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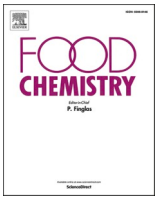

Composition of discarded Sicilian fruits of *Opuntia ficus indica* L.: Phenolic content, mineral profile and antioxidant activity in peel, seeds and whole fruit

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ABSTRACT

Sicily (Italy) is the second producer of *Opuntia ficus-indica* (OFI) fruits after Mexico. To date, huge quantities of fruit are discarded during the selection for the fresh market, generating a large amount of by-product to be valorized. This study aimed to investigate on the composition of OFI discarded fruits from the main Sicilian productive areas, over two harvesting periods. Peel, seeds and whole fruit samples were characterized in terms of minerals and phenolic compounds through ICP-OES and HPLC-DAD-MS. Potassium, calcium and magnesium were the most abundant elements and peel samples showed the highest values. Seventeen phenolic compounds were detected in peel and whole fruit, including flavonoids, phenylpyruvic and hydroxycinnamic acids, while only phenolic acids were found in the seeds. A multivariate chemometric approach highlighted a correlation between the mineral and phenolic content and the different parts of the fruit as well as a significant influence of productive area.

1. Introduction

Opuntia ficus-indica (L.) Mill., also known as prickly pear cactus, is a plant native to Mexico that grows worldwide, such as South America, Africa, Australia and the Mediterranean basin, in areas where no other crops are able to do. The last ten years have seen an increasing interest of the scientific community towards the prickly pear, as evidenced by the numerous articles focused on the study of different parts of the plant, in particular fruits and cladodes (Abbas, Ezzat, El Hefnawy, & Abdel-Sattar, 2022; Barba, Garcia, Fessard, Munekata, Lorenzo, Aboudia, et al., 2022; Di Bella, Lo Vecchio, Albergamo, Nava, Bartolomeo, Macri, et al., 2022; Scarano, Tartaglia, Zuzolo, Prigioniero, Guarino, & Sciarrillo, 2022). FAO considers this plant an important resource for the future mainly because of the low production costs, the low environmental impact and the ability to grow in arid soils without the need for particular agronomic treatments. Thanks to all these factors, *O. ficus indica* (OFI) is recognized by FAO as a natural source capable of guaranteeing an income for producers even in critical environmental conditions (FAO & ICARDA, 2017). The leading producers and consumers of OFI are Mexico and Italy, and it is in Mexico that this species has the highest degree of genetic diversity. Of the approximately 590,000 ha grown worldwide, Mexico and Italy contain 70% and 3.3%, respectively (Martins, Ribeiro, & Almeida, 2023).

Prickly pear is a good source of dietary fiber, vitamins and bioactive compounds which showed interesting biological activities such as anti-inflammatory, hypoglycemic and antimicrobial (Abbas, Ezzat, El Hefnawy, & Abdel-Sattar, 2022; Silva, Albuquerque, Pereira, Ramalho, Vicente, Oliveira, et al., 2021). The phytochemicals detected in the fruits so far belong to different chemical classes. The main ones were phenolic compounds such as flavonoids and cinnamic derivatives, but also pigments belonging to the group of betalains (Amaya-Cruz, Perez-Ramirez, Delgado-Garcia, Mondragon-Jacobo, Dector-Espinoza, & Reynoso-Camacho, 2019; Aruwa, Amoo, & Kudanga, 2018; Butera, Tesoriere, Di Gaudio, Bongiorno, Allegra, Pintaudi, et al., 2002; Galati, Mondello, Giuffrida, Dugo, Miceli, Pergolizzi, et al., 2003; Garcia-Cayuela, Gomez-Maqueo, Guajardo-Flores, Welti-Chanes, & Cano, 2019; Mena, Tassotti, Andreu, Nuncio-Jauregui, Legua, Del Rio, et al., 2018). In particular, Mena et al. (2018) reported 26 polyphenolic compounds detected by

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UHPLC-ESI-MS^n in peel from six Spanish cultivars of prickly pears reporting that phenolic acids such as ferulic acid-hexoside, dihydrosinapic acid-hexoside, and sinapic acid-hexoside had a higher prevalence above flavonols as isorhamnetin-rutinoside. Moreover, in the investigation conducted by Amaya-Cruz et al. (2019) the phytochemical profile of the peel of fruits of different color assessed by UPLC-ESI-QTOF MS^E allowed the identification of 145 compounds including sixty-eight extractable polyphenols, fifteen hydrolysable polyphenols, forty-one betalains, sixteen carotenoids, and five phytosterols. Other authors studying fruits of Mexican and Spanish cultivars, obtained a very similar pattern for the phenolic compounds and particularly for the flavonoids (Garcia-Cayuela, Gomez-Maqueo, Guajardo-Flores, Welti-Chanes, & Cano, 2019); these authors proposed an HPLC methodology useful for the simultaneous and accurate analyses of betalains and phenolic compounds in OFI fruit tissues.

Nowadays, prickly pear by-products including peel, pulp and seed generated in large amount from fresh fruit selection, is drawing more and more attention, as a source of valuable compounds (Barba, Putnik, Kovacevic, Poojary, Roohinejad, Lorenzo, et al., 2017). In particular, the prickly pear peel, which comprises around 30% of the weight of the fruit (Melgar, Dias, Ciric, Sokovic, Garcia-Castello, Rodriguez-Lopez, et al., 2017), has been used for the extraction of polysaccharides and phenolic compounds and is considered an agro-industrial waste that could represent a promising source of bioactive compounds (Barba, et al., 2017).

The market of edible fresh fruits requires to maintain size and shape of the product within specific parameters to guarantee the requested quality. Consequently, for a more sustainable production, the loss of high amount of ripe fruits not includible within the morphological parameters expected by the market is a real problem that requires to be overcame. So far, few efforts have been done to reduce this problem during the production and commercialization of the fresh fruits of OFI.

The purpose of this work was to provide an overview of the composition of discarded fruits of OFI collected from different farms located in the two main production areas of Sicily, the districts of San Cono and Biancavilla (Sicily). For an industrial process, it is important to know the quality of the input material acquiring information on the variability of the composition over different harvesting periods and years. Taking into account the possibility to re-use these fruits, a representative sample of different white, red and yellow pulp of typical Italian cultivars was collected and the different part of the fruits - peel, seeds and whole fruit - in two different years at two harvesting periods were characterized. Some commercial fresh prickly pear samples destined for the fresh produce market were included in the study to compare the quantitative results with those derived from the analyses of discarded fruits. The focus was on mineral content, phenolic compounds and antioxidant activity. A multivariate chemometric approach by principal component analysis (PCA) and linear discriminant analysis (LDA) was applied to identify possible relationships between chemical data, geographical origin, harvesting period and different parts of the fruit.

2. Materials and methods

2.1. Fruit sample collection and preparation

OFI discarded fruits have been provided by different farms located in two different districts in Sicily: San Cono and Biancavilla. Fruits from Biancavilla are protected by the Product Origin Denomination (POD) brand as 'Ficodindia dell'Etna' according to EU Regulation 2081/92 (CE, 1992) and are considered typical of the production area located in the Etna district; prickly pear from San Cono received the PDO (Protected Designation of Origin) logo by Ministry of Agricultural, Food and Forestry Policies (2010). The local climate is semiarid-Mediterranean, with mild winters and hot, rainless summers.

Fruits were collected in 2018 and 2019, at the end of August ("Agostani") and in late-October ("Bastardoni"), throughout the manuscript referred as first and second harvest, respectively. All the sample are reported in Table S1. Each sample was a representative mixture of different white, red and yellow pulp of typical Sicilian cultivars. Different parts of the fruit were considered: peel (P), seed (S) and also the whole fruit (WF) that included peel, pulp and seeds. Within 24 h of the harvesting, about 100 kg of fresh fruits have been washed and manually peeled; the peel obtained (6–7 kg/fresh weight) were dried at 45 °C for 72 h. The seeds were separated from the pulp, washed abundantly with distilled water and then dried at room temperature for 48 h. All the samples were reduced into a fine powder using a blender type M20 (IKA, Germany) before their use. To compare the quantitative results of discarded fruits with those derived from prickly pear fruits destined for the fresh market, four samples were included in the study, as listed in Table S1.

2.2. Determination of the mineral content by ICP-OES iCAP

Macro and microelement analysis were performed using 0.5 g of dried sample digested with 10 mL of HNO_3 (67% v/v) in Teflon reaction vessels, to carry out the mineralization in a microwave oven (Mars 5, CEM Corp., Matthews, NC, USA), using the program 1600 W, 100% power, at 200 °C for 20 min. At the end of the mineralization, the final volume of 25 mL was reached by adding ultra-pure water. The concentrations of Ca, K, Mg, Na, P, Cu, Fe, Mn, Zn, Al and Ba were determined using an inductively coupled argon plasma optical emission spectrometer (ICP–OES iCAP series 7000 Plus Thermo Scientific). A standard method for the different elements was applied, using the QtegraTM Intelligent Scientific Data SolutionTM (ISDS) and the wavelengths selected were 315.8 nm for Ca, 766.4 nm for K, 280.2 nm for Mg, 589.6 for Na, 178.7 nm for P, 324.7 nm for Cu, 239.1 for Fe, 259.3 nm for Mn, 202.5 nm for Zn, 396,1 nm for Al and 493,4 nm for Ba quantification. The calibration was performed with several dilutions of the multi-element standard Astasol®-Mix (ANALYTIKA®, spol. s.r.o., Prague, Czech Republic) in 1% HNO3 (v/v).

2.3. Extraction of phenolic compounds

The powdered peels (1 g) obtained by grinding approximately 2 Kg of fresh fruits, were extracted twice with 10 mL of ethanol 70% (v/v) at room temperature for 45 min under stirring; the solution was then sonicated for 30 min and centrifuged at 5,000 *g* for 10 min. The two supernatants were collected, dried under vacuum at 35 °C using a rotary evaporator and re-dissolved with exactly 10 mL of the same extractive mixture.

The seed powder was defatted twice with hexane (1:10 w/v) and the phenolic compounds were recovered using the same extractive mixture used for the peel. 1 g of sample was mixed with 15 mL of ethanol 70% (v/v); the mixture was stirred for 45 min, sonicated for 30 min and then centrifuged. The supernatant was concentrated under vacuum (35 °C) and reconstituted in 5 mL of the same solution. The hydro-alcoholic extracts were analyzed by HPLC–DAD–MS. Each sample was extracted in triplicate.

2.4. Determination of phenols by HPLC-DAD-MS analysis

The analyses were performed using a HP1200 liquid chromatograph equipped with a DAD detector (Agilent Technologies, Palo Alto, CA, USA). The identification of phenolic compounds was performed by an HP 1260 MSD mass spectrometer with an API/electrospray interface (Agilent Technologies), under the following conditions: gas temperature 350 °C, nitrogen flow rate 10.5 L/min, nebulizer pressure 35 psi, capillary voltage 3500 V. The mass spectra were acquired in the *m/z* range 100–1000 Th in negative ion mode, setting the fragmentation energy between 80 and 120 V.

The separation of phenolic compounds was performed using a

Synergi Max RP 80 A column (150 × 3 mm i.d.; 4 μm particle size, Phenomenex, Castelmaggiore, Bologna, Italy). The mobile phases were: (A) acidified water (pH 3.2) and (B) acetonitrile. The following multi-step linear gradient was applied: from 85% to 80% A in 10 min, 10 min to reach 75% A, then 10 min to reach 100% B, with a final plateau of 4 min. The total time of analysis was 36 min, flow rate was 0.4 mL/min, and oven temperature was 26 ± 0.5 °C. The injection volume was 10 μL.

Chromatograms were registered at 330 and 350 nm for phenolic acids and flavonoids, respectively. These compounds were identified by comparing their retention times, UV–Vis, and MS spectra, with those of the respective standard when possible, or with our previous published data. A six-point calibration curve of ferulic acid from Extrasynthèse (Genay, France; purity 99%) at 330 nm ($R^2 = 1$) was used to evaluate the phenolic acids, while the flavonoid content was determined using a five-point calibration curve of rutin (quercetin-3-*O*-rutinoside) from Extrasynthèse (purity 95%) at 350 nm ($R^2 = 0.998$). The total phenolic content was obtained as sum of individual phenolic compounds.

2.5. Antioxidant capacity (AOC) of the extracts

The DPPH assay was performed according to the method developed by Thaipong (2006), with some modifications. The stock solution was prepared by dissolving 24 mg DPPH with 100 mL methanol and then stored at −20 °C until needed. Absorbance of the solution was adjusted to 1.1 ± 0.02 at 517 nm using methanol. The assay was performed mixing 20 μL of standard or sample with 200 μL of $DPPH^+$ solution and incubated for 30 min in the dark. Decrease of absorbance was monitored at 517 nm at 30 min using an Agilent 8453 G1103A spectrophotometer

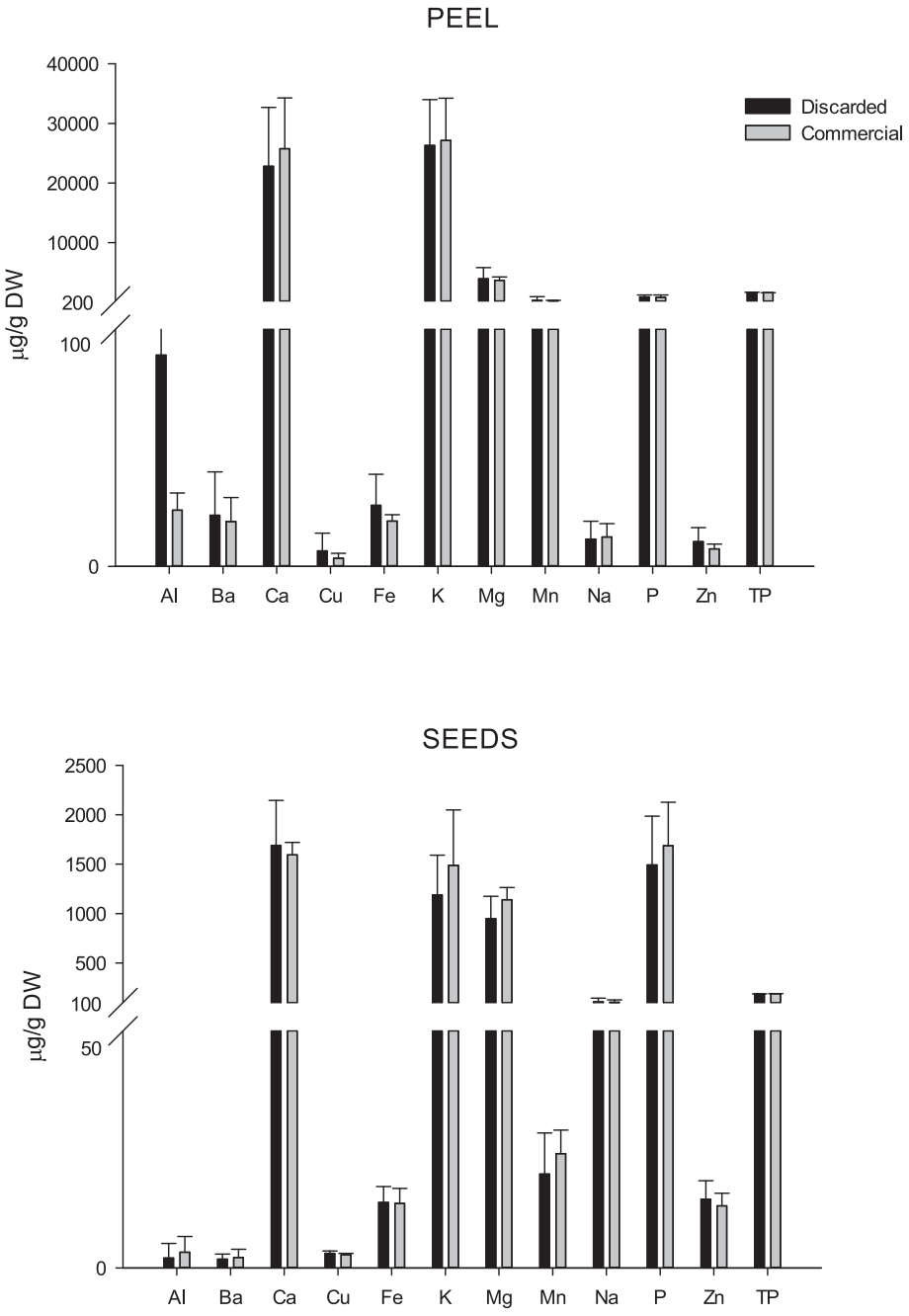

Fig. 1. Mineral and total phenolic (TP) content expressed as μg/g DW ± standard deviation in peel and seeds samples of discarded and commercial fruits of 2018. No significant differences ($p < 0.05$) are found between the two groups.

(Agilent Technologies). Gallic acid (Sigma-Aldrich) served as a standard and the results were expressed in μmol gallic acid/g dry extract (DE). The experiments were performed in triplicate.

2.6. Statistical analysis

Descriptive analysis, including the evaluation of mineral and phenolic content, were carried out in triplicates and the results were expressed as mean ± standard deviation. Analysis of variance and *F*-test ($p < 0.05$) were performed using Microsoft Excel statistical software to evaluate statistical significance. Fisher's LSD test was applied to compare the mean values using the software DSAASTAT v. 1.1. A two-way ANOVA test was employed for mean comparisons among discarded and commercial fruits. A one-way ANOVA model was also applied to compare the antioxidant activity for each botanical part (peel, seeds and whole fruits).

Principal component analysis (PCA), linear discriminant analysis (LDA) and partial least squares (PLS) regression were used from the set of chemometric tools in exploring the differences in mineral and phenolic profile and the different OFI fruit parts, geographical origin, harvesting period and year. ANOVA, PCA, LDA and PLS were run using OriginPro 2018 (OriginLab Corporation, Northampton, MA, U.S.A.).

3. Results and discussion

Sicily (Italy) is the second producer of OFI fruits after Mexico and to date huge quantities of non-standard fruit - in terms of weight, size, shape, color and appearance - are discarded during the selection for the fresh market, generating a large amount of by-product to be valorized.

Among the fruits selected for this study, four representative samples of commercial fresh prickly pear destined for the fresh market were collected in 2018 from the two geographical areas of Biancavilla and San Cono, as reported in Table S1. These samples were included in the study to compare the quantitative results in terms of mineral and phenolic contents with those of discarded fruits. The results obtained applied a two-way ANOVA model considering samples of 2018 did not highlighted significant differences ($p < 0.05$) between the two groups, neither for the mineral and the phenolic contents, as reported in Fig. 1. In light of these results, no commercial fruits were sampled for the year 2019.

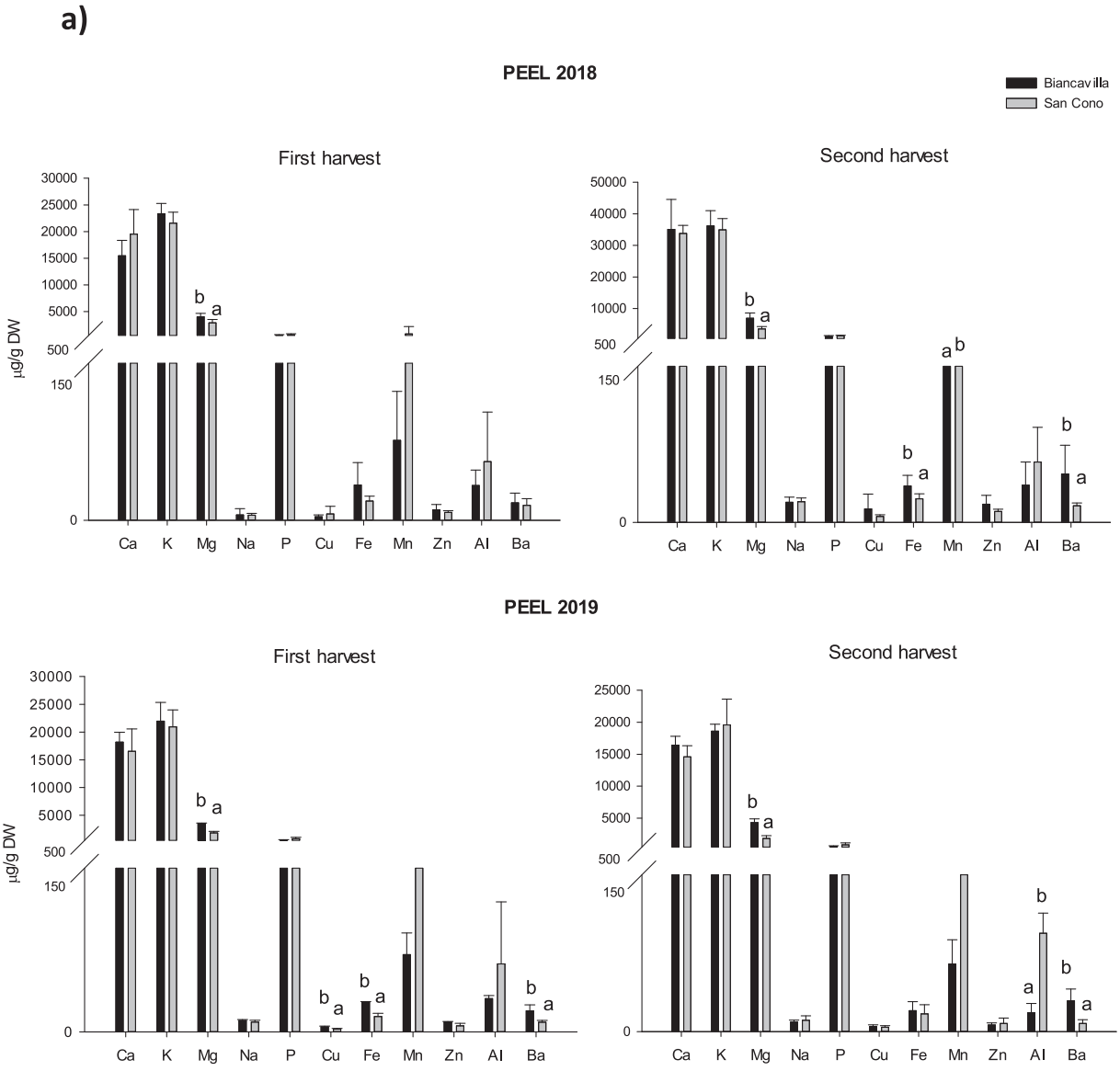

Fig. 2. Mineral content expressed as μg/g DW ± standard deviation in **a)** peel, **b)** seed and **c)** whole fruit samples from Biancavilla and San Cono. For each pair of samples, the letters are reported only when the differences are significant ($p < 0.05$).

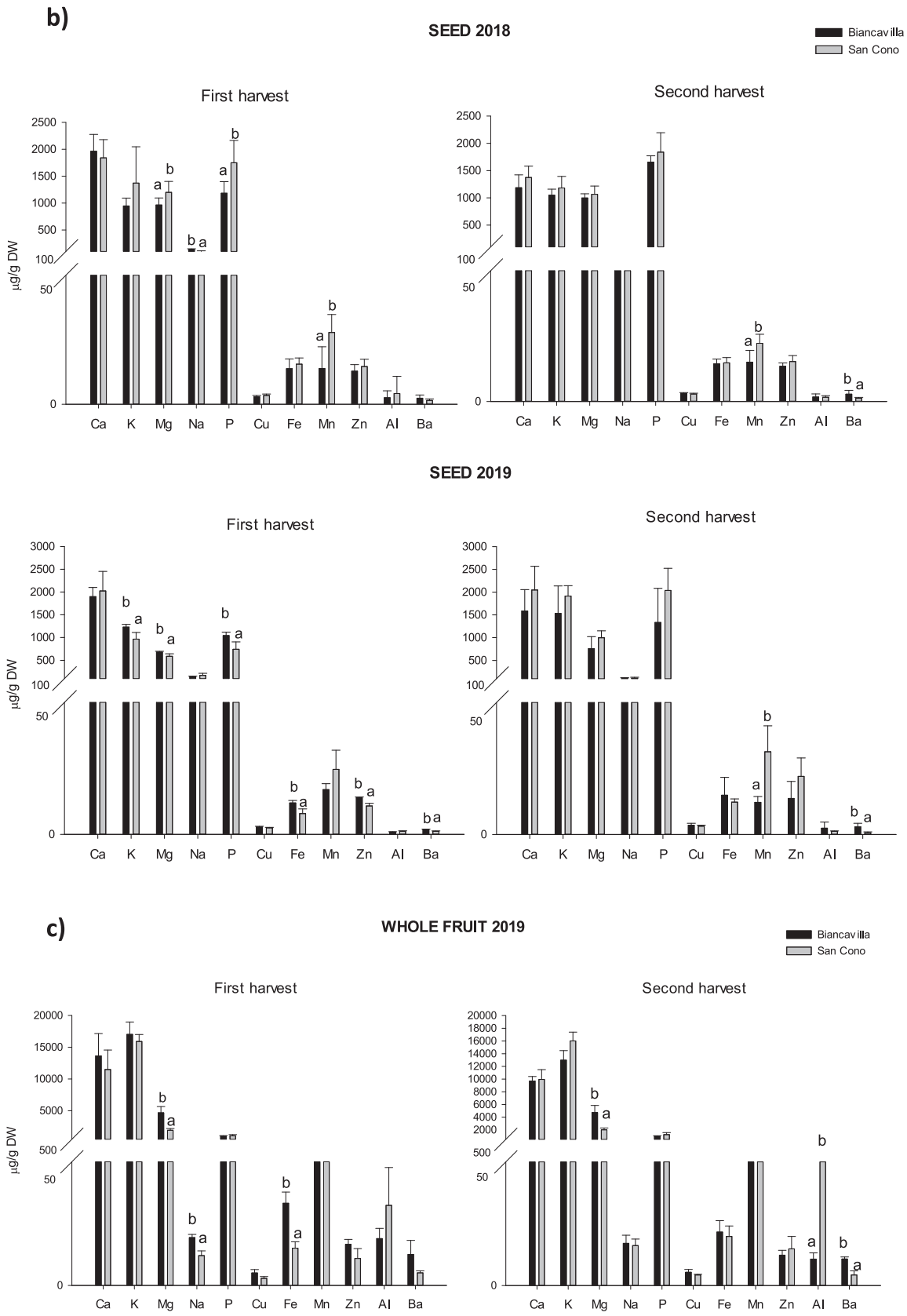

Fig. 2. (*continued*).

During the second year of this study, it was verified the possibility to apply the industrial processing also to the whole fruit and not only to seeds, peel or pulp separately. Therefore, in 2019 also the fruit *in toto* was collected and analyzed to assess its composition. The use of the whole fruit as such, avoiding preliminary separation of the different tissues, would potentially be more advantageous for an industrial

recovery and re-use of these waste fruits. The detailed composition of each sample listed in Table S1 has been reported in Tables S2-S6 for the mineral content and in Tables S8-S10 for the phenolic amount.

3.1. Mineral content

OFI fruit have been reported as a good source of minerals, namely potassium, magnesium, calcium and sodium, with a wide variability in the literature (Silva, et al., 2021). This study aimed to consider several factors affecting mineral content, in particular production area and harvesting period. The objective was to gain an overview of the compositional variability of discarded fruits of typical Sicilian varieties, which included white, yellow and red-fleshed fruits. These data are useful to evaluate the quality of the raw material that can be used for a potential industrial application.

3.1.1. Mineral content in peels

The results showed that the most abundant minerals detected in all peels of OFI samples were potassium, calcium and magnesium (Fig. 2a), in accordance with previous studies (Albergamo, Potorti, Di Bella, Amor, Lo Vecchio, Nava, et al., 2022; El Kossori, Villaume, El Boustani, Sauvaire, & Mejean, 1998). The highest content was found for samples of Biancavilla of the second harvest of 2018, with average values of 36163.2 μg/g for potassium, 35053.5 μg/g for calcium and 6929.6 μg/g DW for magnesium. In particular, the sample 18EtAc showed the highest content for all these three minerals, as reported in Table S2. These data are in agreement with those in the literature, in which values of 3430 mg/100 g, 2090 mg/100 g and 322 mg/100 g DW are reported respectively for potassium, calcium and magnesium in peel samples of prickly pear fruit (El Kossori, Villaume, El Boustani, Sauvaire, & Mejean, 1998). Also in the recent study of Albergamo et al. (2022) similar values were found for potassium (1820 mg/100 g DW) and magnesium (345 mg/100 g DW) in peels of Tunisian OFI. No significant differences were observed for potassium and calcium between the two cultivation areas (Biancavilla and San Cono) for either fruits of 2018 and 2019, while magnesium showed significantly higher values ($p < 0.05$) for Biancavilla samples in both the harvesting years and periods (Fig. 2a). Considering the values reported for each farm, significantly greater amounts were found for potassium, calcium and phosphorus during the second harvest of 2018, both for farms of Biancavilla and San Cono (Table S2). The same trend was not confirmed for the fruits collected in 2019 (Table S3).

Other elements, also important in terms of supplying mineral balance in body and functional effects, are present. High values of phosphorus are recorded, particularly in the peel samples of the second harvest of 2018, with average levels ranging from 1176 to 1339.7 μg/g DW for Biancavilla and San Cono, respectively. In addition, microelements such as manganese, iron, zinc and copper are present in lower amounts. Only in a few cases significant differences were found in the content of these minerals in the peels of the two different districts (Fig. 2a).

Of the non-nutritive microminerals, aluminum has no known specific biological function in humans but high exposure to this metal can result in adverse health effects such as neurodegenerative diseases (Filippini, Tancredi, Malagoli, Cilloni, Malavolti, Violi, et al., 2019). Diet is the primary source of exposure and in our samples the highest levels were found in peels of the second harvest of 2019 of San Cono region (average content of 94.0 μg/g DW, Fig. 1a). These values were higher than those found in the literature so far (Kalegowda, Haware, Rajarathnam, & Shashirekha, 2015), although lower than the tolerable weekly intake (TWI) of 1.00 mg/kg of body weight per week reported by EFSA (EFSA, 2008).

Among the microminerals, the presence of barium was also assessed. This element reached higher average values in the samples from the second harvest of 2018 (46.5 μg/g DW) and its concentration was significantly higher in the samples from Biancavilla for both the harvesting periods of 2018 and 2019. Compared to previous studies (Kalegowda, Haware, Rajarathnam, & Shashirekha, 2015), we found higher values for this element in the peel samples, attributable to a higher presence in the soil and thus a higher uptake by the plant.

3.1.2. Mineral content in seeds

Large differences were observed between the mineral content of the peel and seed samples. These latter showed lower content with a maximum value of 2046.2 μg/g DW, observed for calcium in San Cono samples collected during the second harvest of 2019 (Fig. 2b). In this tissue the most abundant minerals are calcium, potassium, magnesium and also phosphorus. As reported by El-Mostafa et al. (2014), remarkable is also the presence of large quantities of sodium, that showed average values of 95.1 and 131.9 μg/g DW respectively for samples of 2018 and 2019, in agreement also with Albergamo et al. (2022) who reported values of 14.09 mg/100 g DW in seeds from Tunisian OFI.

No significant differences were observed for calcium content between the two districts in 2018 and 2019 (Fig. 2b), while significant differences were found for potassium for the first harvest of 2019 where Biancavilla samples showed higher values compared with San Cono ones. Magnesium showed statistically greater values for San Cono samples of the first harvest of 2018, while the opposite trend was recorded for the second harvest of 2019. Phosphorus showed average values of 1607.7 and 1287.7 μg/g DW for 2018 and 2019 respectively, in agreement with quantities reported by El Kossori et al. (1998); this content was around double in the seed compared to the peel extracts. Significant greater values were reported for phosphorus in San Cono samples compared with Biancavilla ones for the first harvest of 2018, and lower contents in the same period of 2019.

Microelements such as iron and zinc were present in the seeds in similar quantities as in the peel samples (Fig. 2b). Between Biancavilla and San Cono districts, only the samples of the first harvest of 2019 showed significant differences in the content of these two microelements, with greater levels detected for the Biancavilla samples.

Manganese was present in seeds with an average value of 20.5 μg/g DW, a lower content than in the peel. Significant higher amounts were observed for San Cono samples than Biancavilla ones, for both the harvesting periods of 2018 and for the second harvest of 2019. Aluminum and barium are present in very low quantities (below 4 μg/g DW) in this tissue.

3.1.3. Mineral content in whole fruits

As previously observed for peel and seed tissues, the most abundant minerals in the whole fruits of OFI were potassium, calcium, magnesium and phosphorus (Fig. 2c). The average potassium content ranged between 14501.4 and 16471.5 μg/g DW for the second and the first harvest of 2019, respectively. These levels are lower than those found in peel, although in the same order of magnitude. Stronger differences were found with the concentrations reported in seeds. No significant differences were observed for potassium content between the two origin regions. The average calcium content ranged from 9819.6 and 12560.6 μg/g DW respectively for the second and first harvesting period and no significant difference were highlighted between the two districts. For both the harvesting periods, magnesium levels were found significantly higher in the sample of Biancavilla compared to those of San Cono. Literature does not report data for whole fruit of OFI, but several data are available for the pulp. Diaz-Medina et al. (2007) in OFI peeled fruits found 1583 μg/g of potassium and 263 μg/g of calcium. In the review of El-Mostafa et al. (2014) potassium was again reported as the most abundant element in pulp of OFI fruits showing contents of 1610 μg/g; values of 276 μg/g and 277 μg/g are reported for calcium and magnesium, respectively. Similar concentrations were shown by Belviranli et al. (2019) for potassium and calcium in pulp of prickly pear fruits harvested in different areas of Turkey, with amounts from 1908.1 to 3981.9 μg/g, and from 136.8 to 1224 μg/g, respectively. Recently, Mottese et al. (2018) confirmed that potassium, calcium and magnesium were the most abundant minerals in the pulp of OFI fruits from different areas in Sicily and showed that samples from Biancavilla were those

with the highest amount of these minerals (1827.5 μg/g for K, 619.3 μg/g for Ca and 628.9 μg/g for Mg) compared to the other two Sicilian areas of Roccapalumba and Pezzolo. In all these studies focused on the pulp, the mineral content is much lower than that found in our whole fruit samples, and this is certainly due to the presence of the peel in our samples.

The microelements manganese, iron, sodium, zinc and copper are present in the whole fruit samples with average concentrations of 135.5 μg/g, 22.5 μg/g, 16.2 μg/g, 13.7 μg/g and 4.4 μg/g DW, respectively. No statistically significant differences were observed for the content of these elements between the two regions of Biancavilla and San Cono during the second harvest of 2019, while iron and sodium showed higher values for the samples from Biancavilla harvested in the first harvest of 2019. Of the non-nutritive microminerals, aluminum showed values of 25.7 μg/g for the first harvest, while a higher content was observed for the second harvest for which San Cono samples showed average values of 140.3 μg/g. This could be due to a higher concentration of this metal in the soil at that time and in that area. Finally, barium showed a similar average content between the first and second harvest of 2019, although a higher content was found for the Biancavilla samples of the second harvest. Even for these trace elements, the concentration reported in the literature is lower, as these studies referred to the tissue of the pulp only, and not to the whole fruit including the peel and seeds.

3.2. Phenolic compounds

3.2.1. Phenolic compounds in peel extracts

The chromatographic profile at 350 nm of a peel extract - chosen as example – is reported in Fig. S1a. Seventeen compounds were detected and flavonoids were the most relevant class of phytochemicals identified, in accordance with previous studies (Farag, Sallam, Fekry, Zaghloul, & El-Dine, 2020; Melgar, et al., 2017; Mena, et al., 2018).

The identification of phenolic compounds was carried out by comparison with the analytical standards when available, retention times and by comparing their mass spectral characteristics with the literature. In particular, nine flavonoids (compounds 4–8, 11, 13–16), two phenylpyruvic acids (compounds 1 and 2) and three hydroxycinnamic acids (compounds 3, 9 and 17), were identified, as reported in Table S7. Most of these compounds have been previously found by other authors in fruits of *Opuntia* spp (Amaya-Cruz, Perez-Ramirez, Delgado-Garcia, Mondragon-Jacobo, Dector-Espinoza, & Reynoso-Camacho, 2019; Farag, Sallam, Fekry, Zaghloul, & El-Dine, 2020; Melgar, et al., 2017; Mena, et al., 2018) and are reported to be very important for their activities. In particular, the study of Gòmez-Maqueo et al. (2019) reported the antioxidant, antihyperglycemic and anti-inflammatory activity of hydroalcoholic extracts of prickly pear fruit and the contribution of some of the isolated phenolic compounds. It was observed that prickly pear peel extracts had the highest antioxidant and antiinflammatory activity mainly due to the high isorhamnetin glycoside content. Total phenolic acids, total flavonoids, and total phenols content in peel for the two harvesting periods of each of the samples of 2018 and 2019 are reported in Table S8. Differences among the samples were observed: the total content of these compounds varied between 0.87 and 3.53 mg/g DW for the 2018 and from 0.56 to 1.35 mg/g DW for 2019, highlighting average values more than double for 2018 compared to 2019. For both the years, the average total phenolic content in the fruits of the first harvesting period (end of August) was respectively 25% and 20% higher than that observed for the second harvest (late-October). Indeed, most of the samples of 2018 showed statistically significant differences on the content of total phenolic compounds between the two harvesting periods, and for eight samples the first harvest was found to be richer than the second one, while only three samples (18EtMa and the two commercial 18EtCOM and 18ScCOM) showed an opposite trend. Only three samples, 18EtCa, 18ScSp and 18ScGr, did not show significant differences in total phenolic content between the two harvests of 2018. The same trend was observed for the 2019 samples, which confirmed a higher content of total phenolic compounds for the first harvest for 4 out of 6 samples.

The flavonoids were the most abundant compounds in all the peel extracts, representing about the 91% of the total phenols. Their content ranged between 1.32 and 3.26 mg/g DW for the first harvest of 2018 and from 0.74 to 3.00 mg/g DW for the second harvest (Table S8); as observed for the total phenols, lower values were registered for both the harvesting periods of 2019, which ranged from 0.56 to 1.23 mg/g DW and 0.47 to 1.08 mg/g DW for the first and the second harvest, respectively.

Regarding the phenolic acids, their content varied from 0.09 to 0.28 mg/g DW in 2018 and from 0.09 to 0.17 mg/g DW in 2019. Seven samples showed a higher concentration of this class for the first harvest of 2018, while the second harvest of the same year was found richer only for three samples (18EtMa, 18EtRa and 18ScDa). Three of 2019 samples (19EtCr, 19EtMa and 19EtRa) showed higher content of phenolic acids during the second harvest, while only one (19ScLa) showed higher concentration for the first one.

To summarize the main results for phenolic compounds in OFI fruits, the average values were compared in Fig. 3, which reported the content of the two classes of phenolic compounds in the samples from the two areas of origin, for 2018 and 2019. No significant differences were observed for the content of these compounds for the peel sample of 2018, although a trend of higher metabolite concentrations in samples from San Cono has been noted. Significant differences were observed in flavonoid and total phenol contents for samples from the second harvest of 2019.

The variation in phenolic contents of OFI fruits harvested at different times might be due to the variations in environmental conditions (temperature, day length, day light, humidity and rainfall) as already reported in the literature for OFI fruits. For instance, Al Juhaimi et al. (2020) reported the optimum harvesting time to retain high quantities of most phenolic compounds is 1st July considering the period between 15 June and 15 August. In addition, the chemical changes during maturation process of the fruit could also lead to the variation in the total phenolics of prickly fruits harvested as different times. In our case this trend was observed in peel tissue considering the two different harvesting years, as better discussed in paragraph 3.4.

3.2.2. Phenolic compounds in seeds extracts

The detection of phenolic compounds contained in the OFI seeds was performed applying the same analytical method used for peel. The HPLC separation revealed a high complexity in the seed phenolic composition (Fig. S1b), as already reported in a previous study (Chougui, Tamendjari, Hamidj, Hallal, Barras, Richard, et al., 2013). Table S7 reported the mass data of the detected phenolic compounds. Compound 1 exhibited the molecular ion of m/z 517 in MS (-) and the loss of only one fragment of 288 Da from the $[M-H]^-$ ion suggesting the presence of a disaccharide moiety composed by two hexoses. The same fragmentation pattern was already reported in previous studies for feruloyl-sucrose (Chahdoura, Barreira, Barros, Santos-Buelga, Ferreira, & Achour, 2015; Chougui, et al., 2013; Torrico, Nguyen, Li, Mena, Viejo, Fuentes, et al., 2019). Ferulic acid was detected in the extract in trace amount, as confirmed by the analysis of the reference standard. The presence of a feruloyl derivative (compound 9) was also confirmed, as already reported by Chougui et al. (2013).

All the compounds listed in Table S7 were quantified in seed extracts and data on the total phenolic acids content for each sample of 2018 and 2019 are given in Table S9. The concentration of these compounds was very similar between the two years, ranging from 0.11 to 0.25 mg/g DW. Only some samples of 2018 showed statistically significant differences between the two harvesting periods, in particular the four discarded samples 18EtVa, 18EtCa, 18EtAc, 18EtMa and the commercial EtCOM showed a higher phenolic content for the second harvest, while the 18ScLa and 18ScDa samples highlighted an opposite trend. On the other

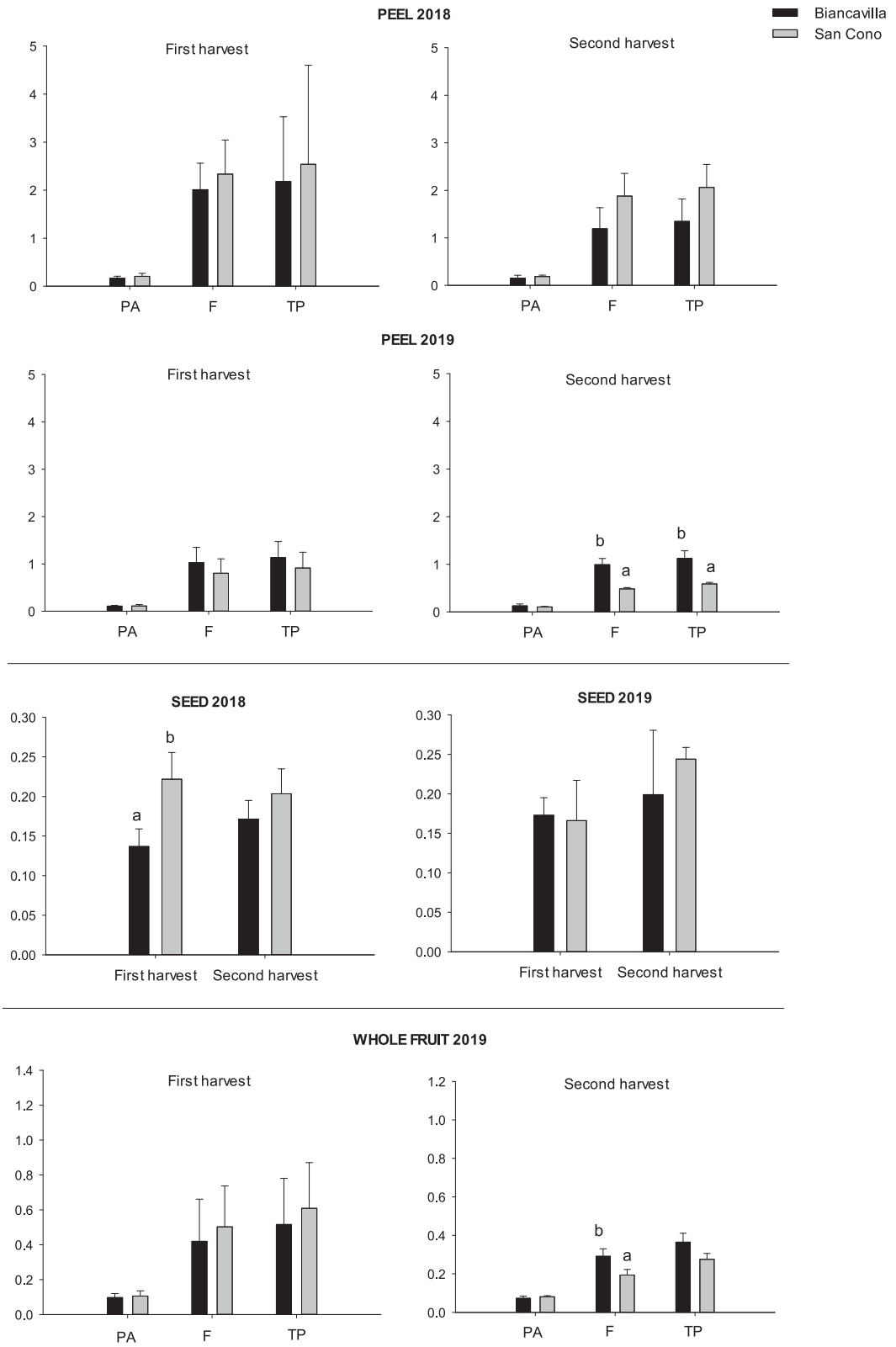

Fig. 3. Phenolic content (PA, phenolic acids; F, flavonoids; TP, total phenols) in peel, seed and whole fruit samples from Biancavilla and San Cono of 2018 and 2019. Data are expressed as mg/g DW ± standard deviation. For each pair of samples, the letters are reported only when the differences are significant ($p < 0.05$).

hand, all the 2019 samples showed significant differences between the two harvests, with the majority of samples showing a higher phenolic acid content for fruits of the second harvesting period.

The average phenolic content in the seed samples from the two different districts in Sicily was compared in Fig. 3 for 2018 and 2019. Significant differences were observed only for samples of the first harvest of 2018, with the higher content of phenolic compounds found in the seed samples from San Cono.

3.2.3. Phenolic compounds in whole fruit extracts

The HPLC analysis of the fruit extracts for each sample of 2019 highlighted the same qualitative profile obtained for the peel samples. The respective quantitative results are summarized in Table S10. Considering both the first and the second harvest, the total phenolic content ranged from 0.25 to 0.83 mg/g DW; statistically significant differences were observed for five samples, which showed a higher concentration for the first harvest. Only the sample 19EtCr did not show significant differences in the total phenols content between the two periods. As observed for peel extracts, flavonoids are the most abundant compounds, representing about the 79% of the total content, with values from 0.20 to 0.68 mg/g DW for the first harvest and from 0.17 to 0.34 mg/g DW for the second one. Significant higher values were found for the first harvesting period in five samples. Again, the sample 19EtCr did not show differences between the two periods.

The phenolic acids content varied from 0.07 to 0.12 mg/g DW and from 0.06 to 0.09 respectively for the first and the second harvesting period. All the samples showed statistically significant differences between the two harvests; particularly, five samples showed higher value of phenolic acids for the first harvest, while only the 19ScBa sample registered the opposite trend.

Fig. 3 reported the content of the different classes of phenolic compounds in the whole fruit extracts of 2019 considering the two Sicilian areas of origin (San Cono and Biancavilla), where the sample were produced and collected. As observed for the peel tissue, significant differences were recorded for samples from the second harvest in which higher flavonoid concentrations were observed in samples from Biancavilla.

Allegra et al. (2015) reported that the content of polyphenols of minimally processed fruit pulp of OFI harvested at two different times (27th August and 16th October) was two times higher in the late fruit than that in summer fruit and that the harvest season strongly influenced the shelf-life of ready-to-use cactus pear fruits packaged under passive atmosphere.

Our data showed significant differences in phenolic content between 2018 and 2019 only for the peel tissue, while no differences were found between summer and winter periods, as discussed in more detail in section 3.4.

3.3. Antioxidant activity of sample of 2019

The ability of the hydroalcoholic extracts from peels, seeds and whole fruits of 2019 to scavenge free radicals *in vitro* was assessed using DPPH assay. Similar mean values, expressed as μmol gallic acid/g DE, were observed for peel and whole fruit samples, while significant lower values were detected for seed samples (Fig. 4a). Indeed, one-way ANOVA results showed significant differences between the antioxidant activity of peel and whole fruits samples from that of the seed ($p < 0.05$), while no significant differences were observed between peel and whole fruit samples. Furthermore, the results showed no statistical differences for the antioxidant activity between the two areas of origin (Biancavilla and San Cono) and the two harvesting periods (data not shown).

As a multivariate method that is a successful tool in calibration, validation and prediction, the partial least squares (PLS) regression, which is a classic multiple linear regression model, was applied. PLS regression showed a good correlation between antioxidant activity and the mineral and phenolic contents, as showed in Fig. 4b.

Phenolic compounds possess an ideal structural chemistry for free radical scavenging activity and contribute to the overall antioxidant potential of plants mainly due to their redox properties. In many studies a linear correlation between antioxidant activity of herbal extracts and their phenolic content are reported. To confirm this, the concentration of the eleven minerals and the total phenolic content were analysed using the regression coefficients for original data and VIP (variable importance in the projection) values, which established the importance of the independent variables highlighting those showing the high values.

In our results, total phenolic content, iron and to a lesser extent copper showed high VIP and regression coefficient values and therefore can be considered the more important variables. Higher total phenolic content and also higher iron concentration corresponded to the highest antioxidant activity, confirming a positive linear correlation with the antioxidant activity of OFI extracts. These results confirmed that not only the phenolic fraction exerts antioxidant activity, but also a mineral as iron, presumably in its form Fe^{2+}.

3.4. Chemometric analysis on mineral and phenolic contents

In order to better evaluate all the results collected in this study, a chemometric analysis by PCA and LDA was used to verify the possibility of discriminating the samples based on the different tissues or geographical area of origin, harvesting period or year.

The analysis was carried out considering the content of the eleven minerals (Ca, K, Mg, Na, P, Cu, Fe, Mn, Zn, Al, Ba) and the total phenolic content (TP) in all the samples analysed. The ninety-two samples were firstly classified by the harvesting year (2018 and 2019), the harvesting period, the geographical origin (Biancavilla and San Cono) and by the different part of fruit (peel, seed and whole fruit), and then subjected to PCA analysis.

The scree plot in Figure S2 shows that the elbow point is reached with approximately four principal components (PCs) with a total

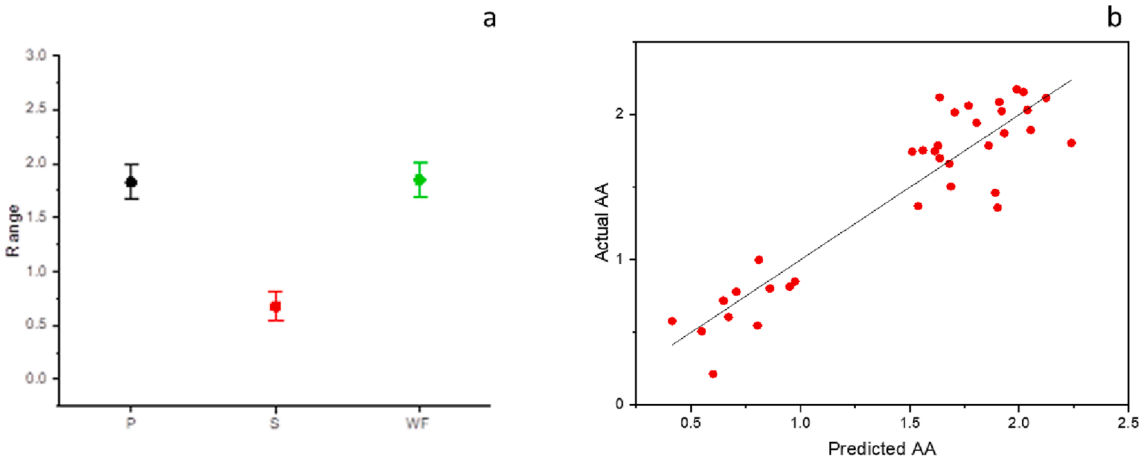

Fig. 4. (**a**) Mean values of antioxidant activity by DPPH assay (expressed as μmol gallic acid/g DE) of the different parts of the OFI fruit (P, peel; S, seed, WF, whole fruit); (**b**) correlation plot for the prediction of antioxidant activity and mineral and phenolic content using PLS regression model.

explained variance of 82%; beyond these four PCs the variance explained by an additional component was irrelevant. The score plots in Fig. 5 reported the 92 samples in two-dimensional space defined by the first two PCs that explain 67% of the total variance according to harvesting year (a), harvesting period (b), geographical origin (c) and different part of fruit (d). Fig. 6a, 6b and 6c reported that the samples are randomly distributed showing that the content of the eleven minerals and of the total phenols do not discriminate by harvesting year, harvesting period and geographical origin. On the contrary, three groups are distinguished in the score plot of Fig. 5d; peel samples are separated from seed and whole fruit samples showing that the minerals and the phenols concentrations discriminate by the three different tissues. Moreover, peel samples are characterized by higher values of all minerals determined, with the exception of sodium, phosphorus and zinc (biplot in Fig. 5d). On the other hand, sodium is present in higher amount in seed samples. The whole fruit samples are placed in an intermediate zone between the two groups.

To confirm the ability of the analytical results to discriminate among the various groups, a LDA (linear discriminant analysis) using four PCs was performed. The confusion matrices obtained from LDA regarding the distribution by harvesting year and period, confirmed the results previously obtained with a random distribution of the data and no homogeneous groups identified with good probability. In particular, the confusion matrix regarding the distribution by harvesting year showed 53.6% and 69.4% of samples correctly placed, as well as 67.4% and 54.4% were correctly classified according to harvesting period. The distribution by geographical origin showed that the chemical data were able to place with sufficient precision only samples from San Cono (89.1% of samples correctly placed), while the same was not observed for the sample from Biancavilla (only 52.2% of samples correctly placed).

Better classification was obtained for the confusion matrix regarding the distribution by different part of fruit that placed in perfectly homogeneous groups seeds and whole fruits (both 100%), with peel samples correctly classified up to 92.5% and only the 7.5% incorrectly placed in the whole fruit group. As reported in Figure S3, which showed the 92 samples in the space of the two canonical variables of the discriminant function, the content of minerals and total phenols significantly discriminate the different parts of the fruit.

PCA and LDA analysis were also performed considering separately the 40 peel samples, the 40 seed samples and the 12 whole fruit samples.

The results obtained by a LDA from 5 PCs (Fig. 6), showed that data from the whole fruits were able to perfectly classify samples by origin (100% of samples correctly placed), while no significant results were obtained for the harvesting period, as only 50% of the samples were correctly placed.

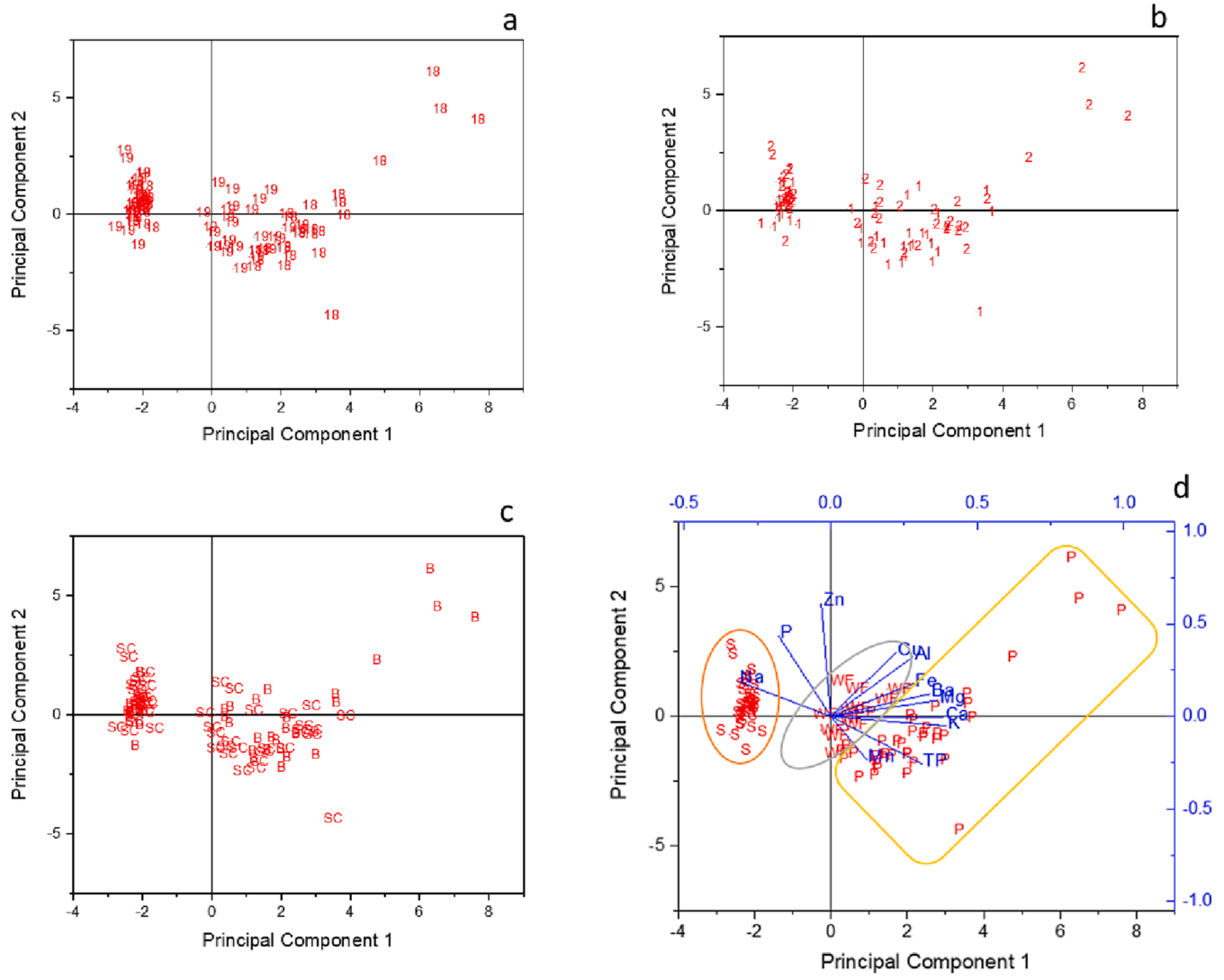

Fig. 5. 2D Score plots of the 92 OFI samples categorized according to harvesting year (**a**), harvesting period (**b**), geographical origin (**c**) and different parts of fruit considered (**d**).

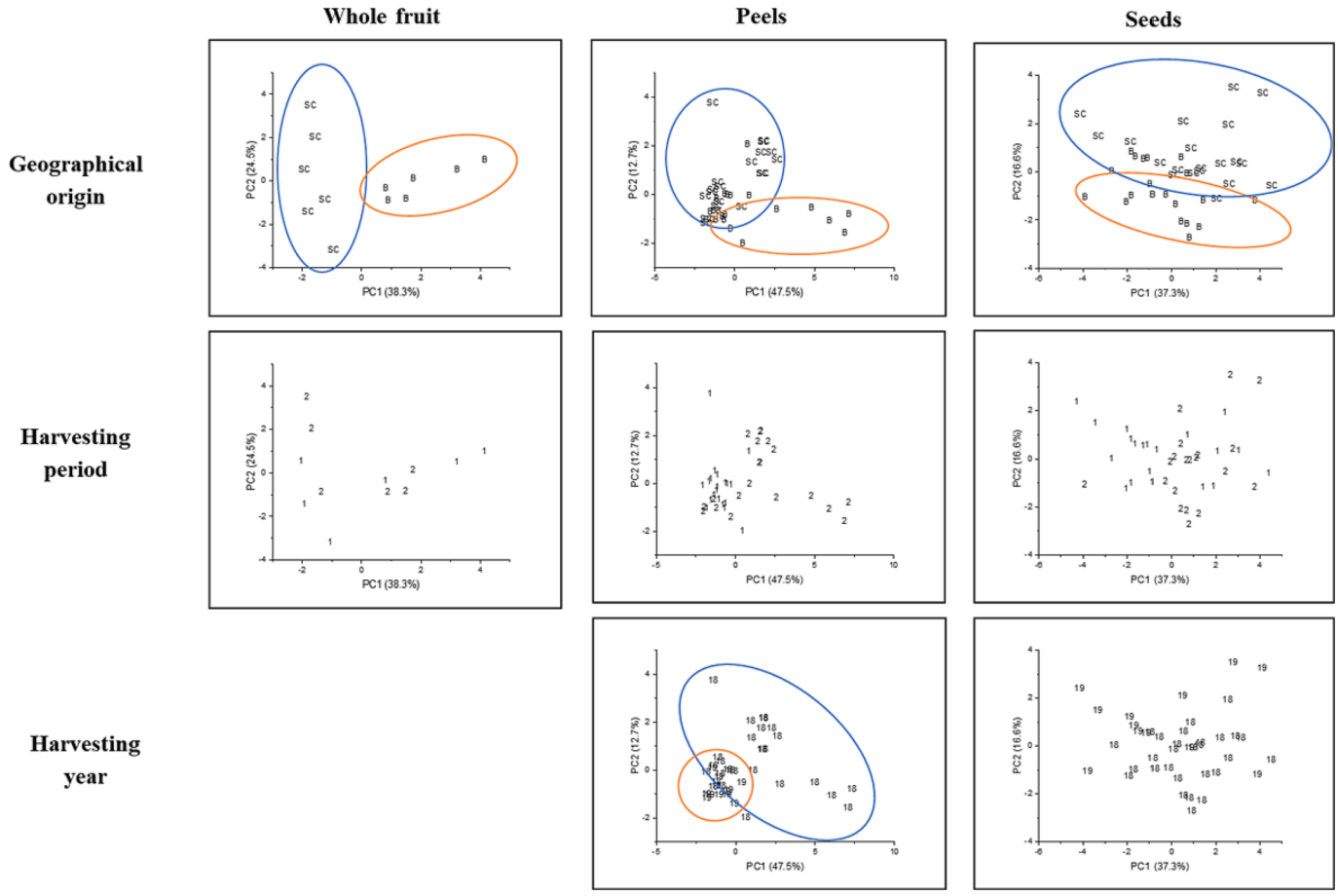

Fig. 6. 2D Score plots of the whole fruit, peel and seed samples categorized according to geographical origin, harvesting period and year.

The results for the peel tissue obtained by a LDA from 7 PCs showed significant results for geographical origin and harvesting year. Regarding the former, the samples are largely placed (90 and 95%) in the corresponding group, while the model correctly placed the 85.7% and 100% of the samples according to harvesting year.

The LDA from 6 PCs on seeds samples showed the geographical origin was statistically significant (samples correctly classified for 85%), while the harvesting year and period did not show significant results (respectively 78%-83% and 75–80% of the samples correctly placed).

Previously, Mottese et al. (2018) reported that the mineral profile of prickly pear pulps showed a strong correlation with the area of origin; in particular, Biancavilla samples were linked to the peculiar environmental conditions due to the geological composition of the volcanic production area. In our study a correlation with the geographical provenance was found for OFI peel and seed, although the best correlation was observed for whole fruit samples (Fig. 6).

Albergamo et al. (2018) demonstrated an effectiveness of multivariate methods, in particular PCA, stepwise CDA, and PLS-DA, in the chemometrical characterization of prickly pear produced in different Sicilian areas.

Regarding the harvesting year, our data showed significant results only for peel tissue. On the other hand, no correlation was observed in the mineral and phenolic content between the two harvesting seasons, showing that these factors do not always play a significant role.

4. Conclusions

The loss of high amount of material with morphological parameters not accepted for the market of fresh fruits represents a real problem to be faced. Little information on the annual and seasonal composition variability is reported for these fruits, and more knowledge on this aspect can be particularly important for their industrial reuse. The results reported in this study showed that no significant differences in mineral and total phenolic content were observed between the discarded and commercial fruits. An interesting result in view of their possible industrial use was the low variability in the composition of the discarded fruits. They are a good source of minerals, such as potassium, calcium and magnesium and the peel showed the highest concentration. Flavonoids are the most relevant phenolic class in peel and whole fruit. It should be underlined that through PCA and LDA the minerals and phenolic compounds of whole fruits and peel are able to differentiate the geographical provenance of OFI fruits, suggesting their potential use as "traceability markers". On the opposite, the variability related to the season did not significantly affect the composition. These data on the composition of discarded OFI fruits may be useful in highlighting the potential role of this waste material as a source of novel food ingredients for fortification and/or new product formulation.

CRediT authorship contribution statement

Maria Bellumori: Conceptualization, Formal analysis, Investigation, Data curation, Writing – original draft. **Marzia Innocenti:** Supervision, Writing – review & editing. **Luisa Andrenelli:** Formal analysis, Data curation. **Fabrizio Melani:** Formal analysis, Investigation, Data curation. **Lorenzo Cecchi:** Formal analysis, Data curation. **Gaetano Pandino:** Methodology, Formal analysis, Conceptualization. **Giovanni Mauromicale:** Methodology, Conceptualization. **Stefano La Malfa:** Methodology, Conceptualization. **Nadia Mulinacci:** Resources, Supervision, Writing – review & editing, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2023.136756.

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