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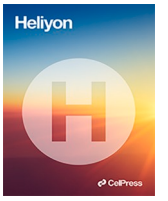

Research article

Upcycling fruit pomaces (orange, apple, and grape-wine): The impact of particle size on phenolic compounds' bioaccessibility

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ABSTRACT

This work aimed to analyse the effect of particle size on bioactive compounds of different by-products. Orange, apple, and grape-wine by-products obtained from industrial production were dried and ground at two sizes: 1 mm and 0.5 mm. Pomaces were analysed in composition (protein, fat, carbohydrates, moisture, and ash contents) and bioactive compounds (total phenol content by Folin– Ciocalteu method and antioxidant capacity by FRAP assay) and submitted to an *in-vitro* digestion. FESEM was used to observe the microstructure of samples. All pomaces showed high fibre content (21.7, 31.2, and 58.9 g/100 g, in apple, orange, and grape pomace respectively). Total phenol content in raw material was higher in grape > orange > apple, with no differences (apple) or slight differences (grape and orange) between 1 mm and 0.5 mm particle size. Grape pomace was observed as a porous, more accessible structure, where extracting polyphenols was easier. Orange pomace', was compact and apple pomace structure was even more compact hindering the raw materials polyphenol extraction. After digestion, total phenol content increased in orange and apple pomace for both particle size. In apple, bioaccessibility of phenolic compounds showed a 5 fold increase for 1 mm sample size and a 4 fold increase for 0.5 mm sample size. In orange, for both sizes bioaccessibility increased but to a lesser extent (2.4 fold). In the case of grape pomace, although polyphenol content decreased after digestion (0.7 fold for both sizes), they showed the highest antioxidant capacity. Regarding the effect of particle size on total polyphenol content and antioxidant capacity, no trend was found in this work for the fruit pomaces studied. In the case of grape and apple, grinding at 1 mm should be adequate regarding antioxidant capacity while in the case of orange, it may be better to use a pomace ground at 0.5 mm.

1. Introduction

High amount of biodegradable waste are generated by the food industry, discarding large quantities of residues with a high biochemical oxygen demand and chemical oxygen demand contents [1]. According to a report published by FAO [2] nearly one-third of worldwide food production for human consumption is lost or wasted. More precisely, along the food supply chains, 54 % of total residues, including production and postharvest, are generated as upstream processes, and 46 % of waste, including processing,

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distribution, and consumption as downstream processes. Concerning production, the juice industry is one of the most relevant, with residues remaining after juice processing that range from 15 % for grapes to 50 % for citrus approximately [3]. During apple juice processing, apple pomace is produced, comprising peel, pulp, seeds, and stems, representing 25–30 % of the original fruit weight [4]. Considering that 11 million tons of apples are processed worldwide per year [5], it turns out that 3.3 million tons of apple pomace are generated annually, which industries must dispose of properly. Another industry that generates a large amount of waste is the winemaking industry. Grape pomace is the primary waste generated during the wine production process, accounting for 30%w/w of the initial grapes used [6].

Agro-industrial residues, better called by-products, as the food that is lost or wasted, are mainly discarded, incurring the industry in costs for its final disposal and negatively impacting the environment. From a circular economy perspective, by-products may be not considered as a waste but as resources to be upcycled, leading to a solution to reduce raw material input and waste production [1].

Searching for new uses for fruit by-products and reducing environmental impact, the functional, nutritional and technological potential of different pomaces has been studied. Agri-food processing residues, with their particular composition, can be sustainably used to produce food, feed, chemicals, and energy [1,7,8].

In many cases, by-products, rich in phytochemicals may be used to enhance the nutritional profile of mass-consumption foods. In the last decade, large scientific literature has shown the potential of pomace as a source of bioactive compounds such as dietary fibers and polyphenols [8–11]. Polyphenols, potents antioxidants, offers numerous health benefits (decrease the risk of cardiovascular diseases, cancer, neurodegenerative diseases, diabetes and osteoporosis) [12–15]. The antioxidant capacity of phenolics extracted from plant materials and its benefits on health and the effect of dietary fiber bounded to antioxidants has been largely studied by Saura-Calixto [16–20].

[21], through an exhaustive review, analysed the emerging trends to handling by-product from fruit and vegetable processing, like pomaces. They discussed the potential of pomaces as an ingredient to enrich food products and the impact of its inclusion on various physical, chemical, and sensory attributes. In this sense, with the focus on obtaining the greatest levels of bioactive compounds, effort should be in selecting appropriate processing parameters, from the obtaining of the ingredient to its uses. In that context, a common practice for obtention of by-product ingredients is through the drying process followed by milling the pomace choosing the adequate method can reduce the loss of bioactive compounds [22–24]. Drying process allows pomace to be conserved, and milling allows its incorporation as an ingredient [25]. studied how the particle size of dried apple pomace affects its surface properties and its potential as soluble dietary fibre source in processed foods. In the case of phenolic compounds, it is known that although they hold all the characteristics required to be considered valuable functional ingredients, they should be analysed for their bioaccessibility and bioavailability after digestion, and these may be affected by the particle size of fruit pomaces [26]. studied the effect of particle size of seed pumpkin by-products powders on the bioaccessibility of carotenoids and antioxidant capacity during *in vitro* digestion process observing a slightly higher ferric reducing abilities for the bigger size pomace. On the other hand [27], studied how particle size affects the phytochemical content and antioxidant properties of two persimmon flours. While the impact wasn't always clear, smaller particles generally had higherlevels of bioactive compounds and antioxidants. Currently, there is no research that investigates the impact of particle size on the functional properties of other fruit pomaces following the digestion process.

The objective of this work was to evaluate the effect of particle size on bioaccessibility of phenolic compounds and microstructure of different agro-industrial by-products with potential to be used as food ingredients.

2. Materials and methods

2.1. Fruit pomace powder preparation

The agro-industrial by-products were obtained from apple, orange and grapes. Fresh pomace from oranges and apples were obtained as a by-product from industrial juice production, from Montevideo, Uruguay. Grape wine pomace (Tannat variety) was obtained as a by-product from wine production from a local winery (Canelones, Uruguay).

Orange, apple and grape pomaces were dried in a convection oven at 50 °C ± 2 °C until the moisture content was less than 10 g/100 g, to achieve microbiological stability. Once each pomace reached room temperature, they were ground in a laboratory mill (Retsch ZM 200) at two sizes: samples passed through 1 mm and 0.5 mm to obtain a powder particle size of less than 1 mm and 0.5 mm respectively. So, six samples were obtained: orange pomace powder 1 mm (OPP1); orange pomace powder 0.5 mm (OPP0.5); apple pomace powder 1 mm (APP1); apple pomace powder 0.5 mm (APP0.5); grape pomace powder 1 mm (GPP1) and grape pomace powder 0.5 mm (GPP0.5). Regarding the grinding process, the speed was adjusted between samples to prevent a significant increase in the temperature of the ground sample, and the grinding time varied accordingly.

2.2. Proximate composition

Proximate analyses were performed on dried orange, apple, and grape pomace. Analyses were done according to methods proposed by the AOAC Association of Official Analytical Collaboration [28]. Protein content was determined by Kjeldahl method and performed according to method 981.10 of the AOAC International with a conversion factor of 6.25. Fat content was determined by Soxhlet method as described in AOAC 2003.5. Moisture content was determined by gravimetric analysis in a convection oven at 105 °C until constant weight in accordance with AOAC 925.09. Quantification of ash content was performed with a furnace in accordance with AOAC 923.02. Total dietary fibre was measured with a gravimetic-enzimatic method according to AOAC 985.29. Total carbohydrate was determined by the difference method: 100 - (weight in grams [moisture + ash + protein + total fat + total dietary fiber in 100 g of

food).

2.3. Particle size distribution

Particle size distribution analysis of orange, apple and grape pomace milled at 1 and 0.5 mm particle size (OPP1, OPP0.5, APP1, APP0.5, GPP1 and GPP0.5 respectively) were performed with a Laser Diffraction Particle Size Analyzer (Mastersizer, UK), cell ID1550, with distilled water as dispersant. The percentage of volume (% volume) corresponding to each observed population was calculated. Calculation was done with the software provided with the equipment (Microtrac FLEX 0.5.2).

2.4. Microstructure

2.4.1. Light microscopy

Samples of orange, apple and grape pomace milled at 1 and 0.5 mm particle size (OPP1, OPP0.5, APP1, APP0.5, GPP1 and GPP0.5 respectively) were placed on slides and observed at a magnification of 20× under a light microscope (Nikon Eclipse 80i, Japan). The micrographs were stored at 1280 × 1024 pixel resolution using the microscope software (NIS-Elements F, Version 4.0, Nikon, Tokyo, Japan).

2.4.2. Field emission scanning electron microscopy (FESEM)

Surface structure of orange, apple and grape pomace milled at 1 and 0.5 mm particle size (OPP1, OPP0.5, APP1, APP0.5, GPP1 and GPP0.5) was observed in a field emission scanning electron microscope (model Ultra 55 FESEM, Zeiss, Oberkochen, Germany). Pomace powders were attached to the stubs using double-sided adhesive tape, vacuum coated with platinum, and observed under the microscope.

2.5. Simulated in vitro gastrointestinal digestion

To study the potential bioaccessibility of phenolic compounds, an *in vitro* digestion was carried out on samples following the methods proposed by Refs. [29,30]. The digestion process was conducted in a "Carousel 6 Plus" reaction station (Radleys, UK) under controlled temperature (37 °C), agitation (0.296 g), without light, and under an N_2 atmosphere. The method consists of three stages: salivary, gastric and intestinal.

5 g of samples of orange, apple and grape pomace milled at 1 and 0.5 mm particle size (OPP1, OPP0.5, APP1, APP0.5, GPP1 and GPP0.5, respectively) were used for the digestion simulation.

After digestion, digested solution was transferred to tubes to be centrifuged for 30 min at 27,666×*g*, 4 °C. The supernatant was filtered (0,45 μm pore size filter), corresponding to the accessible fraction, and frozen at −80 °C until analysis.

2.6. Sample extraction process

Polyphenol compounds of undigested sample and digested samples were extracted with ethanol, according to the following methodology, 5 g of undigested samples or 5 mL of digested sample, of orange, apple and grape pomace milled at 1 and 0.5 mm particle size (OPP1, OPP0.5, APP1, APP0.5, GPP1 and GPP0.5) were homogenized in an Ultraturrax with 25 mL of 960 g kg- 1 ethanol. The homogenate was centrifuged (27,666×*g*, 20 min, 4 °C) and filtered. The supernatant was kept. More supernatant was extracted from the pellet with 25 mL of 960 g kg^{-1} ethanol and added to the first supernatant. The total supernatant was brought up to 100 mL with 960 g kg^{-1} ethanol. From this solution, the sample was collected to assay total polyphenol content and antioxidant capacity. Extracts were kept frozen at −18 °C until analysis were performed.

2.7. Antioxidant capacity and total phenolic content determination

2.7.1. Total phenolic content (TPC)

The total phenolic content of the digested and undigested fruit pomaces was determined according to the Folin– Ciocalteu method [31]. 1 mL of sample was placed in a tube, adding 6 mL of deionized water and then 0.5 mL of the Folin–Ciocalteu reagent. The mixture was shaken. After 3 min, 1 mL of a saturated sodium carbonate solution was added, and the mixture was shaken. Finally, 1.5 mL of deionized water was added. Tubes were incubated for 90 min in the dark and then absorbance was read at 760 nm in a Shimadzu 1800 UV–Visible spectrophotometer. The calibration curve was obtained using different concentrations of gallic acid (from 10 to 120 ppm) in 960 g kg-1 ethanol. Results were expressed as milligrams of gallic acid per 100 g of dry weight. Measurements were performed in triplicate.

2.7.2. Antioxidant capacity by ferric reducing antioxidant power (FRAP) assay

Antioxidant capacity of the digested and undigested fruit pomaces was determined according to Ref. [32] with minor modifications. Distilled water (30 μL), sample (30 μL), and FRAP reagent (900 μL) were placed in each cuvette. The cuvettes were incubated for 30 min in a water bath at 37 °C and the absorbance was measured at 595 nm in a Shimadzu 1800 UV–Visible spectrophotometer. The calibration curve was obtained using different concentrations of Trolox (from 50 to 150 μM) in 960 g kg^{-1} ethanol. The results were expressed as μmol $_{Trolox/g}$ of sample. Measurements were performed in triplicate.

2.8. Recovery index

To analyse the effect of *in vitro* digestion on total phenolic content, the recovery index was used. The recovery index quantifies the difference between the phenolic content in the undigested fraction and digested fractions [33]. The recovery index (RI) was measured using equation (1).

$$Recovery\ index\ RI\ (\%) = \frac{DF}{UDF} \times 100 \quad (1)$$

DF (digested fraction) is the content of bioactive compounds in the digested fraction and UDF (undigested fraction) is the content of bioactive compounds quantified in fresh samples.

2.9. Data analysis

Analyses were performed in triplicate, and all data reported as mean ± SD. t-Student test was performed on results of TPC and AC assays for each fruit pomace between the two sizes and for each fruit pomace, between before and after digestion for the same particle size ($\alpha \leq 0.05$). Analyses were performed using XLSTAT Version 2011 (Addinsoft 1995–2010, France).

3. Results and discussion

3.1. Proximate composition

Proximate composition of orange, apple, and grape dried pomace, including contents for protein, fat, ash, dietary fibre, moisture and carbohydrate are shown in Table 1. As notable features, all pomaces presented high contents of dietary fibre, especially grape pomace. The amount of total dietary fibre (TDF) is consistent with those reported in the bibliography for grape [34,35], apple pomace [36,37], and orange pomace [38]. The high TDF content in fruit pomaces makes them a valuable ingredient for product formulation [21].

Orange and apple pomace have also a high carbohydrate content (calculated by difference). This carbohydrate content is composed mainly of sugars; therefore, it would be possible to use these ingredients to add sweetness, which reduces the amount of sugar needed in product formulation [39].

The main difference between pomaces is the higher fat and protein content presented in grape pomace, reliably related to seed contribution. Lipids, proteins, and ash content in grape pomace are very similar to those found by Refs. [40–42].

3.2. Particle size distribution

Fig. 1 shows the particle size distribution of samples of orange, apple and grape pomaces milled and passed through 1 mm and 0.5 mm to obtain a powder particle size of less than 1 mm and 0.5 mm respectively.

Although all pomaces were grounded to the same two sizes and passed through the same mesh (1 mm and 0.5 mm), pomace powders showed differences between them. Observing pomaces grounded to 1 mm, apple pomace presented smaller particles than grape pomace and orange pomace: at 99 % tile, 675.0 μm, 862.1 μm and 946.8 μm respectively. In the case of pomaces at 0.5 mm, considering 99 % tile, grape pomace (495.5 μm) was smaller than apple pomace (538.2 μm) and orange pomace (666.0 μm).

The particle size of pomaces may vary due to differences in their fibre composition, mainly in the insoluble fiber components. According to Ref. [43] lignin content is about 6 % in apple pomace and 17 % in orange pomace. Lignin is an amorphous complex cross-linked phenolic polymer of phenylpropane units (p-coumaryl, coniferyl and sinapyl alcohol), held together by different linkages [44] and responsible for the hardness of the fruit matrix. The differences in size distribution between different pomaces may be related to the hardness of the matrix: as orange pomace is harder, grinding rendered bigger particles.

When using flour pomaces as ingredients in product formulation, it is important to consider their particle size. The particle size may impact the sensory characteristics of the final product. Smaller particles expand the potential applications of ingredients [45,46].

Table 1
Fruit pomace powder composition per 100 g (db).

Component	Mean Value		
	Orange Pomace	Apple Pomace	Grape Pomace
Proteins (g)	6.55	3.40	10.46
Lipids (g)	1.01	1.20	10.07
Total dietary fibre (g)	33.8	23.1	63.9
Carbohydrates (g)[a]	55.7	70.4	11.1
Ash (g)	2.93	1.86	4.55
Moisture (g)	7.88	6.12	8.53

[a] Carbohydrates were determined by difference (100-protein-lipids-ash- total dietary fibre).

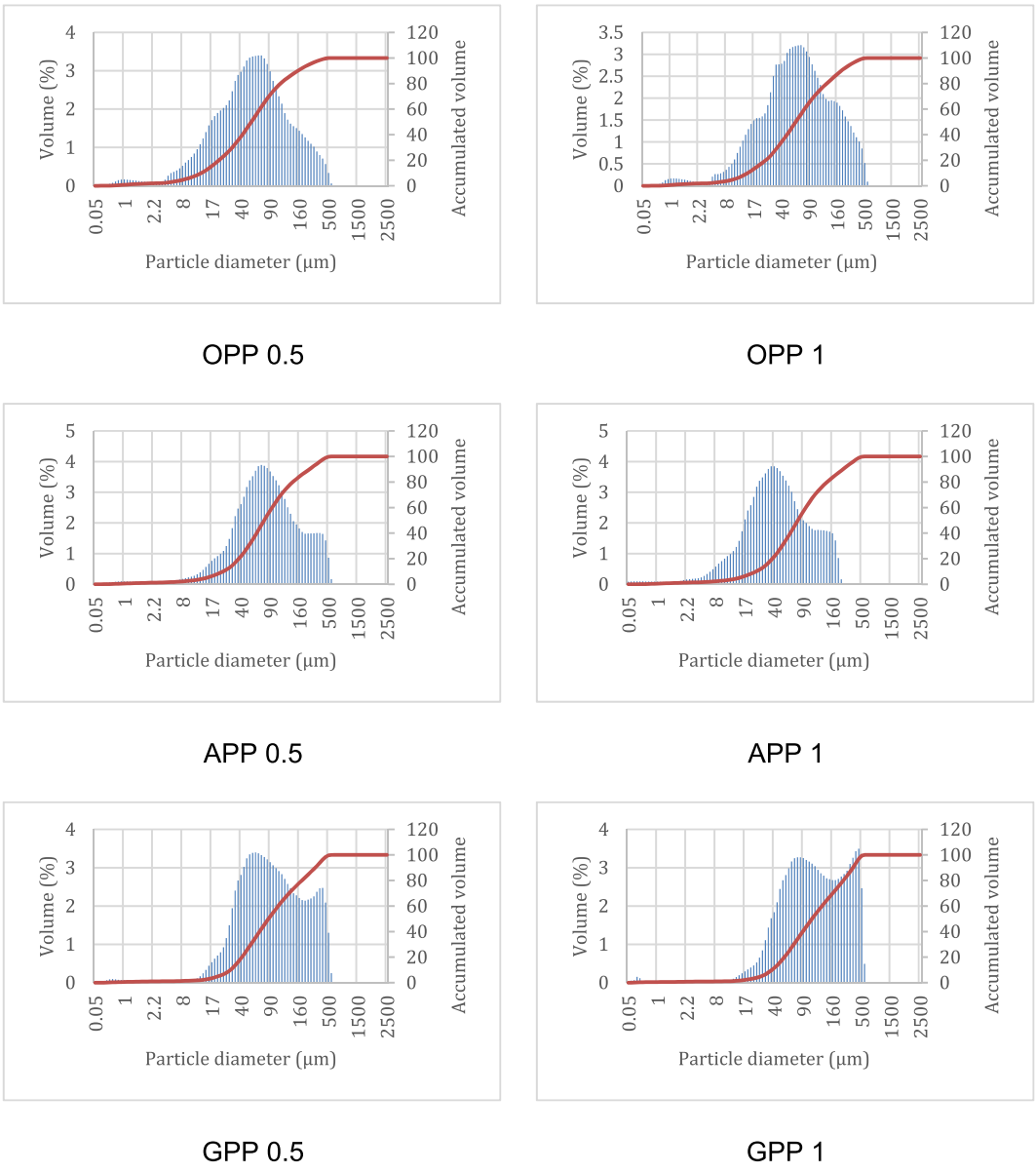

Fig. 1. Particle size distribution of pomaces milled at two sizes: 0.5 mm and 1 mm: orange powder pomace (OPP 0.5 and OPP 1), apple powder pomace (APP 0.5 and APP 1) and grape powder pomace (GPP 0.5 and 1). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

3.3. Microstructure

Photomicrographs of the undigested orange, apple, and grape pomace, milled at 0.5 and 1 mm are shown in Fig. 2. As observed in particle size distribution, although all fruit samples were milled at the same size (0.5 and 1 mm), pomaces showed differences in particle size observed using light microscopy. Differences follow the same trends observed in particle size distribution (Fig. 1). At both sizes, orange pomace seems to be bigger than grape and apple pomaces. As explained previously, differences may be attributed to differences in fibre composition, especially due to lignin content.

Fig. 3 shows FESEM images of the orange, apple, and grape pomace with 0.5 mm particle size at 100 × and 1500 ×. Grape pomace is observed as a porous, more accessible structure, which may ease the polyphenol extraction. On the other hand, orange pomace structure was more compact. Finally, apple pomace structure was even more compact than orange structure forming sheets, which may hinder the polyphenols' extraction.

According to Ref. [47], protein and fat content may affect the structure of pomace powder. Specifically, the presence of protein seems to favour the agglomeration of the particles, while the fat acts as a binder that prevents the fracture of the particles into smaller

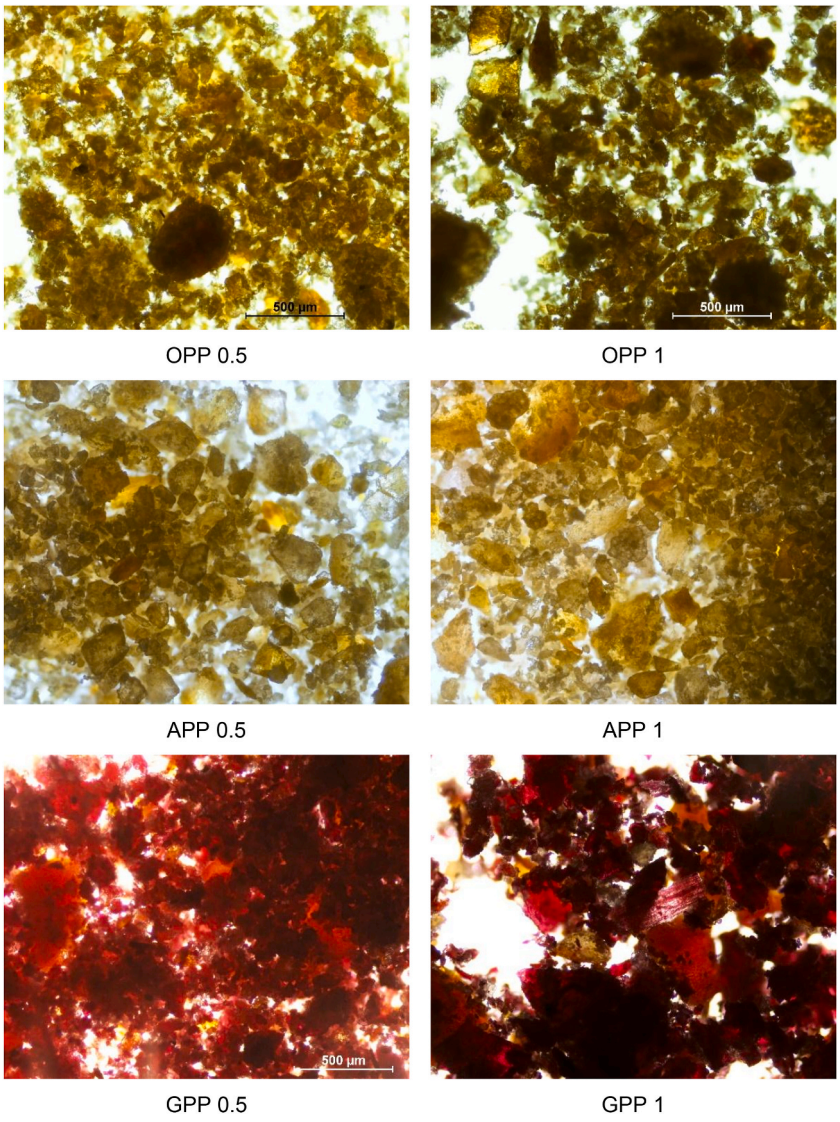

Fig. 2. Light microscopy images of orange (OPP), apple (APP) and grape (GPP) pomace powders milled at 0.5- and 1-mm particle size. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

pieces. In our study, AP powders, which had a low content of protein and fat, do not show an agglomerated structure but one built by small and unstructured pieces superimposed one on the other.

3.4. Total phenolic content and antioxidant capacity

3.4.1. The effect of an in-vitro digestion process

Results of total phenolic content (TPC) and antioxidant capacity (AC) of samples in the undigested raw material (non-digested) and after digestion (digested) are shown in Figs. 4 and 5 respectively.

The total phenolic content (TPC) of fruit pomaces varied significantly (p-value <0.0001) before and after digestion (Fig. 4). Before digestion, grape pomace had the highest TPC, followed by orange and apple pomaces. After digestion, TPC decreased for grape pomace but increased for orange and apple pomace, resulting in an order of orange $>$ grape $>$ apple.

The high TPC value of grape pomace before digestion may be attributed to the presence of oligomeric and polymeric proanthocyanidins (PAs) which are the most abundant phenolic constituents of grape antioxidant dietary fiber [48]. According to Refs. [49–51], some of the PAs found in grape antioxidant dietary fiber, which is similar to grape pomace, are associated with the fiber matrix through weak interactions. Due to these weak bonds, they may be extracted by solvents and quantified as TPC in analytical determination.

After digestion, TPC in grape pomace decreased significantly [52]. found that gastrointestinal digestion had a reducing effect on TPC and antioxidant activity of winemaking by-products extracts. This reduction may be related to the reduction in anthocyanin

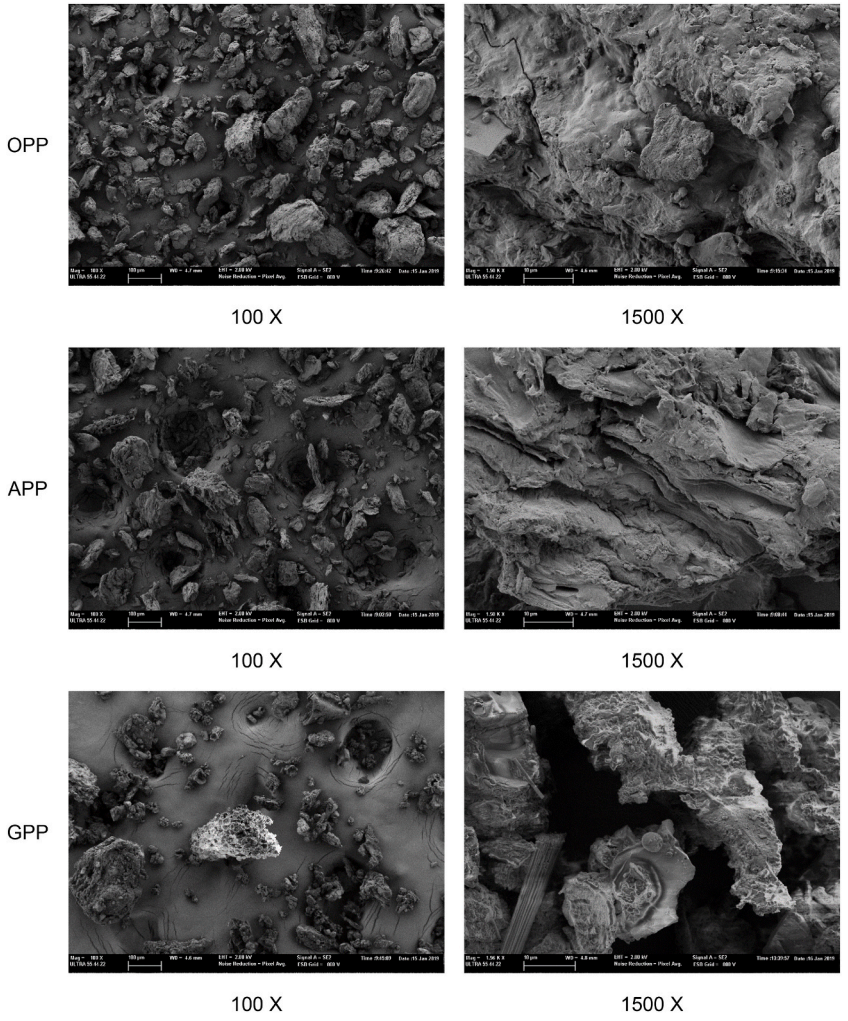

Fig. 3. FESEM images of orange (OPP), apple (APP) and grape (GPP) pomace powdered at 0.5 mm particle size, at 100 × and 1500 ×.

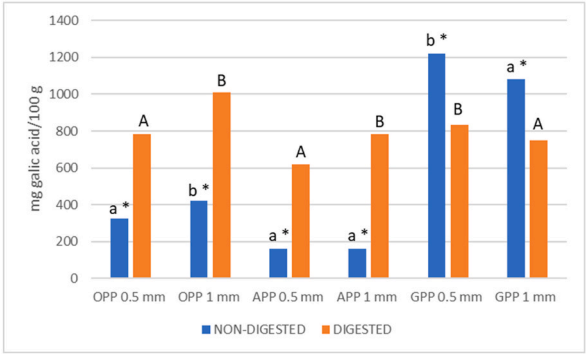

Fig. 4. Total Phenolic Content of fruit pomaces at 0.5 mm and 1 mm, on the non-digested and digested samples.
For each fruit pomace, different letters mean significant difference between size according to t-student test ($p < 0.05$): letters in lowercase for the non-digested samples and in capital letter for digested samples. For each fruit pomaces, the presence of an asterisk (*) means significant differences between the non-digested and the digested sample, for the same particle size, according to t-student test ($p < 0.05$).

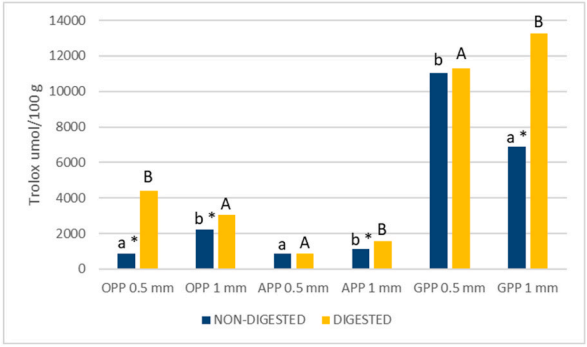

Fig. 5. Antioxidant Capacity of samples of fruit pomaces at 0.5 mm and 1 mm, on the non-digested and digested samples.
For each fruit pomace, different letters mean significant difference between size according to t-student test ($p < 0.05$): letters in lowercase for the non-digested samples and in capital letter for digested samples. For each fruit pomaces, the presence of an asterisk (*) means significant differences between the non-digested and the digested sample, for the same particle size, according to t-student test ($p < 0.05$).

contents. According to Ref. [53] *in vitro* digestion process led to drastic qualitative and quantitative reductions in the phenolic compounds profile of grape pomace, especially in total anthocyanin compounds which showed a reduction of almost 5-fold.

In the case of apple and orange pomace, TPC increased after digestion when compared to raw material. Similar results were observed by Ref. [11] who analysed the bioactive compounds and bioaccessibility of apple, blueberry, raspberry and cranberry dried pomace. These authors observed an increase in TPC in apple pomace after digestion, attributing this increase to the fact that phenolics were bound with the macromolecules, especially dietary fibres, which were subsequently released during digestion through the action of digestive enzymes. These free phenolics were made available to be absorbed in the human body. The digestion process in the intestine can lead to the release of phenolic compounds from food due to the solubilization of food in intestinal fluids and the action of digestive enzymes that hydrolyze proteins, carbohydrates, and lipids [54]. [55] found that more bioavailable polyphenols can be present after digestion due to the hydrolysis of non-extractable polyphenols. It is worth noting that non-extractable polyphenols (NEPPs) make a significant contribution to the total polyphenol content in plant-based foods. However, during the typical aqueous-organic treatments used to evaluate polyphenol content in foods, NEPP are not extracted [17], and therefore may not be quantified as part of raw material's TPC.

Another way to evaluate and quantify the effect of the matrix composition on the digestion of the phenolic compounds, is through the calculation of the recovery index (RI). This index represents the amount of phenolic compounds recuperated after the *in vitro* digestion, by comparison with the amount in the undigested sample (Table 2).

No statistical differences ($p > 0.05$) were found for recovery index value between the different sizes for orange and grape pomace. However, in the case of apple pomace, the recovery index showed higher values for the sample with higher size. Comparing the different pomaces, the recovery index values were higher for apple > orange > grape.

Concerning antioxidant capacity (AC), although the trend was similar to those observed for total phenolic content (TPC) (Figs. 4 and 5), the difference in TPC between fruit pomaces after digestion, was less pronounced than the one in the AC. AC varied to a greater extent between pomaces and different particle sizes (Fig. 5). This indicates that even if different fruit pomaces have similar TPC values after digestion, the antioxidant capacity may vary depending on the phenol compounds and their chemical stability as observed previously by Ref. [56]. Concerning antioxidant capacity before digestion, grape > orange > apple, and, in all cases, AC increased or showed no differences after digestion. According to Ref. [57], some polyphenols could be decomposed to components with higher antioxidant potential due to the action of digestive enzymes and pH changes during the digestive process, causing an increase in the AC concerning the TPC. In this context, grape pomace showed the expected behaviour according to Ref. [9], who studied bioaccessibility of bioactive compounds from pomaces, observing that TPC decreased after digestion while antioxidant capacity analysed by ORAC-FL assay was unchanged or increased after digestion. On the contrary, other studies carried out with others type of pomaces as persimmon

Table 2
Recovery index (RI) of total phenolic content after the simulated gastro-intestinal digestion of orange, apple, and grape pomace, milled at 0.5 and 1 mm.

Samples	RI (%)
OPP 0.5 mm	239
OPP 1 mm	239
APP 0.5 mm	382
APP 1 mm	483
GPP 0.5 mm	69
GPP 1 mm	69

[27], wild blueberries [56], and red grape skin [10] observed loss of antioxidant compounds during the digestion process. In a study conducted by Ref. [58], it was found that the flavonoids and antioxidant activity in citrus peel extracts were sensitive to pH conditions during *in vitro* gastrointestinal digestion [59]. conducted a study on the effects of pH changes during *in vitro* digestion of oregano. The authors observed that these changes led to a decrease in the antioxidant capacity of polyphenols.

3.4.2. The effect of particle size

Regarding the impact of particle size on TPC and AC, variations were observed among the different fruit pomaces. There were differences in total phenolic content (TPC) for grape pomace and orange pomace before and after digestion, depending on the particle size. For grape pomace, TPC at 0.5 mm particle size was higher than at 1 mm particle size, while for orange pomace it was the opposite, with TPC at 1 mm particle size being higher than at 0.5 mm particle size. In the case of apple pomace, there was no significant difference in TPC between both particle sizes before digestion. For apple pomace, however, after digestion, TPC for 1 mm particle size in was found to be higher than at 0.5 mm particle size.

For the antioxidant capacity (AC), the undigested GPP with a particle size of 0.5 mm had almost double AC compared to grape pomace with a particle size of 1 mm. However, after digestion, the AC of grape pomace with a particle size of 1 mm increased significantly and was higher than grape pomace's with a particle size of 0.5 mm.

The AC of the orange pomace was nearly 3 times bigger for the 1 mm sample size compared to the 0.5 mm sample size before digestion. However, after digestion, the AC of the sample at 0.5 mm increased considerably, surpassing even the sample at 1 mm, which only showed a slight increase in AC.

In the case of apple pomace (APP), the undigested 1 mm particle size pomace had a bigger AC than the 0.5 mm particle size pomace. However, for the 0.5 mm size, there was no significant difference found between the samples before and after digestion. In the case of APP at 1 mm, the AC was slightly bigger after digestion compared to before digestion.

The decrease in particle size can affect the levels of antioxidant compounds. Several factors, including increased surface area, particle size distribution, and cellular release of bioactive compounds, may contribute to this effect. These factors might be linked to the observed differences in bioactive compounds concentration and antioxidant activity among varying particle sizes [27]. It is expected that smaller particle sizes increase the amount of polyphenol content and antioxidant capacity due to the corresponding increase in surface area. [26], studied the antioxidant activity of pumpkin by-product powders milled at 2 different particle sizes and observed slightly higher ferric reducing abilities for the bigger size pomace. These authors observed a reduction in the ability of samples to reduce other substances during the entire digestive process. This decrease might be attributed to the actions of various digestive enzymes found in the gastrointestinal tract. According to Ref. [60] pepsin and trypsin exhibited different impacts on the FRAP antioxidant activity of the active substrates. According to Ref. [61] for whole wheat bran, as the particle size decreased, the levels of phenolic acids, anthocyanins, carotenoids and ORAC value also increased.

On the other hand [62], conducted a study on the impact of particle size distribution on the functional properties of unripe banana flour, and they did not find a clear trend in the antioxidant capacity of samples [27]. studied the effect of particle size on phytochemical composition and antioxidant properties of persimmon flours and, although the influence of particle size in all these properties was not always consistent, in general, the smallest particles tended to have the highest levels of bioactive compounds and the strongest antioxidant capacity.

This works highlights the sustainable and functional applications of fruit pomaces as upcycled food ingredients, which align with the principles of a circular economy and waste reduction. These pomaces are not only rich in bioactive compounds like phenolics, which have significant antioxidant properties, but also high in dietary fibers that contribute to health benefits.

Moreover, the use of these pomaces can lead to economic benefits by transforming what would typically be waste products into valuable food ingredients. This approach not only helps in reducing the environmental impact but also adds value to the food production chain.

4. Conclusions

According to proximate composition, all pomaces have the potential to be used as food ingredients with a special contribution to enhancing fibre content. Regarding particle size distribution, although pomaces were ground at the same size (0.5 and 1 mm), pomaces exhibited different particle size distributions, possibly due to differences in the hardness of the matrixes. This fact could affect the sensory properties of future products to be developed with these flours, so attention should be paid to this matter.

Concerning the availability or extractability of polyphenol compounds after digestion, it was increased for orange and apple pomaces. In the case of grape pomace, although polyphenol content decreased after digestion, they showed the highest antioxidant capacity.

Regarding the effect of particle size on total polyphenol content and antioxidant capacity, no trend was found in this work for the fruit pomaces studied. In the case of grape and apple, it seems grinding at 1 mm should be adequate regarding antioxidant capacity while in the case of orange it may be better to use a pomace ground at 0.5 mm. As particle size reduction can be a useful strategy for processing fruit by-products, the effect of particle size in food products and consumer response should be investigated.

The results of this study can inform strategies for reducing fofood waste and lay the groundwork for further investigations.

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CRediT authorship contribution statement

Patricia Arcia: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ana Curutchet:** Writing – review & editing, Investigation. **Claudia Pérez-Pirotto:** Writing – review & editing, Formal analysis, Data curation. **Isabel Hernando:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:Patricia Arcia reports travel was provided by Banco Santander Uruguay. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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