, A. K., Gupta, R. K., & Mangal, A. K. (2016). Utilization of food processing by-products as dietary, functional, and novel fiber: A review. *Critical Reviews in Food Science and Nutrition*, *56*(10), 1647–1661. https://doi.org/10.1080/10408398.2013.794327

Tejada-Ortigoza, V., Garcia-Amezquita, L. E., Serna-Saldívar, S. O., & Welti-Chanes, J. (2015). Advances in the Functional Characterization and Extraction Processes of Dietary Fiber. *Food Engineering Reviews*, *8*(3), 251–271. https://doi.org/10.1007/s12393-015-9134-y

Wang, L., Xu, H., Yuan, F., Fan, R., & Gao, Y. (2015). Preparation and physicochemical properties of soluble dietary fiber from orange peel assisted by steam explosion and dilute acid soaking. *Food Chemistry*, *185*, 90–98. https://doi.org/10.1016/j.foodchem.2015.03.112

Wilkins, M. R., Suryawati, L., Maness, N. O., & Chrz, D. (2007). Ethanol production by Saccharomyces cerevisiae and Kluyveromyces marxianus in the presence of orange-peel oil. *World Journal of Microbiology and Biotechnology*, *23*(8), 1161–1168. https://doi.org/10.1007/s11274-007-9346-2

Zhang, S., Zheng, L., Zheng, X., Ai, B., Yang, Y., Pan, Y., & Sheng, Z. (2019). Effect of steam explosion treatments on the functional properties and structure of camellia (Camellia oleifera Abel.) seed cake protein. *Food Hydrocolloids*, *93*, 189–197. https://doi.org/10.1016/j.foodhyd.2019.02.017

Figure captions

Figure 1 - Processing of the orange pomace for fibre extraction

Figure 2 - Glucose, fructose, sucrose y total sugar content of powders obtained after each extraction treatment. HW: hot water; EHW: extrusion + hot water; JC: jet cooker; and JCHW: jet cooker + hot water. Results are expressed in g/100 g dwb. Bars with different letters in the same group were significantly different ($p<0.05$).

Table 1 – Values (mean ± SD) of powders' high (HMWDF), low (LMWDF) molecular weight and total dietary fibre (TDF). HW: hot water; EHW: extrusion + hot water; JC: jet cooker; and JCHW: jet cooker + hot water

	HMWDF (g/100 g dwb)	**LMWDF (g/100 g dwb)**	**TDF (g/100 g dwb)**
1 - HW	8.00 ± 0.32[a]	1.48 ± 0.56[a]	9.48 ± 0.45[a]
2 - EHW	14.15 ± 0.80[c]	6.05 ± 3.07[b]	20.20 ± 3.69[b]
3 - JC	7.96 ± 1.37[a]	2.29 ± 1.30[a]	10.25 ± 0.89[a]
4 - JCHW	10.01 ± 0.24[b]	3.03 ± 1.57[ab]	13.03 ± 1.8[a]

Mean values in each column followed by different letters were significantly different ($p<0.05$).

Table 2 – Values (mean ± standard deviation) of total polyphenol content and antioxidant capacity of powders obtained after each extraction treatment. HW: hot water; EHW: extrusion + hot water; JC: jet cooker; and JCHW: jet cooker + hot water. GAE = gallic acid equivalent.

	Total polyphenol content (mg GAE/g sample dwb)	**DPPH (µmol Trolox/g sample dwb)**	**FRAP (µmol Trolox/g sample dwb)**
1 – HW	4.34 ± 0.16[b]	11.12 ± 0.37[a]	48.26 ± 1.24[b]
2 – EHW	8.17 ± 0.38[d]	14.36 ± 0.19[b]	89.94 ± 3.25[c]
3 – JC	3.70 ± 0.26[a]	10.67 ± 0.01[a]	36.64 ± 1.55[a]
4 – JCHW	5.14 ± 0.28[c]	11.86 ± 0.78[a]	52.12 ± 2.52[b]

Mean values in each column followed by different letters were significantly different ($p<0.05$).

Table 3 – Pearson correlation table.

	HMWDF	LMWDF	TDF	Glucose	Fructose	Sucrose	Total sugars	TPC	DPPH	FRAP
HMWDF	**1**	0,983	**0,997**	-0,986	**-0,995**	**-0,994**	**-0,993**	**0,990**	0,978	0,971
LMWDF	0,983	**1**	**0,994**	-0,974	-0,984	-0,988	-0,983	0,955	0,938	0,927
TDF	**0,997**	**0,994**	**1**	-0,985	**-0,995**	**-0,996**	**-0,993**	0,980	0,966	0,957
Glucose	-0,986	-0,974	-0,985	**1**	**0,997**	**0,997**	**0,999**	-0,990	-0,989	-0,984
Fructose	**-0,995**	-0,984	**-0,995**	**0,997**	**1**	**1,000**	**1,000**	**-0,991**	-0,985	-0,979
Sucrose	**-0,994**	-0,988	**-0,996**	**0,997**	**1,000**	**1**	**1,000**	-0,988	-0,980	-0,974
Total sugars	**-0,993**	-0,983	**-0,993**	**0,999**	**1,000**	**1,000**	**1**	-0,990	-0,985	-0,979
TPC	**0,990**	0,955	0,980	-0,990	**-0,991**	-0,988	-0,990	**1**	**0,997**	**0,995**
DPPH	0,978	0,938	0,966	-0,989	-0,985	-0,980	-0,985	**0,997**	**1**	**1,000**
FRAP	0,971	0,927	0,957	-0,984	-0,979	-0,974	-0,979	**0,995**	**1,000**	**1**

Values in bold are significantly different from 0 ($p<0.01$)