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Maximizing the sustainable use of pomegranate, orange, and banana peel wastes as natural antioxidant resources

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Abstract

One way to sustainably use environmental resources is the valorization of industry by-products. In the present study, seven phenolic compounds: p-coumaric acid, catechin, chlorogenic acid, caffeic acid, ferulic acid, punicalagin (A+B mixture), and gallic acid, were quantified using High Performance Liquid Chromatography (HPLC) in extracts obtained from orange (OR), banana (BN) and pomegranate (PG) peels derived from juicing by-products. Additionally, the antioxidant capacity of the peel extracts and the total phenolic content (TPC) were determined. All samples were extracted with 50% aqueous ethanol using three different solid-to-solvent ratios: 1 g dry peel (DP)/40 mL solvent (OR-40, BN-40, PG-40), 1 g DP/50 mL solvent (OR-50, BN-50, PG-50), and 1 g DP/60 mL solvent (OR-60, BN-60, PG-60). According to the results, the highest antioxidant capacity was detected in PG-60, with a value of 0.62 ±0.060 mmol Trolox Equivalent Antioxidant Capacity (TAE)/g DP. Regarding TPC, the Folin–Ciocalteu assay revealed the highest values for OR peel in the OR-40 (42 ±2.9 mg gallic acid equivalents (GAE)/g DP), for BN peel in BN-40 (51 ±2.5 mg GAE/g DP), and for PG peel in the PG-60 (380 ±19 mg GAE/g DP). These findings highlight ways to optimize the potential of fruit peel waste as a promising, low-cost resource of bioactive compounds, supporting sustainable practices and contributing to the development of value-added products in the framework of a circular economy.

Keywords: waste valorization; pomegranate; banana; orange; peel waste; industry by-products; polyphenols

Introduction

Fruit juice plants generate significant amounts of solid and liquid waste, making proper management essential to prevent serious environmental impacts. During juice production, pomegranate peels constitute about 26–30% of the total fruit weight (Mo et al., 2022). Citrus peel waste, such as orange peels, represents around 50–60% of the total fruit mass, making it one of the main by-products of juice processing (Demie et al., 2023). Banana peels constitute about 35–40% of the fruit's weight. In 2017, banana by-product production was

estimated at 101 million tons, mainly peels (Campos et al., 2020). Solid wastes from fruit juice plants, such as fruit peels, pits, and pulp, is rich in organic matter and can be repurposed for animal feed, fertilizers, and biogas production through anaerobic digestion. Energy recovery through biogas production provides a sustainnable solution, reducing greenhouse gas emissions and dependence on fossil fuels (Selvarajoo et al., 2022). Additionally, pectins and other chemical compounds extracted from this waste type may be used in different applications by the pharmaceutical and cosmetics indu-

istries (Pedro et al., 2024; Alnaimy et al., 2017). Liquid waste, primarily from the fruit washing process, contains high concentrations of organic compounds and nutrients, which, if discharged untreated, can pollute water receivers. Treatment processes such as chemical flocculation, stabilization ponds, and biological treatment with activated sludge, help mitigate environmental impacts (Pedro et al., 2024; Zema et al., 2019). Effective waste management in juice production can therefore support the circular economy and contribute to sustainable development. Polyphenols stand out among the most valuable compounds found in the solid by-products of a juice plant due to their high bioactive potential and beneficial properties. These compounds exhibit significant antioxidant, antimicrobial and anti-inflammatory properties, which makes them attractive for various applications in the pharmaceutical, food, and cosmetic industries (Kandemir et al., 2022). Phenolic compounds constitute one of the largest and most widely distributed groups of secondary metabolites in plants. Polyphenols are involved in defense against different types of stress, and are known to protect against UV radiation. Additionally, they substantially contribute to the organoleptic properties of plants, foods, and cosmetics, such as color and flavor. Polyphenols represent a vast group of at least 10,000 different compounds, containing one or more aromatic rings with one or more hydroxyl groups attached (Brglez, et al., 2016). They are classified into flavonoids and non-flavonoids based on the number of phenolic rings and their intramolecular connection at their structure (Swallah, et al., 2020, Truzzi et al., 2021, Manach, et al., 2004) (Fig. 1). Non-flavonoids include phenolic acids, stilbens and lignans. Phenolics are classified into hydroxycinnamic and hydroxybenzoic acids (Brglez, et al., 2016) based on the position of the hydroxyl group. The most common hydroxybenzoic acids are vanillic, syringic and gallic acids and among the hydroxycinnamic acids the most notable are caffeic, p-coumaric, and ferulic acid (Tsao, 2010). Stilbens have a basic C6-C2-C6 skeleton, where two aromatic rings are linked by an ethylene bridge (Zhang et al., 2021). Resveratrol (3,5,4'-trihydroxystilbene) is one of the most common stilbens found in red wine, grapes, peanuts and berries (Oluwole et al., 2022). Lignans are natural compounds derived from the biosynthetic pathway of shikimic acid. They consist of a basic skeleton with two or more phenylpropanoid units. They are mainly found in cereals, fruits, flax seeds, and vegetables (Hazafa et al., 2021, Cui et al., 2020). Flavonoids are the most widely distributed phenolic

compounds in plant-based foods and contribute significantly at fruit and vegetables flavor and color. They consist of two benzene rings connected by a pyran ring. Based on the oxidation state of the central C ring, flavonoids are divided into flavonones, flavanols, flavonols, isoflavones, flavones and anthocyanidins (Swallah, et al., 2020, Tsao, 2010). Tannins, which are among the non-flavonoids, are one of the most important groups of phenolic compounds. They can be classified into hydrolysable and condensed tannins (procyanidins). Hydrolysable tannins are derivatives of gallic acid while catechins are also included in tannins. These substances are often responsible for the black color and stewed taste of the fruit. They are commonly found in apples, peaches, grapes, almonds, coffee, tea (Abbas, et al., 2016). Several ways of extracting polyphenols from fruit peels have been proposed such as solvent extraction (Díaz et al., 2021, Haya et al., 2022), ultrasound assisted extraction (UAE) (Pan et al., 2011, Paula, et al., 2019), microwave assisted extraction (MAE) (Bouras et al., 2015, Dahmoune et al., 2014), supercritical fluid extraction (SFE) (Ameer et al., 2017, Shi et al., 2005), pulsed electric field (PEF) extraction (Maza et al., 2020, El Kantar et al., 2018),

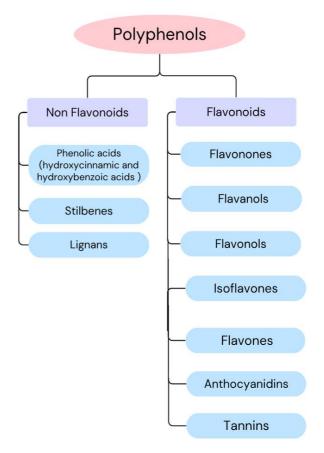


Figure 1. Classification of polyphenols.

enzyme assisted extraction (EAE) (Gligor et al., 2019, Benucci et al., 2017). In the present study, extraction of polyphenols from banana, orange and pomegranate peels was carried out using 50% ethanol aqueous solution as extraction solvent. Ethanol is a green solvent produced by fermenting renewable sources such as sugar and starch. It is low-cost, biodegradable, eco-friendly, and is known as a polar protic solvent, similar to water (Mondal et al., 2024). The fruits used in the present study were selected as they are rich in phenolic compounds known for their antioxidant, antimicrobial and therapeutic properties. Specifically, bananas contain flavonoids that act as antioxidants, neutralizing free radicals derived from reactive oxygen species (ROS), which are responsible for aging and various diseases. Flavonoids found in orange, such as hesperidin, narirutin and naringin, exhibit important pharmacological properties including antioxidant and anticancer activity (Luengo et al., 2013). The main phenolic compounds present in orange peel are hydroxycinnamic acids, such as caffeic, p-coumaric, ferulic and sinapic acids (Camacho et al., 2022). Pomegranate (Punica granatum L.) contains numerous bioactive compounds with many of them found in pomegranate peel, such as alkaloids, ellagic acid, and punicalagin among other ellagitannins, anthocyanins, flavonoids, tannins and other phytochemicals (Melgarejo Sánchez, et al., 2021, Pirzadeh, et al., 2020). In this study, the total phenolic content (TPC) and antioxidant capacity of extracts from orange, banana, and pomegranate waste fruit peels was determined. The primary objecttive was the detection and quantification of p-coumaric acid, catechin, chlorogenic acid, caffeic acid, ferulic acid, punicalagin (A+B mixture), and gallic acid using High Performance Liquid Chromatography (HPLC). Through the implementation of the research plan, juice industry waste will be evaluated as a poten-tial source of polyphenols, exploring possible reuse applications. The ultimate aim of the present study is the sustainable use of industrial waste resources.

Materials and Methods

Chemicals and reagents

Orange (Citrus sinensis (var. navel)) and pomegranate (Punica granatum) peels were obtained from LOUX MARLAFEKAS S.A., while bananas (Musa acuminata) were obtained from the local market. All chemicals and solvents were of analytical grade. Standards of chlorogenic acid hydrate (purity >98%), caffeic acid (purity >98%), p-coumaric acid (purity >98%), were obtained

from TCI (Tokyo, Japan). Gallic acid (purity ≥98%) was supplied by Sigma-Aldrich (St. Louis, MO, USA), punicalagin (A+B mixture) (purity ≥95%) was provided by PhytoLab Gmb & Co. KG (Germany) and ferulic acid (purity 99%) was purchased from ACROS Organics (Geel, Belgium). Ethanol (EtOH, purity ≥99.8%) was obtained from ThermoScientific (Geel, Belgium), and methanol (MeOH, purity ≥99.95%, LC-MS-Grade) was obtained from Carl Roth (Germany). Folin–Ciocalteu reagent, used for the determination of total phenolics, was supplied by VDWR Chemicals (France), and 1,1-diphenyl-2-picrylhydrazyl (DPPH) was purchased from Sigma-Aldrich (St. Louis, MO, USA).

Sample preparation

The fruit peels were freeze-dried (Zirbus Technologies) for 44 hours to ensure complete moisture removal. The dried peels were then ground into a fine powder using an electric grinder and sieved through an 800 µm sieve to achieve uniform particle size.

Solid-liquid extraction (SLE) of polyphenols from peel

The SLE extraction of phenolic compounds from each fruit peel was performed as follows. One gram (g) of dry fruit peel powder was mixed with 50% of ethanol/water at solid-to-liquid ratios of 1:40, 1:50, 1:60. The samples were subjected to ultrasonic extraction at 40°C for 1h using an ultrasonic bath (Raypa Ultrasonic Cleaner, 400 W, 40 kHz), followed by incubation for 24 h in a thermostatic shaking water bath to ensure exhaustive extraction. To achieve complete phase separation, the samples were centrifuged (Heraeus Kendro Laboratory) at 5000 rpm for 10 min. The supernatants were then filtered through a 0.2 µm PVDF syringe filter to remove any remaining impurities before analysis. The extracts were labeled according to the fruit source and extraction conditions. Orange peel extracts were designated as OR-40, OR-50, and OR-60, banana peel extracts as BN-40, BN-50, and BN-60, and pomegranate peel extracts as PG-40, PG-50, and PG-60. Each extraction condition was analyzed in triplicate (n=3). All extracts were stored at 4°C until further analysis.

Determination of total phenolic content (TPC) in the extracts

The TPC was determined using the Folin-Ciocalteu method (Soyollkham et al., 2011). The sample (0.5 mL) was mixed with 0.5 mL of Folin-Ciocalteu reagent and allowed to react for 3 min. Then, 2.5 mL of Na₂CO₃

solution was added to adjust the pH to 10, followed by the addition of 2.5 mL of distilled water. The mixture was vortexed for 30 sec and incubated in the dark at room temperature for 60 min. Absorbance was measured at 750 nm using a Lambda 35 spectrophotometer (±0.001 nm). Gallic acid was used as the reference compound, and the TPC was expressed as mg GAE/g DP.

DPPH (1,1-diphenyl-2-picrylhydrazyl) free radical scavenging assay

The antioxidant activity of orange, banana and pomegranate peel extracts was measured using the DPPH assay, following the method described by Marinova et al., (2011) with some modifications. A DPPH solution was prepared at a concentration of 2 x 10⁻⁴ M. Then, 2 mL of ethanolic extract was mixed with 2 ml of DPPH solution. The mixture was vortexed for 30 seconds and kept in the dark at room temperature for 30 min. Absorbance was measured at 517 nm using a Lambda 35 spectrophotometer (±0.001 nm). Results were expressed as mmol Trolox equivalents per gram of dry product (mmol TAE/g DP), based on a standard curve constructed using Trolox solutions of known concentrations. The RSC (Radical Scavenging Capacity) was calculated based on the percentage of DPPH radical scavenged (Gülçin et al., 2023).

$$\% RSC = \left(\frac{Ao - As}{Ao}\right) * 100$$
 [1]

where $A\theta$ and As are the absorbances in the absence and in the presence of antioxidant respectively.

High performance liquid chromatography analysis of phenolic compounds

Identification and quantification of phenolic acids and flavonoids in the extracts were performed using High Performance Liquid Chromatography (HPLC). Fruit peel extract samples filtered through $0.2\,\mu m$ PVDF syringe filter were injected into a reverse phase HPLC system (Shimadzu LC-20AD), C18 column (150 × 4.6 mm, 5 μ m particle size, 10 nm pore diameter). The mobile phase consisted of a gradient mixture of solvent A (distilled water with H_3PO_4 0.01%) and solvent B (methanol). The following gradient program was used for the separation of flavonoids and phenolic acids: 10% B (0-6 min), 20% B (6-12 min), 30% B (12-30 min). The analyses were conducted at a flow rate of 1 mL/min with Diode-Array Detection (DAD) set at 250, 271, 280, 319 nm. A standard mixture of phenolic

compounds was prepared in the concentration of 5 mg/L. Each sample injection volume was 20 μ L. Peaks were identified based on the retention time of those of the standard mixture of commercial phenolic standards. The quantification of each phenolic compound was carried out at its individual maximum absorption wavelength, determined from the DAD spectra, and in combination with the calibration curve obtained from the multi-standard solution.

Results and Discussion

Total Phenolic Content (TPC)

Using the Folin-Ciocalteu method, a total of twentyseven (27) extracts were analyzed to quantify the TPC. The extraction yield (%, w/w, dry basis), defined as the percentage of dry extract obtained relative to the initial dry weight of the fruit peel (% w/w), varied across the different samples. The highest yield was observed for pomegranate peel extracts, reaching 38% at a solid-tosolvent ratio 1:60 (PG-60). In comparison, banana and orange peel extracts exhibited yields of 5.1% and 4.2%, respectively, both at a 1/40 solid-to-solvent ratio (BN-40 and OR-40). The TPC ranged from 27 ± 6.0 to 380 ±19 mg GAE/g DP (Table 1). The highest TPC values were observed in pomegranate extracts, with the PG-60 showing a concentration of 380 ±19 mg GAE/g DP. These findings agree with Feng et al. (2022), who reported a maximum TPC value of 350 ±27 mg GAE/g DP. Similarly, Russo et al., (2018) documented TPC values ranging from 90 ±0.61 to 24 ±1.4 mg GAE/g DP for different pomegranate cultivars extracted with a 1:1 methanol-water solvent system. For orange extracts, the highest total phenolic concentration was observed in OR-40, yielding 42 ±2.1 mg GAE/g DP. This result is consistent with Charunivedha et al., (2024), who investigated the extraction of polyphenols from orange peels and reported a maximum TPC of 38 mg GAE/g DP under optimized conditions (53% ethanol, 51°C, and 96 min extraction time). Regarding banana extracts, the highest TPC was found in BN-40, achieving 51 ±2.5 mg GAE/g DP. This aligns with the findings of Chaudhry et al., (2022), who used ultrasound-assisted extraction with different polar solvents and identified ethanol (50%) as the optimal extraction medium, yielding a TPC of 31 mg GAE/g DP. Furthermore, additional pomegranate extracts with DP/solvent ratios of 1/40 and 1/50 demonstrated similarly high polyphenol concentrations (Fig. 2) of 320 \pm 16 mg GAE/g DP and 330 \pm 16 mg GAE/g DP, respectively.

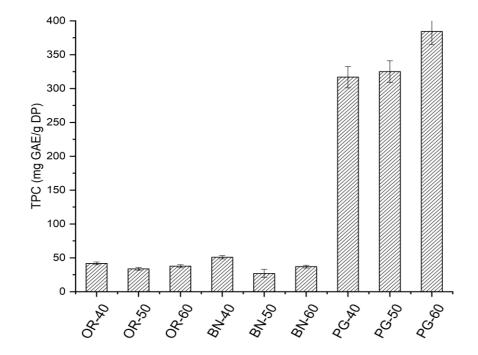


Figure 2
Total phenolic content (TPC)
measured in extracts obtained from
orange (OR), banana (BN), and
pomegranate (PG) peel wastes, using
different extraction ratios. V alues
are expressed as mg GAE/g DP.

Fruit peel waste	TPC (mg GAE/g DP)	DPPH (mmol TAE/g DP)	RSC (%)
OR-40	42 ±2.1	0.14 ± 0.003	92.0
OR-50	33 ± 2.0	0.17 ± 0.005	90.8
OR-60	38 ± 2.7	0.20 ± 0.002	89.4
BN-40	51 ±2.5	0.020 ± 0.001	66.5
BN-50	27 ± 6.0	0.040 ± 0.003	70.5
BN-60	37 ± 2.1	0.090 ± 0.002	73.8
PG-40	320 ± 16	0.43 ± 0.008	93.3
PG-50	330 ± 16	0.52 ± 0.005	90.8
PG-60	380 ± 19	0.62 ± 0.060	89.6

Table 1
Total phenolic content (TPC), 1, 1-diphenyl-2-picrylhydrazyl (DPPH) antioxidant activity and radical scavenging capacity (RSC) of extracts from orange (OR), banana (BN), and pomegranate (PG) peel wastes.

Antioxidant activity of fruit peels

In the case of orange peels, the antioxidant capacity ranged from 0.14 ± 0.003 to 0.20 ± 0.002 mmol TAE/g DP, with the highest value observed at OR-60. As presented in Table 1, banana peel extracts exhibited antioxidant capacity values between 0.020 ± 0.001 and 0.090 ± 0.002 mmol TAE/g DP. Pomegranate peels demonstrated the highest antioxidant capacity, with values ranging from 0.43 ± 0.008 to 0.62 ± 0.060 mmol TAE/g DP, the maximum of which was recorded for the extract obtained at the 1/60 ratio. The findings for pomegranate peels are consistent with those reported by Russo et al., (2018). Similarly, orange peels' antioxidant capacity values align with those reported by Chen et al., (2017). Regarding banana peels, the results are in

agreement with the study by González-Montelongo et al., (2010), who reported an antioxidant capacity of 0.050 mmol TAE/g DP when using an ethanol: water (1:1) solvent system for extraction.

Identification and quantification of phenolic compounds in fruit peels waste extracts

The HPLC analysis identified and quantified key phenolic compounds in banana, orange, and pomegranate peel extracts, with extraction efficiency varying depending on the dry peel-to-solvent ratio. This identification was achieved using a multi-standard approach, as shown in the chromatogram of Figure 3, which was obtained at 280 nm. According to the literature, the dominant phenolic compounds in orange peels include hesperidin, naringin, catechin, quercetin,

and various phenolic acids (Sáenz, et al., 2023; Zhao et al., 2019). In the present study, the polyphenolic compounds identified in orange peel extracts included

gallic acid, chlorogenic acid, caffeic acid, p-coumaric acid, and ferulic acid (Fig. 4).

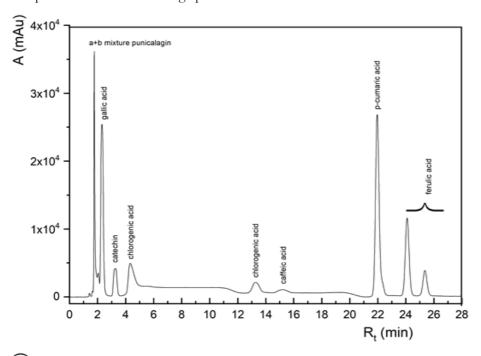


Figure 3
HPLC/DAD (at 280 nm)
chromatogram obtained from
the mixture of punicalagin
(A+B mixture), gallic acid,
catechin, chlorogenic acid, caffeic
acid, p-coumaric acid and
ferulic acid.

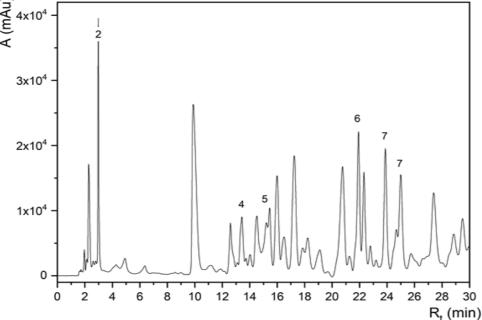


Figure 4
Representative HPLC/DAD
chromatogram of the phenolic
compounds identified in extracts
obtained in OR-40. The
identified phenolic compounds
are: (2) gallic acid, (4)
chlorogenic acid, (5) caffeic acid,
(6) p-coumaric acid and (7)
ferulic acid.

The highest gallic acid content (0.66 \pm 0.070 mg/g DP) was recorded at OR-60, while ferulic acid (0.52 \pm 0.070 mg/g DP) peaked in the OR-50 sample. The concentrations of chlorogenic acid and p-coumaric acid remained relatively stable across all tested ratios. Caffeic acid (5.9 \pm 0.50 mg/g DP) was most abundant at the 1/40 ratio, which was identified as the optimal extraction condition. As shown in Table 2, the 1/60 ratio did not result in the

extraction of most polyphenols. These results are in agreement with Kaur et al., (2024), who reported similar values for gallic, p-coumaric, ferulic, and chlorogenic acids using conventional and non-conventional extraction methods. Likewise, Gómez-Mejía et al., (2019) found comparable levels of p-coumaric and ferulic acids using 60% ethanol as a solvent. In banana peel and pulp extracts, the main

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Compounds mg/g DP OR-40 OR-50 OR-60 Orange Gallic acid 0.13 ± 0.090 0.30 ± 0.050 0.66 ± 0.070 0.40 ± 0.010 Chlorogenic acid 0.44 ± 0.020 0.49 ± 0.020 Caffeic acid 5.9 ± 0.50 2.9 ± 0.30 ND p-coumaric acid 0.090 ± 0.006 0.090 ± 0.006 ND Ferulic acid 0.25 ± 0.050 0.52 ± 0.070 0.32 ± 0.030 Banana BN-40 BN-50 BN-60 Gallic acid 0.070 ± 0.008 0.080 ± 0.002 0.25 ± 0.070 Catechin 0.20 ± 0.010 0.34 ± 0.020 0.25 ± 0.050 Chlorogenic acid 0.13 ± 0.030 0.14 ± 0.007 0.15 ± 0.010 Pomegranate PG-40 PG-50 PG-60 Punicalagin (A+B mixture) 22 ± 0.60 25 ± 0.17 28 ± 0.80 Gallic acid 12 ±1.4 13 ± 1.3 13 ± 0.20 Catechin 17 ± 3.0 20 ± 0.40 20 ± 0.40 Chlorogenic acid 2.9 ± 0.10 3.7 ± 0.28 3.3 ± 0.10 p-coumaric acid 1.2 ± 0.060 1.5 ± 0.040 1.4 ± 0.040

Table 2
Concentration of phenolic compounds identified by HPLC in extracts from orange, banana, and pomegranate peel wastes.

ND: Not Detected.

extraction of most polyphenols. These results are in agreement with Kaur et al., (2024), who reported similar values for gallic, p-coumaric, ferulic, and chlorogenic acids using conventional and non conventional extraction methods. Likewise, Gómez-Mejía et al., (2019) found comparable levels of p-coumaric and ferulic acids using 60% ethanol as a solvent. In banana peel and pulp extracts, the main p-coumaric acids; as well as other phenolics, including lignin and tannins (Kritsi et al., 2023). Figure 5 presents a representative HPLC chromatogram at 280 nm of the BN-40 extract, indicating the presence of gallic acid, catechin, and chlorogenic acid. The highest concentration of gallic acid (0.25 \pm 0.07 mg/g DP) was detected in the BN-60 sample, while catechin reached its peak level (0.34 ± 0.02 mg/g DP) at the 1/50 ratio. Chlorogenic acid levels remained relatively consistent across all tested ratios. The optimal ratio for polyphenol extraction, as supported by both HPLC and the Folin-Ciocalteu assay, was observed in the BN-40 sample. These findings align with Manthey et al., (2016), who reported a catechin concentration of 0.16 mg/g DP and with Candelaria et al., (2003), who identified gallic acid concentrations ranging from 0.05 to 1.5 mg/g DP in banana peels from Tenerife. The polyphenolic profile of pomegranate peel extracts (PG-60) included punicalagin (A+B mixture), gallic acid, catechin, chlorogenic acid, and p-coumaric acid, as shown in Figure 6. These findings are consistent with previous studies (Shalaby et al., 2019; Saparbekova et al., 2023). The highest total punical agin concentration (28 ±0.80 mg/g DP) was observed at a DP/solvent ratio of 1/60, which also yielded the highest gallic acid levels (13 ±0.20 mg/g DP). In contrast, chlorogenic acid (3.7 ±0.28 mg/g DP), catechin (20 ± 0.40 mg/g DP), and p-coumaric acid (1.5 ±0.040 mg/g DP) reached their peak concentrations at a 1/50 ratio. These results align with the findings of Shalaby et al., (2019), who reported high levels of gallic acid, catechin, and chlorogenic acid in pomegranate peels extracted with aqueous and ethanolic solvents. Similarly, Russo et al., (2018) and Venkataramanamma et al., (2016) emphasized the influence of the solvent system on phenolic yield. The detection was performed using a diode array detector (DAD). The quantification of each phenolic compound was carried out at its individual maximum absorption wavelength, determined from the DAD spectra, and in combination with the calibration obtained from the multi-standard solution. Wavelengths of 250 nm and 280 nm were selected as they provided the most distinct chromatographic profiles, ensuring optimal peak resolution, visualization, and integration for all

identified compounds. The quantification of each phenolic compound was carried out in combination with

the calibration curve obtained from the multi-standard solution.

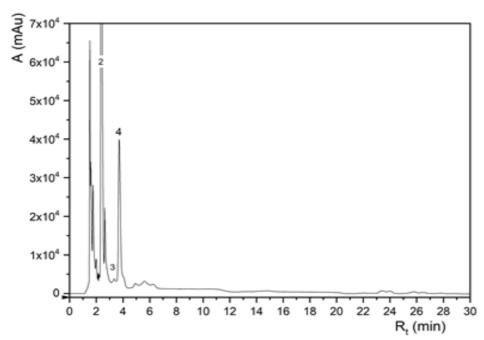


Figure 5
Representative HPLC/DAD
chromatogram of the phenolic
compounds identified in BN-40.
The identified phenolic
compounds are: (2) gallic acid,
(3) catechin and (4) chlorogenic
acid.

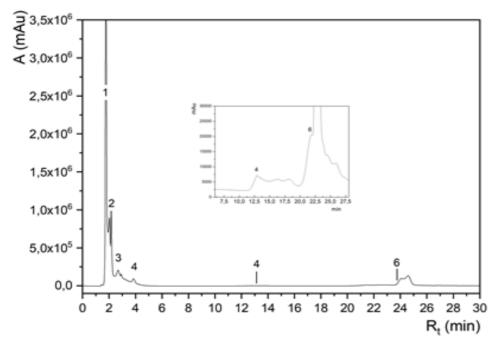


Figure 6
Representative HPLC/DAD
chromatogram (250 nm) of the
phenolic compounds identified in
PG-60. The identified phenolic
compounds are: (1) punicalagin
(A+B mixture), (2) gallic acid,
(3) catechin, (4) chlorogenic acid
and (6) p-coumaric acid.

Ratio of dry peel mass to solvent volume

The solid-to-liquid ratio is a critical operational parameter that significantly influences the overall profitability of the process. Several studies have reported that TPC increases with a higher solid-to-solvent ratio at room temperature, as well as under elevated temperatures or ultrasonic-assisted extraction conditions (Bucić-Kojić et al., 2007). An increased concentration gradient -i.e., a greater concentration difference

between the solid and solvent- enhances diffusion and mass transfer, leading to a more rapid saturation of the solvent. However, once the solvent becomes saturated, polyphenols remain in the fruit peel, reducing the overall extraction efficiency (Frempong et al., 2021; Jovanovic et al., 2017). Based on the results presented in Tables 1 and 2, the 1/40 ratio was identified as optimal for both orange and banana peels, providing the highest polyphenol yield. In contrast, further increases

in solvent volume (ratios of 1/50 and 1/60) led to decreased extraction efficiency, despite the theoretical expectation that higher solvent-to-solid ratios prevent solvent saturation and promote better extraction. This decline may be attributed to the rapid attainment of equilibrium polyphenol concentration in the solvent, which limits further extraction even though polyphenols remain in the solid matrix. Regarding pomegranate peels, an increase in TPC was observed with a greater solvent volume. This is likely due to the higher polyphenol content of pomegranate peels, along with the high solubility of these compounds in the extraction medium. As a result, the concentration gradient remains elevated, sustaining high diffusion flux and mass transfer without reaching equilibrium within short extraction times. Consequently, applying a lower solidto-solvent ratio in pomegranate peel extraction is expected to lead to solvent saturation, disabling the extraction of the maximum possible amount of polyphenols.

Conclusions

The findings of this study highlight the potential of fruit peels waste, an abundant by-product of industrial juice production as a valuable source of natural antioxidants and polyphenols. The use of 50% aqueous ethanol, an effective, simple, low-cost, and environmentally friendly method for recovering bioactive compounds from peel wastes of orange, banana, and pomegranate, was evaluated at varying dry peel-tosolvent ratios. Among the tested samples, the highest antioxidant capacity, evaluated by the DPPH assay, followed the order PG-60 > PG-50 > PG-40 > OR-60> OR-50 > OR-40 > BN-60 > BN-50 > BN-40. Values ranged from 0.62 ± 0.06 to 0.020 ± 0.001 mmol TAE/g dry peel across all fruit peel extracts. The TPC followed a different trend, with the order PG-60 > PG-50 > PG-40 > BN-40 > OR-40 > OR-60 > BN-60 > OR-50 > BN-50. Values for banana and orange peel extracts at different dry peel-to-solvent ratios were relatively similar (51 ± 2.5 - 27 ± 6.0 mg GAE/g DP) and considerably lower compared to those of pomegranate (320 \pm 16 - 380 \pm 19 mg GAE/g DP). The HPLC analysis confirmed significant differences in the phenolic profiles among banana, orange, and pomegranate peel extracts. Pomegranate extracts exhibited the highest concentrations of key bioactive compounds, particularly punicalagin (A+B mixture), gallic acid, and catechin. In comparison, orange peel extracts presented moderate concentrations of gallic and chlo-

rogenic acids, alongside the presence of caffeic and ferulic acids. Banana extracts, on the other hand, exhibited a more limited phenolic profile, characterized by consistently low levels of gallic acid, catechin, and chlorogenic acid. These findings emphasize the importance of the dry peel-to-solvent ratio for each fruit peel in optimizing extraction efficiency and developing a sustainable extraction protocol. Nevertheless, each recycling process needs to balance two aspects environmental gain (waste management) and economic gain. A future study should evaluate the financial gain, including all steps of the waste-to-product transformation (e.g., process cost, scalability, and stability of desired antioxidants during processing). Moreover, the potential presence of crop protection products in the peel wastes was not investigated in the present study, despite their possible impact on both safety and quality of the final extracts. Future research should therefore combine techno-economic feasibility assessments with comprehensive contaminant analyses to ensure that valorization of fruit peel wastes can be safely and effectively integrated into the circular economy.

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