A comparative evaluation of the phenol and lycopene content of tomato by-products subjected to different drying methods

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ABSTRACT

This study is focused on exploring tomato by-products to provide ingredients rich in bioactive compounds with higher functional value through applying innovative techniques (microwave and spiral flash dryer) and conventional techniques (freeze-drying and hot air-drying) to the dehydration of industrial tomato pomace. The effect of the drying method on the bioactive composition of the tomato by-products (phenols, lycopene and soluble dietary fiber) was analyzed. The phenolic composition was determined using a targeted chromatographic approach based on UHPLC-QqQ-MS/MS. Large amounts of naringenin (194.7–949.4 mg/kg) were detected, together with quercetin, caffeic acid, coumaric acid and 4-hydroxybenzoic acid. Microwave dehydration improved the retention of flavanone-like compounds, especially naringenin, and lycopene, while tomato products dehydrated with Spiral Flash dryer showed higher concentrations of flavonols and phenolic acids. The results showed that the industrial application of drying processes using Spiral Flash, and especially with microwaves, could be promising for producing high added-value ingredients from tomato by-products.

1. Introduction

The tomato (*Lycopersicon esculentum*) is one of the most widely cultivated vegetables in the world, with a production of about 180 million tons (FAO, 2019). Due to the seasonality and high perishability of this fruit, only a proportion of tomato production is consumed as fresh product. In 2019, about 367,000 tons of tomatoes were processed by the food industry (FAO, 2019). Commercially, there are different tomato products, such as whole peeled canned tomato, juice, puree, paste, sauce, and ketchup (Rajan et al., 2022). Industrial tomato processing generates a large amount of waste, commonly called tomato pomace (TP) (Bhatkar et al., 2021). The composition of this TP varies depending on the type of final product, e.g. canned tomatoes generate a residue consisting mainly of peel, while in the production of juice, paste and puree, the TP consists of a mixture of peel and seeds (Bhatkar et al., 2021) representing between 3% and 5% of the fresh tomato by weight (Rajan et al., 2022). Tomato and tomato by-products contain a great variety of biologically active compounds, including carotenoids, proteins, minerals, dietary fiber and oils (Grassino et al., 2020; Zuorro et al., 2013).

Seeking a valorization of the TP, previous studies have shown that dehydrated TP can be added to such products as ketchup (Belović et al., 2018), meat products (Eyiler & Oztan, 2011) and bread (Majzoobi et al., 2011) without affecting their technological properties and with a high sensory acceptance. Other works also reinforce that, besides nutritionally enriching such products as pasta (Padalino et al., 2017) and cracker biscuits (Isik & Topkaya, 2016), and even ice-cream (Rizk et al., 2014), the addition of TP led to higher antioxidant activity and storage stability due to the presence of phenolic compounds. Nowadays, there is growing interest in the study of the bioactivity and health properties of tomato by-products. Studies with tomato (peel and seeds) and by-products show antioxidant and antimutagenic activities (Kumar et al., 2021; Valdez-Morales et al., 2014), and protection from oxidative stress in an *in-vitro* model of human endothelial vascular cells (HUVEC) (Cesare et al., 2021). Recently, Perea-Domínguez et al. (2021) observed that the phenolic fractions obtained from industrial tomato by-products showed

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antioxidant properties and an important anti-proliferative activity in the MCF-7 breast cancer cell line.

Despite the potential for valorization of tomato by-products, currently, the waste from tomato processing does not generate profits for the industries, resulting in a major management problem, mainly storage and preservation issues (Bhatkar et al., 2021; Silva et al., 2019). In particular, tomato processing is carried out during the summer, and, due to the high moisture content of the TP (by-product), the accumulation of this waste leads to uncontrolled anaerobic fermentation with the consequent environmental problems (Selvaggi et al., 2021). In the context of the circular bio-economy, the reuse of these agri-food residues to recover bioactive compounds is the key to reducing the disposal of organic waste from industrial processing (Selvaggi et al., 2021).

Undoubtedly, the vegetable processing industry's greatest challenge in handling waste is to stabilize it to obtain stable products that maintain their highly perishable bioactive compounds and nutritional properties. Drying is one of the oldest methods used to remove water for food preservation, as a lower water potential (water activity) is achieved for food stability during storage (Pateiro et al., 2022). Generally, methods involving hot air are used to dehydrate plant by-products (Bhatkar et al., 2021). However, some bioactive compounds from TP, such as lycopene, are hightly sensitive to high temperatures (Bakir et al., 2023). Accordingly, such technologies as microwaves (Bakić et al., 2019), infrared drying process (Bakir et al., 2023) and ohmic technology (Coelho et al., 2019) have been used to improve the retention of bioactive compounds. However, many of these technologies have been explored on a laboratory scale, or combined with other extraction technologies to improve the process for obtaining lycopene-rich extracts from tomato by-products. The aim of this study was to search for the optimal drying technology for tomato by-products, easily scalable at an industrial level and allows ingredients to be obtained directly with a high shelf-stability and maximum retention of the valuable bioactive compounds, such as phenolic compounds, lycopene and soluble dietary fiber. Various dehydration processes were studied: freeze-drying, air-drying (40 °C and 60 °C), micro-wave assisted drying and Spiral Flash air drying. The tomato waste used in this study was the TP from industrial tomato processing (paste and pure), with a subsequent physical separation after dehydration into two fractions rich in peel and seeds, respectively. The phenolic composition of different tomato products was determined using a targeted chromatographic approach based on ultra-high performance liquid chromatography with tandem mass spectrometry (UHPLC-QqQ-MS/MS).

2. Materials and methods

2.1. Chemicals and reagents

Acetonitrile (HPLC-MS grade), methanol (HPLC grade) and formic acid (HPLC grade) were purchased from Scharlab Chemie (Sentmenat, Catalonia, Spain). The quercetin, quercetin-3-glucuronide, isorhamnetin-3-glucoside, syringetin-3-glucoside, epicatechin, dimer B1 and B2 and quercetin standards were purchased from Extrasynthese (Genay, France). Gallic acid, caffeic acid, ferulic acid, vanillic acid, syringic acid, p-hydroxybenzoic acid, 3,4-dihydroxybenzoic acid (protocatechuic acid), p-coumaric acid, (+)-catechin were acquired from Sigma-Aldrich (St. Louis, USA). Kaempferol-3-glucoside and caftaric acid were purchased from Purifa-Cymit (Barcelona, Spain). Coutaric acid and naringerin and were purchased from Phytolab (Madrid, Spain) and Fluochem (Hadfield, England), respectively. Stock solutions of standard compounds were prepared by dissolving each compound in methanol at a concentration of 1000 mg/L and stored in a dark flask at −20 °C. The water used was Milli-Q quality (Millipore Corp, Bedford, MA, USA).

2.2. Plant material

Tomato pomace (TP) samples, from the same tomato variety, were obtained from an industrial processing plant from J. Heinz Manufacturing Spain, S.L.U., located at Carretera Rincón de Soto-Corella, km. 2.8, 26,540 Alfaro, La Rioja (Spain), in October–November 2021, on 8 distinct days (8 batches of TP). The processing steps to produce tomato concentrate included washing, cold breaking, evaporating, and pasteurization to generate the final standard sauce (average 10°Brix, pH 4.2). The TP was obtained by squeezing tomatoes and was composed of a mixture of peel, seeds and a small proportion of pulp. Each batch (approximately 15 kg of TP) was collected in plastic containers immediately after being produced and transported to the food pilot plant of Centro Tecnológico Agroalimentario CTIC CITA (Calahorra, La Rioja, Spain). TP samples were frozen in an air-freezer chamber and stored (−20 °C) until the dehydration processes. The average composition of the TP was: moisture (53 g/100 g), protein (10 g/100 g), fat (8 g/100 g), ash (2 g/100 g) and fiber (soluble and insoluble fiber) (27 g/100 g).

2.3. Dehydration process of tomato by-products

In this study, the following dehydration processes were studied: freeze-drying, hot air-drying, a microwave system and Spiral Flash air drying (Fig. S1 Supporting Information). Prior to the drying process, the tomato pomace was thawed in a cold chamber (5-8 °C). This step was performed for all processes except for freeze-drying, where the samples were placed directly into the freeze-dryer.

2.3.1. Freeze-drying

A laboratory-scale lyophilizator (Scanvac-CoolSafe-95-16-Pro freeze-dryer control, Bjarkesvej, Denmark) was used for freeze drying. The device has ice production capacity of 2.5 kg in 24 h. The TP samples were placed in Petri dishes, weighed (approximately 100 g) and frozen at ultra-low temperature (−80 °C) for 24 h before being placed in the freeze dryer chamber. The samples were dried for 24 h until complete dehydration in the freeze dryer operating at 0.1 bar pressure with a temperature ramp of −20 to 0 °C.

2.3.2. Hot air oven drying

The drying process was carried out in an air oven (POL-EKO, SLW400-STD, Wodzisław Śląski, Poland). The TP samples (approximately 5 g) were deposited in a thin layer (±1 cm) on aluminum trays in the oven at 40 and 60 °C, respectively. For each temperature, the dehydration time was determined by weighing the sample at various time intervals during drying until the weight became constant. The optimized drying times for each air temperature were 8 and 3 h at 40 °C and 60 °C, respectively. Fig. S2 of Supporting Information shows the kinetic of dehydration of TP at 40 and 60 °C respectively.

2.3.3. Spiral flash air dryer

The dehydration process with the Spiral Flash dryer was carried out in the plant of the INGETECSA company (Barcelona, Spain). This dryer system is composed of a vertical chamber with a static blade ring and upper extraction, which can simultaneously dry and disperse organic materials without any risk of explosion due to mechanical friction (Fig. S1 of Supporting Information). In this system, the filtered hot air (140-160 °C) is pushed into the drying chamber by a fan, flowing through a static blade ring. The blades have a fixed orientation which generates a highly turbulent air flow. The TP to be dehydrated is introduced above the blade ring, and as the product falls, it is mixed with the hot air flow and the drying process is carried out quickly (20–60 s). Summarizing the process, the moist material (TP) is dosed into the drying chamber through a feeder, and dried by stirring and vortex flow. The temperature of the drying air is between 140 and 160 °C, but as it is a very turbulent process, the product does not exceed 45 °C, discharging

at around final humidity (10 g/100 g).

2.3.4. Microwave system

The process of dehydrating the TP was done in a prototype continuous-flow microwave oven (Model SI MAQ0101; Sairem Iberica S. L., Barcelona, Spain). This microwave system contains 4 magnetrons, each with a power of 1000 W. The TP samples were placed in a polypropylene tray and microwave-treated at 1500 W, 2250 W and 3000 W corresponding to 50, 75 and 100% respectively, of the output power using 3 magnetrons. These power levels were chosen based on preliminary experiments. In addition, cold air was introduced to avoid an excessive rise in temperature inside the TP samples, and the conveyor belt system moved back and forth to ensure the treatment was homogeneous.

2.3.5. Conditioning of dehydrated tomato by-products

In order to obtain different ingredients, one composed of a fraction rich in peel and the other in seeds, the dehydrated TP was manually sieved using various sieve sizes (CISA CEDACERIA INDUSTRIAL, S.L., Barcelona, Spain): ø 3.0, 2.5 and 1.8 mm (Fig. 1) to obtain five fractions with different particle size (F1 to F5). For the study of the phenolic and lycopene composition, the fraction retained on the ø 2.5 mm sieve, rich in tomato seeds (F2) was selected. The other selected fraction was the TP fraction passing through the sieve of ø 1.8 mm, rich in tomato peel (F5). After separation, the F2 and F5 fractions were weighed, and these corresponded respectively to approximately 60% and 30% of the tomato paste (TP). The dehydrated samples (tomato pomace TP and F2 and F5 fractions) were crushed directly in an IKA Instruments grinder (Staufen, Germany) with a power 420 W, transferred to falcon tubes and then stored at −80 °C until their analysis.

2.4. Determination of individual phenolic compounds by ultra-high-performance liquid chromatography coupled to tandem mass spectrometry (UHPLC-QqQ-MS/MS)

2.4.1. Solid-liquid extraction of phenolic compounds

The phenolic compounds of the tomato by-products (tomato pomace TP, rich-seeds (F2) and rich-peel (F5) fractions) were extracted by a solid-liquid extraction (SLE) procedure, immediately before chromatographic analysis. For the extraction of phenols from the dehydrated (200 mg) and fresh (600 mg) samples, a volume of 4 mL of methanol/Milli-Q water/formic acid (79:20:1, v/v/v) was added. The mixture was vortexed and macerated overnight at 4 °C in the dark. The samples were then sonicated at a frequency of 40 Hz for 5 min in an ultrasonic bath (Ultrasons P. Selecta, Barcelona, Spain). Then, each sample was centrifuged (9000 rpm/10 min/20 °C) in a Sorvall LYNX 4000 Superspeed Centrifuge (Thermo Scientific™, Madison, WI, USA). The supernatant was collected and filtered with a 0.22 μm PTFE filter (Scharlab Chemie, Catalonia, Spain) and placed in a chromatographic vial (2 mL) prior to analysis.

2.4.2. Ultra-high-performance liquid chromatography coupled to tandem mass spectrometry (UPLC-QqQ-MS/MS)

The phenolic composition of the tomato by-products was evaluated using the method described by Costa et al. (2022). The phenol extracts were analyzed using ultra-high-performance liquid chromatography with triple-quadrupole mass spectrometry (UPLC/QqQ-MS/MS). The analyses were carried out in a liquid chromatograph Shimadzu Nexera (Shimadzu Corporation, Japan) coupled to a QTRAP mass spectrometer (AB Sciex 3200QTRAP®, Sciex, USA). The polyphenol separation was performed using a column reversed-phase (Waters AcQuity BEH C18 column, 100 mm × 2.1 mm, 1.7 μm) equipped with a VanGuard™ AcQuity BEH C18 Pre-Column (5 × 2.1 mm, 1.7 μm) supplied by Waters (Milford, MA, USA). The chromatographic parameters used were flow rate: 0.45 mL/min; injection volume: 2.5 μL; temperature autosampler:
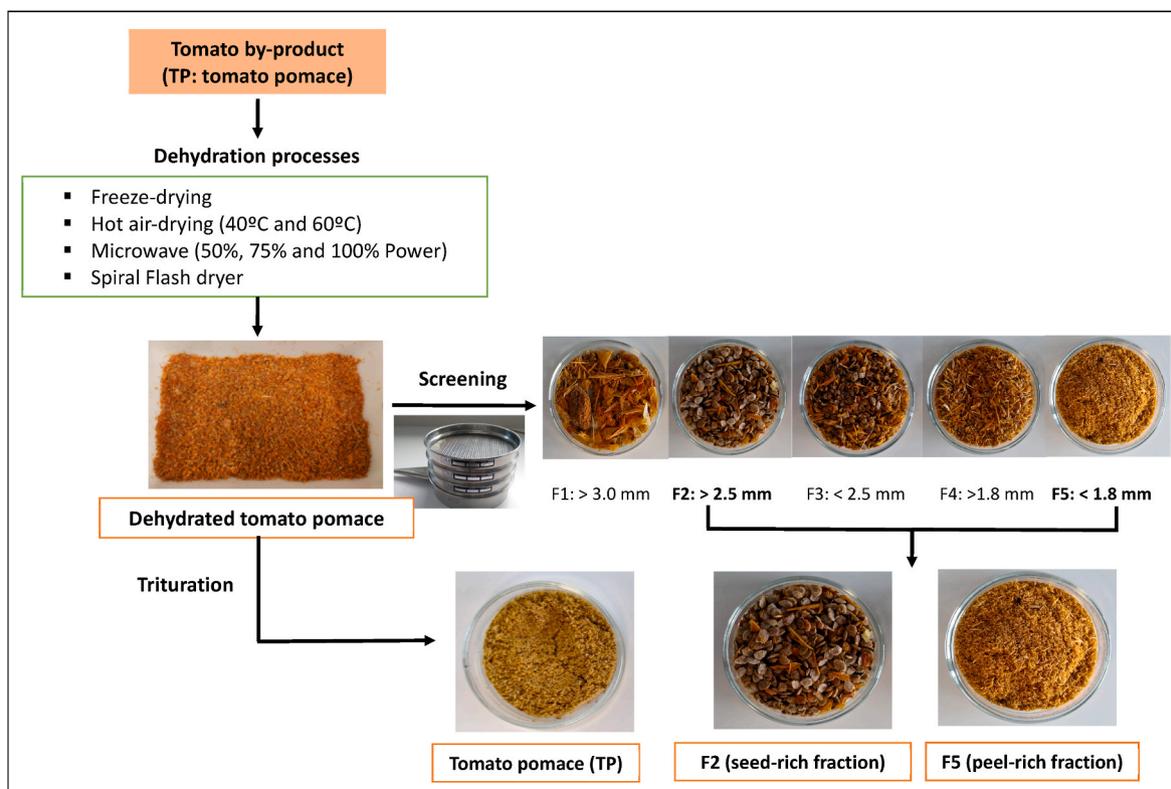
Fig. 1. Scheme of the dehydration treatments and the steps to obtain the different fractions of tomato by-products.

5 °C; and oven temperature: 40 °C.

For the analysis, a mobile phase of formic (0.1 mL/100 mL) acid in water (solvent A) and formic acid (0.1 mL/100 mL) in acetonitrile (solvent B) were used. The eluted compounds were analyzed using a 3200QTRAP triple quadrupole mass spectrometer (AB Sciex, USA) equipped with an electrospray ionization source (ESI Turbo V™ Source). The mobile phase elution gradient and the mass spectrometer parameters are described in Costa et al. (2022). For each phenolic compound, the retention time and MRM transitions, including the individual declustering potential (DP), entrance potential (EP), collision cell entrance potential (CEP), collision energy (CE) and collision cell exit potential (CXP) were acquired (Table S1 Supporting Information). The data acquisition was conducted with the Analyst® 1.6.2 software (AB Sciex, USA).

For the identification of each phenolic compound, the retention time and spectra of its respective externally injected standard was used. The other compounds for which no standards were available were identified with the mass of the parent ion (M−H) and the typical MS fragmentation pattern described in the literature. The phenolic quantification was performed using the calibration curves of their corresponding pure commercial standards (Table S2 of Supporting Information). In the case of compounds without standards available, the quantification was performed using the calibration curves of standards with similar chemical structures. The results were expressed as mg compound/kg tomato pomace (TP), seed-rich fraction (F2) or peel-rich fraction (F5), while the results for the fresh TP samples (raw) were expressed as mg/kg dry weight (raw sample), in order to compare with the dehydrated samples. The phenolic compounds identified and quantified were classified into flavonoids: flavonols and flavanones, and phenolic acids (hydroxybenzoic and hydroxycinnamic acids).

2.5. Determination of lycopene

Lycopene was determined by a spectrophotometer method, as described by Davis et al. (2003). The tomato by-product sample (0.6 g) was weighed in a Falcon tube (50 mL), and 5 mL of acetone with BHT (0.05 mL/100 mL), 5 mL of ethanol (95 mL/100 mL) and 10 mL of hexane were added and vortexed immediately. The sample was centrifuged (200 rpm, 15 min, 4 °C) and 3.0 mL milli-Q water was added. The sample was left at room temperature for 10 min to allow for phase separation and for all air bubbles to disappear, and the absorbance at 503 nm was determined in the supernatant. The lycopene content was calculated based on the weight of the samples using the absorbance at 503 nm, and the results were expressed as mg lycopene/kg sample.

2.6. Soluble dietary fiber content

The soluble dietary fiber content was analyzed in dehydrated samples (TP, F2 and F5 fractions) by gravimetric determination according to the method described by Maran (2015). Briefly, the sample was weighed (1 g) in a 250 mL Erlenmeyer flask and 20 mL de HCl (0.02 M) were added. The soluble dietary fiber extraction was performed at 85 °C (40 min) in an agitation bath. The Erlenmeyer flask was covered with a plastic wrap to prevent evaporation of the extraction solvent. After incubation, the mixture was filtered through cheese cloth (Miracloth-Rapid, EMD Millipore, Billerica, MA 01821, USA) and allowed to cool to room temperature (30 °C). Ethanol (95%) at 60 °C was added to the filtrate in a 4:1 ratio and stored at 4 °C for 24 h. The polysaccharides containing soluble dietary fiber were removed by centrifuging (9,000 rpm, 30 min at 22 °C). The wet soluble fiber was dried in a hot air oven at 55 °C until a constant weight was achieved. The results are expressed as g soluble dietary fiber/kg sample.

2.7. Statistical analysis data

Concentration values of the phenolic compounds, lycopene and

soluble fiber were reported as means ± standard deviation (SD). For each by-product tomato sample (TP, F2 and F5 fractions), one-way analysis of variance (ANOVA) and Fisher's test at a level of 0.05 were used to determine the significance of differences among the dehydration processes. All analyses were performed using Statistic Package for Social Science (SPSS)(IBM, Armonk, NY).

3. Results and discussion

3.1. Impact of drying processes on the phenolic composition of tomato by-products

To estimate the effect of the dehydration process on the phenolic composition of tomato by-products, a targeted chromatographic approach by UHPLC-QqQ-MS/MS was used. A total of nineteen compounds, belonging to the flavonoids (naringenin and other minor flavanols, mainly quercetin derivatives) and phenolic acids (hydroxycinnamic and hydroxybenzoic acids) groups, were identified and quantified. The results of the phenol quantification for each type of tomato by-product (Fig. 1): tomato pomace (TP) (Table 1), seed-rich fraction (F2) (Table 2) and rich-peel fraction (F5) (Table 3), are described in the following sections. To evaluate the impact of each drying process, the concentration of each phenolic compound expressed on a dry-weight (DW) basis of raw sample (TP, F2 and F5, respectively) was used as a control.

3.1.1. Impact of drying processes on tomato pomace (TP) phenolic composition

The results of the impact of the different drying processes on the stability of the phenolic compounds detected in the tomato pomace (TP) are shown in Table 1. Total phenolic content ranged between 336.66 mg/kg (freeze-dried TP) and 568.37 mg/kg (microwave TP). Regardless of the dehydration procedure applied, the average phenolic composition of the TP was mainly flavonoids and phenolic acids. Within the flavonoid group, the flavanone naringenin was the main phenol detected in the TP (about 75.3% of the total phenols) with a concentration ranging from 253.03 to 468.76 mg/kg TP (Table 1). This was similar to that reported by Abbasi-Parizad et al. (2020) in TP dried in a vacuum oven (at 50 °C for 300 min).

Comparing the different drying techniques, independently of the power applied (50%, 75% or 100% of 3 magnetrons, each with a power of 1000 W), the microwave was revealed to be the best process for the greater retention of naringenin (Table 1). Microwave dried TP samples showed an average concentration of 557 mg/kg, significantly higher (p < 0.05) than the raw product (245.06 mg/kg DW) (Table 1). The air-drying and Spiral Flash drying processes showed similar results, resulting in greater retention of naringenin in the TP samples than with the freeze-drying process. These results are in agreement with Tomas et al. (2017) who found that the naringenin concentration was 20-fold and 43-fold higher in industrial and home processed sauces respectively, compared to fresh tomato. This increase is attributed to the high temperature at low pH, under which conditions conversion of naringenin chalcone to naringenin is stimulated (Capanoglu et al., 2008). Moreover, since the phenolic compounds, including naringenin, are trapped in the insoluble polyesters of the cuticle, which are constituents of tomato peel fiber, hot air and microwave processing could facilitate the release of these compounds from the cutin matrix by breaking the interactions and thus increasing their extractability (Martínez-Huélamo et al., 2015). Naringenin is a compound that is gaining increasing attention from researchers for its bioactive properties (Sharma et al., 2021), so the results obtained in our study reveal the potential use of TP as a raw material to obtain naringenin for future nutraceutical applications. Recent studies with naringenin have reported anti-inflammatory and immunomodulatory effects (Abbasi-Parizad et al., 2020; Kataoka et al., 2021), anti-carcinogenic activity (Ćetković et al., 2012), and neuroprotective effects against degenerative diseases,

Table 1
Impact of drying processes on the phenolic content of the tomato pomace (TP). The results are expressed as mg/kg sample.

Compound (mg/kg)	Raw TP (dry weight)	Freeze-drying	Air-drying 40 °C	Air-drying 60 °C	Spiral Flash	Microwave 50% 3 MG	Microwave 75% 3 MG	Microwave 100% 3 MG
Naringenin	245.06 ± 10.63^a	253.03 ± 6.14^{ab}	315.99 ± 13.84^c	296.59 ± 10.64^{bc}	331.01 ± 10.36^c	468.76 ± 11.12^d	454.23 ± 2.82^d	429.62 ± 24.15^d
Naringenin-hexose	1.30 ± 0.19^{bc}	1.26 ± 0.00^{bc}	1.36 ± 0.20^c	1.20 ± 0.19^{bc}	1.31 ± 0.02^{bc}	1.04 ± 0.09^{abc}	0.83 ± 0.05^{ab}	0.68 ± 0.05^a
Total flavanones	246.36 ± 10.82^a	254.29 ± 6.15^{ab}	317.35 ± 14.04^c	297.79 ± 10.83^{bc}	332.32 ± 10.37^c	469.80 ± 11.21^d	455.06 ± 2.87^d	430.30 ± 24.21^d
Quercetin	7.92 ± 0.60^a	7.50 ± 0.41^a	10.06 ± 0.20^b	9.55 ± 0.31^b	23.95 ± 0.16^e	11.40 ± 0.44^c	12.33 ± 0.07^{cd}	13.16 ± 0.11^d
Quercetin-gluc	4.90 ± 0.27^c	1.61 ± 0.26^a	3.15 ± 0.12^b	2.56 ± 0.33^{ab}	4.58 ± 0.32^c	2.75 ± 0.18^{ab}	2.64 ± 0.50^{ab}	2.65 ± 0.22^{ab}
Kaempferol	1.11 ± 0.11^a	0.95 ± 0.04^a	1.43 ± 0.21^a	1.14 ± 0.09^a	2.43 ± 0.25^b	1.36 ± 0.02^a	1.33 ± 0.23^a	1.48 ± 0.15^b
Kaempferol-gluc	3.49 ± 0.38^c	0.61 ± 0.04^a	1.48 ± 0.22^b	0.74 ± 0.10^a	1.30 ± 0.06^{ab}	0.78 ± 0.17^{ab}	0.67 ± 0.09^a	1.09 ± 0.13^{ab}
Syringetin	0.31 ± 0.02^a	0.34 ± 0.03^{ab}	0.41 ± 0.03^{abc}	0.42 ± 0.01^{bc}	0.87 ± 0.01^e	0.41 ± 0.02^{abc}	0.55 ± 0.04^d	0.47 ± 0.04^{cd}
Laricitrin	0.27 ± 0.02^a	0.31 ± 0.09^a	0.35 ± 0.03^a	0.37 ± 0.05^a	0.41 ± 0.03^a	0.29 ± 0.02^a	0.33 ± 0.01^a	0.41 ± 0.18^a
Total flavonols	18.01 ± 1.40^c	11.32 ± 0.87^a	16.89 ± 0.81^{bc}	14.77 ± 0.89^b	33.53 ± 0.83^d	16.99 ± 0.85^{bc}	17.86 ± 0.93^c	19.26 ± 0.82^c
Total flavonoids	264.37 ± 12.22^a	265.62 ± 7.02^a	334.24 ± 14.85^{bc}	312.56 ± 11.73^{ab}	365.85 ± 11.21^c	486.79 ± 12.05^d	472.92 ± 3.80^d	449.56 ± 25.03^d
Caffeic acid	21.94 ± 2.49^c	8.28 ± 0.47^a	14.36 ± 0.79^b	14.60 ± 0.27^b	27.78 ± 0.90^d	23.15 ± 0.94^{cd}	24.54 ± 0.81^{cd}	25.57 ± 1.45^{cd}
Caffeic acid-hexose	10.18 ± 0.02^a	8.76 ± 0.23^a	8.83 ± 0.85^a	8.13 ± 0.69^a	15.06 ± 1.65^a	10.68 ± 0.17^a	9.85 ± 0.10^a	8.72 ± 2.12^a
Coumaric acid	16.53 ± 1.25^d	16.16 ± 2.07^{cd}	18.01 ± 0.07^d	19.14 ± 2.07^d	11.51 ± 1.08^{bc}	5.45 ± 0.93^a	6.63 ± 0.04^{ab}	6.76 ± 0.02^{ab}
Coumaric acid-hexose	3.56 ± 0.48^a	3.23 ± 0.14^a	2.87 ± 0.19^a	4.30 ± 0.79^a	10.02 ± 0.30^b	4.08 ± 0.64^a	3.89 ± 0.02^a	3.60 ± 0.05^a
Ferulic acid	5.39 ± 0.06^e	3.24 ± 0.05^{ab}	4.47 ± 0.06^{cd}	3.80 ± 0.52^{bc}	4.12 ± 0.23^{bc}	2.79 ± 0.31^a	3.16 ± 0.24^{ab}	3.21 ± 0.14^{ab}
Total HC acids	57.60 ± 4.30^c	39.67 ± 2.97^a	48.55 ± 1.97^{abc}	49.97 ± 4.34^{bc}	68.49 ± 4.17^d	46.16 ± 2.99^{ab}	48.07 ± 1.21^{abc}	47.87 ± 3.78^{ab}
4-Hydroxybenzoic acid	35.63 ± 6.39^c	18.41 ± 0.62^a	27.70 ± 0.63^{bc}	23.14 ± 0.84^{ab}	28.11 ± 0.31^{bc}	20.54 ± 0.48^{ab}	25.27 ± 0.22^{ab}	26.85 ± 0.46^{abc}
Protocatechuic acid	5.56 ± 0.98^{ab}	4.29 ± 0.07^a	6.09 ± 1.03^{ab}	4.50 ± 0.33^a	7.17 ± 0.37^{bc}	8.84 ± 1.17^c	9.48 ± 0.57^{cd}	11.27 ± 2.00^d
Vanillic acid	4.01 ± 0.69^b	4.19 ± 0.12^b	3.91 ± 0.25^{ab}	4.28 ± 1.29^b	4.16 ± 0.51^b	2.39 ± 0.20^a	3.61 ± 0.91^{ab}	3.09 ± 0.13^{ab}
Gallic acid	1.34 ± 0.00^b	1.04 ± 0.03^a	1.55 ± 0.02^{cd}	1.47 ± 0.06^{bc}	4.47 ± 0.05^f	1.52 ± 0.08^{cd}	1.67 ± 0.01^{de}	1.77 ± 0.02^d
Gallic acid-gal	2.05 ± 0.04^b	2.55 ± 0.01^c	2.60 ± 0.03^c	2.63 ± 0.14^c	1.99 ± 0.00^b	1.31 ± 0.03^a	1.20 ± 0.04^a	1.35 ± 0.02^a
Syringic acid	0.72 ± 0.04^a	0.90 ± 0.04^{abc}	1.02 ± 0.06^{bc}	1.11 ± 0.10^{cd}	1.33 ± 0.04^d	0.84 ± 0.08^{abc}	0.76 ± 0.12^{ab}	0.81 ± 0.03^{ab}
Total HB acids	49.31 ± 8.14^c	31.38 ± 0.88^a	42.87 ± 2.02^{bc}	37.12 ± 2.76^{ab}	47.23 ± 1.28^c	35.43 ± 2.04^{ab}	41.98 ± 1.86^{bc}	45.13 ± 2.66^{bc}
Total phenolic acids	106.91 ± 12.44^{cd}	71.05 ± 3.85^a	91.42 ± 3.99^{bc}	87.10 ± 7.10^{ab}	115.72 ± 5.45^c	81.58 ± 5.04^{ab}	90.05 ± 3.07^{abc}	93.00 ± 6.43^{bc}
Total phenolic content	371.28 ± 24.66^{ab}	336.66 ± 10.87^a	425.66 ± 18.83^c	399.66 ± 18.83^{bc}	481.58 ± 16.66^d	568.37 ± 17.09^e	562.96 ± 6.87^e	542.56 ± 31.46^e

HC: hydroxycinnamic acid, HB: hydroxybenzoic acid. MG: 3 magnetrons, each with a power of 1000 W. Different letters in the same row indicate significant differences between drying processes (P < 0.05).

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Table 2

Impact of drying processes on the phenolic content of the seed-rich fraction (F2) of tomato pomace. The results are expressed as mg/kg sample.

Compound (mg/kg)	Raw F2 (dry weight)	Freeze-drying	Air-drying 40 °C	Air-drying 60 °C	Spiral Flash	Microwave 50% 3 MG	Microwave 75% 3 MG	Microwave 100% 3 MG
Naringenin	185.92 ± 9.54^b	137.00 ± 12.61^a	137.05 ± 1.42^a	124.02 ± 4.02^a	162.22 ± 4.72^{ab}	299.80 ± 40.21^d	251.34 ± 0.94^c	245.15 ± 17.98^c
Naringenin-hexose	1.50 ± 0.02^b	0.90 ± 0.01^a	0.97 ± 0.14^a	0.88 ± 0.09^a	1.25 ± 0.04^{ab}	1.26 ± 0.23^{ab}	1.03 ± 0.06^a	0.82 ± 0.44^a
Total flavanones	187.43 ± 9.57^b	137.90 ± 12.63^a	138.01 ± 1.56^a	124.90 ± 4.11^a	163.46 ± 4.76^{ab}	301.06 ± 40.45^d	252.37 ± 1.00^c	245.97 ± 18.42^c
Quercetin	5.99 ± 0.13^b	4.38 ± 0.47^a	4.00 ± 0.31^a	4.60 ± 0.37^a	15.05 ± 0.27^d	7.55 ± 1.11^c	7.22 ± 0.06^c	6.58 ± 0.09^{cd}
Quercetin-gluc	3.74 ± 0.31^c	1.41 ± 0.14^a	1.70 ± 0.24^a	1.73 ± 0.01^a	3.18 ± 0.04^c	2.37 ± 0.55^b	1.74 ± 0.06^{ab}	1.82 ± 0.28^{ab}
Kaempferol	1.09 ± 0.44^a	0.91 ± 0.00^a	1.15 ± 0.19^a	1.05 ± 0.21^a	1.40 ± 0.49^a	1.08 ± 0.04^a	1.04 ± 0.04^a	1.34 ± 0.08^a
Kaempferol-gluc	2.31 ± 0.21^d	0.53 ± 0.16^a	0.65 ± 0.03^{ab}	0.70 ± 0.06^{ab}	1.21 ± 0.08^c	0.86 ± 0.10^b	0.59 ± 0.08^{ab}	0.80 ± 0.09^{ab}
Syringetin	0.33 ± 0.07^{ab}	0.32 ± 0.01^a	0.38 ± 0.02^{abc}	0.40 ± 0.04^{abc}	0.57 ± 0.03^d	0.41 ± 0.04^{abc}	0.43 ± 0.05^{bc}	0.48 ± 0.03^{cd}
Laricitrin	0.23 ± 0.01^a	0.28 ± 0.04^{ab}	0.41 ± 0.06^{bc}	0.34 ± 0.03^{abc}	0.47 ± 0.15^c	0.29 ± 0.01^a	0.31 ± 0.02^{ab}	0.29 ± 0.01^{ab}
Total flavonols	13.70 ± 1.17^c	7.82 ± 0.83^a	8.30 ± 0.84^a	8.84 ± 0.71^a	21.87 ± 1.05^d	12.56 ± 1.85^{bc}	11.32 ± 0.30^b	11.31 ± 0.58^b
Total flavonoids	201.12 ± 10.74^c	145.72 ± 13.45^{ab}	146.31 ± 2.40^{ab}	133.74 ± 4.81^a	185.34 ± 5.81^{bc}	313.62 ± 42.29^e	263.69 ± 1.30^d	257.28 ± 19.00^d
Caffeic acid	18.84 ± 1.24^b	8.56 ± 1.84^a	9.69 ± 1.09^a	9.73 ± 0.89^a	26.08 ± 3.30^c	26.16 ± 0.72^c	26.81 ± 0.29^c	26.92 ± 0.38^c
Caffeic acid-hexose	11.49 ± 0.23^{ab}	12.25 ± 2.50^{ab}	10.10 ± 0.05^{ab}	7.60 ± 0.36^a	14.36 ± 2.33^b	13.56 ± 2.70^b	12.89 ± 2.79^b	11.70 ± 1.20^{ab}
Coumaric acid	17.69 ± 1.74^e	14.00 ± 0.88^d	12.54 ± 0.16^{cd}	11.05 ± 0.46^c	7.08 ± 0.01^b	5.10 ± 0.33^a	5.22 ± 0.65^a	5.84 ± 0.23^{ab}
Coumaric acid-hexose	3.85 ± 0.26^a	3.36 ± 1.00^a	3.02 ± 0.60^a	2.63 ± 0.43^a	6.74 ± 2.59^a	5.15 ± 0.11^{ab}	4.83 ± 0.21^{ab}	4.54 ± 0.41^a
Ferulic acid	5.24 ± 0.38^c	3.27 ± 0.17^{ab}	3.33 ± 0.10^{ab}	2.79 ± 0.05^a	3.49 ± 0.08^{ab}	2.79 ± 0.45^a	3.00 ± 0.01^{ab}	2.83 ± 0.35^a
Total HC acids	57.11 ± 3.84^b	41.46 ± 6.39^a	38.67 ± 2.01^a	33.79 ± 2.19^a	57.74 ± 8.30^b	52.75 ± 4.32^b	52.75 ± 3.96^b	51.83 ± 2.58^b
4-Hydroxybenzoic acid	30.87 ± 0.78^c	17.31 ± 0.95^a	18.00 ± 0.66^a	16.36 ± 0.69^a	18.59 ± 0.84^a	21.01 ± 2.09^{ab}	26.06 ± 4.06^{bc}	25.03 ± 3.83^b
Protocatechuic acid	5.27 ± 0.71^{ab}	4.62 ± 0.38^a	4.06 ± 0.04^a	3.72 ± 0.16^a	6.65 ± 0.81^{bc}	8.56 ± 0.86^d	9.13 ± 1.23^d	7.82 ± 0.44^{cd}
Vanillic acid	4.33 ± 0.00^b	4.20 ± 1.58^b	3.69 ± 0.26^{ab}	3.30 ± 0.26^{ab}	2.55 ± 0.18^a	3.10 ± 0.04^{ab}	3.32 ± 0.50^{ab}	3.56 ± 0.47^{ab}
Gallic acid	1.24 ± 0.10^b	1.03 ± 0.01^a	1.24 ± 0.10^b	1.18 ± 0.05^{ab}	1.78 ± 0.01^d	1.43 ± 0.11^c	1.43 ± 0.06^c	1.47 ± 0.07^c
Gallic acid-gal	2.42 ± 0.00^d	2.47 ± 0.10^d	1.95 ± 0.08^c	1.76 ± 0.06^b	1.42 ± 0.00^a	1.49 ± 0.11^a	1.42 ± 0.02^a	1.40 ± 0.04^a
Syringic acid	0.83 ± 0.01^a	1.05 ± 0.21^a	0.93 ± 0.04^a	0.93 ± 0.00^a	0.72 ± 0.37^a	0.83 ± 0.09^a	0.87 ± 0.15^a	0.81 ± 0.05^a
Total HB acids	44.96 ± 1.61^d	30.68 ± 3.23^{ab}	29.87 ± 1.17^{ab}	27.24 ± 1.21^a	31.71 ± 2.21^{ab}	36.52 ± 3.29^{bc}	42.22 ± 6.01^{bc}	40.10 ± 4.90^{bc}
Total phenolic acids	102.07 ± 5.45^b	72.13 ± 9.61^a	68.55 ± 3.18^a	61.03 ± 3.40^a	89.45 ± 10.50^b	89.28 ± 7.61^b	94.98 ± 9.96^b	91.93 ± 7.48^b
Total phenolic content	303.19 ± 16.19^{bc}	217.85 ± 23.07^a	214.86 ± 5.59^a	194.77 ± 8.22^a	274.79 ± 16.31^b	402.89 ± 49.90^e	358.66 ± 11.27^{de}	349.21 ± 26.48^{cd}

* HC: hydroxycinnamic acid, HB: hydroxybenzoic acid. MG: 3 magnetrons, each with a power of 1000 W. Different letters in the same row indicate significant differences between drying processes (P < 0.05).

Table 3
Impact of drying processes on the phenolic content of the peel-rich fraction (F5) of tomato by-products. The results are expressed as mg/kg sample.

Compound (mg/kg)	Raw F5 (dry weight)	Freeze-drying	Air-drying 40 °C	Air-drying 60 °C	Spiral Flash	Microwave 50% 3 MG	Microwave 75% 3 MG	Microwave 100% 3 MG
Naringenin	557.04 ± 0.37 [c]	533.49 ± 17.63 [bc]	556.66 ± 25.92 [c]	517.98 ± 3.25 [b]	370.63 ± 6.43 [a]	821.99 ± 0.76 [d]	710.98 ± 29.01	745.68 ± 1.90 [c]
Naringenin-hexose	1.87 ± 0.05 [e]	1.26 ± 0.08 [d]	1.01 ± 0.08 [bc]	0.80 ± 0.01 [ab]	1.21 ± 0.11 [cd]	0.82 ± 0.02 [ab]	0.72 ± 0.18 [a]	0.81 ± 0.02 [ab]
Total flavanones	558.91 ± 0.42 [c]	534.75 ± 17.71 [bc]	557.67 ± 26.00 [c]	518.78 ± 3.26 [b]	371.84 ± 6.53 [a]	822.82 ± 0.78 [e]	711.70 ± 29.18 [d]	746.49 ± 1.92 [d]
Quercetin	14.59 ± 0.11 [b]	12.84 ± 0.47 [a]	14.66 ± 0.32 [b]	13.24 ± 0.75 [a]	21.96 ± 0.07 [e]	17.19 ± 0.01 [c]	16.50 ± 0.09 [c]	20.88 ± 0.58 [d]
Quercetin-gluc	3.07 ± 0.46 [b]	1.87 ± 0.00 [a]	3.20 ± 0.17 [b]	2.92 ± 0.00 [b]	4.06 ± 0.08 [c]	3.39 ± 0.06 [b]	3.14 ± 0.25 [b]	3.87 ± 0.05 [c]
Kaempferol	1.68 ± 0.04 [a]	1.39 ± 0.04 [a]	1.69 ± 0.07 [a]	1.61 ± 0.16 [a]	1.81 ± 0.18 [a]	1.98 ± 0.37 [a]	1.97 ± 0.56 [a]	1.90 ± 0.19 [a]
Kaempferol-gluc	1.38 ± 0.13 [d]	0.64 ± 0.02 [a]	1.24 ± 0.06 [cd]	0.98 ± 0.08 [b]	1.04 ± 0.00 [bc]	1.10 ± 0.01 [bc]	0.93 ± 0.16 [b]	1.21 ± 0.07 [cd]
Syringetin	0.38 ± 0.02 [a]	0.38 ± 0.01 [a]	0.51 ± 0.01 [bc]	0.46 ± 0.05 [bc]	0.60 ± 0.02 [c]	0.51 ± 0.04 [bc]	0.46 ± 0.03 [ab]	0.51 ± 0.10 [bc]
Laricitrin	0.28 ± 0.11 [a]	0.34 ± 0.15 [a]	0.30 ± 0.03 [a]	0.39 ± 0.05 [a]	0.37 ± 0.04 [a]	0.38 ± 0.10 [a]	0.44 ± 0.14 [a]	0.38 ± 0.11 [a]
Total flavonols	21.37 ± 0.87 [c]	17.45 ± 0.69 [a]	21.60 ± 0.66 [c]	19.60 ± 1.08 [b]	29.83 ± 0.39 [e]	24.55 ± 0.60 [d]	23.45 ± 1.22 [d]	28.74 ± 1.10 [e]
Total flavonoids	580.29 ± 1.29 [c]	552.20 ± 18.40 [bc]	579.27 ± 26.66 [c]	538.38 ± 4.34 [b]	401.67 ± 6.93 [a]	847.37 ± 1.38 [f]	735.14 ± 30.41 [d]	775.23 ± 3.02 [e]
Caffeic acid	21.26 ± 0.05 [bc]	10.78 ± 0.28 [a]	15.94 ± 0.63 [ab]	17.93 ± 3.48 [ab]	27.10 ± 0.69 [cd]	32.46 ± 6.27 [d]	32.40 ± 5.22 [d]	32.04 ± 0.57 [d]
Caffeic acid-hexose	12.85 ± 0.49 [c]	8.00 ± 0.36 [ab]	6.54 ± 0.39 [a]	6.17 ± 0.54 [a]	15.88 ± 1.81 [d]	9.26 ± 1.33 [b]	9.51 ± 0.79 [b]	9.15 ± 0.95 [b]
Coumaric acid	27.17 ± 2.83 [c]	19.72 ± 3.80 [b]	16.89 ± 0.94 [b]	16.01 ± 0.15 [b]	10.80 ± 1.86 [a]	8.26 ± 1.26 [a]	8.68 ± 1.19 [a]	10.48 ± 0.71 [a]
Coumaric acid-hexose	5.44 ± 0.22 [cd]	3.83 ± 0.07 [abc]	2.73 ± 0.39 [a]	3.15 ± 0.97 [ab]	11.20 ± 0.41 [e]	6.32 ± 1.25 [d]	6.04 ± 1.05 [d]	4.76 ± 0.43 [bcd]
Ferulic acid	5.92 ± 0.07 [c]	4.08 ± 0.03 [b]	4.37 ± 0.02 [b]	4.25 ± 0.10 [b]	3.62 ± 0.06 [a]	3.42 ± 0.08 [a]	3.63 ± 0.16 [a]	4.09 ± 0.28 [b]
Total HC acids	72.63 ± 3.65 [c]	46.41 ± 4.53 [a]	46.47 ± 2.37 [a]	47.50 ± 5.23 [ab]	68.59 ± 4.83 [c]	59.72 ± 10.19 [bc]	60.25 ± 8.41 [bc]	60.52 ± 2.95 [bc]
4-Hydroxybenzoic acid	32.07 ± 1.50 [bc]	23.76 ± 1.43 [a]	28.36 ± 5.01 [ab]	22.70 ± 0.19 [a]	31.03 ± 4.59 [bc]	27.34 ± 0.29 [ab]	31.72 ± 1.08 [bc]	34.09 ± 0.00 [c]
Protocatechuic acid	6.65 ± 0.80 [b]	4.59 ± 0.23 [a]	4.86 ± 0.27 [a]	3.96 ± 0.17 [a]	7.64 ± 1.89 [bc]	8.41 ± 0.26 [bc]	9.40 ± 0.61 [cd]	10.45 ± 0.20 [d]
Vanillic acid	4.60 ± 0.11 [c]	3.87 ± 0.58 [abc]	4.48 ± 0.08 [bc]	4.16 ± 0.67 [abc]	3.72 ± 0.03 [abc]	3.01 ± 0.69 [a]	3.14 ± 0.70 [a]	3.38 ± 0.19 [ab]
Gallic acid	2.01 ± 0.04 [f]	1.19 ± 0.01 [a]	1.38 ± 0.01 [b]	1.48 ± 0.03 [bc]	2.93 ± 0.01 [g]	1.51 ± 0.03 [c]	1.62 ± 0.11 [d]	1.79 ± 0.03 [e]
Gallic acid-gal	3.71 ± 0.16 [e]	2.86 ± 0.11 [d]	2.11 ± 0.08 [c]	2.12 ± 0.01 [c]	1.43 ± 0.03 [b]	1.14 ± 0.02 [a]	1.15 ± 0.01 [a]	1.22 ± 0.01 [a]
Syringic acid	1.03 ± 0.04 [ab]	0.97 ± 0.19 [ab]	1.07 ± 0.28 [ab]	0.99 ± 0.07 [ab]	1.31 ± 0.07 [b]	0.96 ± 0.12 [ab]	0.79 ± 0.07 [a]	0.89 ± 0.43 [ab]
Total HB acids	50.07 ± 2.65 [d]	37.23 ± 2.55 [ab]	42.24 ± 5.72 [bc]	35.42 ± 1.14 [a]	48.06 ± 6.61 [cd]	42.37 ± 1.41 [bc]	47.83 ± 2.57 [cd]	51.81 ± 0.86 [d]
Total phenolic acids	122.70 ± 6.29 [d]	83.64 ± 7.08 [a]	88.71 ± 8.10 [ab]	82.92 ± 6.38 [a]	116.65 ± 11.44 [cd]	102.09 ± 11.61 [bc]	108.08 ± 10.99 [cd]	112.33 ± 3.81 [cd]
Total phenolic content	702.99 ± 7.59 [c]	635.84 ± 25.48 [b]	667.99 ± 34.76 [bc]	621.30 ± 10.72 [b]	518.32 ± 18.37 [a]	949.46 ± 12.99 [e]	843.22 ± 41.40 [d]	887.56 ± 6.83 [d]

* HC: hydroxycinnamic acid, HB: hydroxybenzoic acid. MG: 3 magnetrons, each with a power of 1000 W. Different letters in the same row indicate significant differences between drying processes (P < 0.05).

such as Alzheimer's (Ghofrani et al., 2015).

The flavonol content, mainly quercetin derivatives, significantly increased in Spiral Flash dehydrated TP samples, while the other drying technologies showed similar flavonol concentrations to the raw TP (Table 1). In relation to the phenolic acids, different effects were observed for each dehydration process (Table 1). Spiral Flash drying resulted in better retention of phenolic acids (115.72 mg/kg), similar to that detected in the raw TP sample (106.91 mg/kg DW). Hot air drying at 40 °C and microwave drying using 75% and 100% power showed similar results, leading to a loss of approximately 15% of the phenolic acids, compared to the raw TP sample. Surprisingly, freeze-drying resulted in a significant reduction (p<0.05) in the phenolic acid concentration (71.05 mg/kg) when compared to the raw TP (106.91 mg/kg) (Table 1). Freeze-drying removes moisture from products through the sublimation of solid ice and, due to the low temperature and low pressure environment, freeze-drying produces high-quality dried products with better appearance and higher nutrient retention compared with other drying methods (Chumroenphat et al., 2021). Nevertheless, freeze-drying is a time-consuming process, which limits its applications in the food industry. On the other hand, it is important to point out that low-molecular-weight phenolic acids in plants are covalently bound to other compounds (i.e. pectin, cellulose, and proteins) and structural changes may occur during drying processes (Chao et al., 2023). In relation to this, the low temperature and long-time conditions applied during freeze-drying would not be enough to liberate the phenolic acids from TP cuticle matrix. In addition, the long time of the freeze-drying process in our study (24 h) in relation to the other drying processes could favour the degradation of the free phenolic acids, resulting in a significant (p < 0.05) reduction in their concentration compared with the raw material (Table 1).

3.1.2. Impact of drying processes on the phenolic composition of seed-rich tomato fraction (F2)

The seeds are an important fraction of the tomato by-products, accounting for about 38.5% of the total tomato pomace (Bhatkar et al., 2021). A recent review by Kumar et al. (2021) points out important biological properties of tomato seeds for health, such as anti-oxidant, anti-cancer and anti-microbial activities. A study by Concha-Meyer et al. (2020) analyzing ultrasound-assisted extracts of tomato seed observed significant anti-platelet aggregation activity of these extracts. In the present study, the tomato seed-rich fraction (F2) showed a total phenol concentration ranging from 194.77 to 402.89 mg/kg (Table 2) similar to that observed by Valdez-Morales et al. (2014) in seeds of different tomato varieties (grape, cherry, ball and saladette), and in industrial tomato by-products. Comparing the dehydration techniques, freeze-drying and hot air-drying significantly (p < 0.05) reduced the total phenolic content in the seed-rich fraction compared to the control (raw F2) (Table 2). While Spiral Flash dryer did not affect the total phenol concentration, microwave drying resulted in an increase mainly related with the significant increase (p<0.05) in the naringenin concentration (Table 2). Similarly to what was observed in the TP, Spiral Flash drying led to a higher retention of flavonols related with the higher (p<0.05) concentration of quercetin (15.05 mg/kg) compared to the raw product (5.99 mg/kg DW) (Table 2). On the other hand, the use of the microwave technology resulted in a slight decrease in the flavonol concentration in the F2 seed-rich fraction (Table 2).

Unlike what was observed in the TP samples, in which the hot air treatments produced an increase in the contents of naringenin and other phenolic compounds, in the seed-rich F2 fraction these dehydration procedures produced a significant decrease in the concentration of naringenin in particular and in total phenol content. These differences could be related to the seed matrix composition (average carbohydrate 26 g/100 g, protein 26 g/100 g and fat 25 g/100 g) compared with the TP composition (average carbohydrate 43 g/100 g, protein 17 g/100 g and fat 15 g/100 g) (Rajan et al., 2022). These differences could explain the higher phenol retention, especially of naringenin, observed in the F2

samples submitted to microwave dehydration, mainly at the lowest power studied (50% 3 MG) (Table 2). Microwave drying is based on the transmission of electromagnetic waves, where the heat generated by molecular vibration passes through vegetal tissue generating an oscillation of the molecules, which produces the thermal energy used to evaporate water promoting porous products as a result of the drying mechanism (Pateiro et al., 2022). This increase in the porosity of the seed matrix could explain the increase in the extractability of phenolic compounds, especially naringenin, from the sample (Table 2).

3.1.3. Impact of drying processes on the phenolic composition of rich-peel tomato fraction (F5)

Regardless of the type of tomato processing, the peel constitutes the major part of the residue generated by the industry, about 61.5% of the tomato pomace (Bhatkar et al., 2021). Table 3 shows the individual phenolic compounds detected in the peel-rich tomato fraction (F5) and the effect of the different drying processes. The total phenolic content ranged from 518.32 to 949.46 mg/kg sample, 85.5% being flavonoids and 14.5%, phenolic acids. The highest retention of phenolic compounds was observed in the F5 samples obtained by microwave drying applying power at 50%, 75% and 100% with respective concentrations of total phenols of 949.46, 843.22 and 887.56 mg/kg (Table 3), and this trend was observed in the naringenin concentration. This compound was less sensitive to freeze-drying and hot-air drying (40 °C and 60 °C) maintaining similar concentrations to the raw product (Table 3). On the other hand, the Spiral Flash dryer led to a lower retention of naringenin derivatives (flavanones) (Table 3). These results suggest that microwave technology was the most efficient dehydration procedure for the retention of naringenin derivatives, similarly that observed in the TP and the F2 fraction. Regardless of the drying method, the concentrations of naringenin detected in our study were higher than those found in previous studies. Valdez-Morales et al. (2014) reported concentrations of naringenin from 0.00 to 48.6 mg/kg DW in tomato peel from different varieties. Another study by Cesare et al. (2021) reported a concentration of between 11.3 and 13.2 mg/kg DW in Italian tomato varieties grown under drought stress conditions.

Regarding flavonols, quercetin was the main compound quantified in the F5 tomato fraction with a concentration ranging from 12.84 to 20.88 mg/kg, similar values to those previously described in the literature (Grassino et al., 2020; Valdez-Morales et al., 2014). Freeze-drying and hot air-drying at 60 °C led to a slight reduction in quercetin concentrations compared to those found in the raw product. The highest retention of quercetin derivatives was observed in samples from the Spiral Flash dryer and microwave at maximum power (100% 3 MG) (Table 3). In contrast to the results observed for flavonoids, phenolic acids were more sensitive to the drying techniques studied (Table 3). All technologies resulted in a significant decrease in the total content of phenolic acids in the peel-rich tomato fraction (F5) compared to the raw product, where freeze-drying and hot air-drying (40 and 60 °C) produced higher reductions (Table 3). With the exception of the phenolic acids, the microwave drying technology showed the best results in relation to retention of phenolic compounds in the peel-rich tomato fraction (F5). This was probably related to the increase in the porosity of the peel cuticle that could result in an increase in the extractability of phenolic compounds from the peel matrix, especially the main flavanone naringenin.

3.2. Effect of drying processes on lycopene content

Lycopene is a bioactive pigment that occurs naturally in plants, and is found in large quantities in tomatoes (Martini et al., 2022). This compound can be extracted from tomato by-products and used as a food colorant, providing a color ranging from red to yellow, similar to natural and synthetic lycopene (Rizk et al., 2014; Silva et al., 2019). Due to its sensitivity to thermal processes (Bakir et al., 2023), it is fundamental to seek drying methods that obtain high retention and stability of this

compound in the dehydrated products. The results of the spectropho-tometric quantification of lycopene for each type of tomato by-product (tomato pomace, the seed-rich F2 and peel-rich F5 fractions) are shown in Fig. 2. Among the three tomato fractions studied, the peel-rich fraction (F5) showed the highest lycopene concentration (47.6–98.9 mg/kg), while the content of lycopene in the TP ranged from 11.9 to 55.6 mg/kg, and in the seed-rich fraction (F2), it varied between 10.9 and 24.6 mg/kg, similarly to that previously described in the literature (Kumar et al., 2021). Analyzing the effect of the drying process on the TP lycopene content, hot air drying (40 °C and 60 °C) resulted in a degra-dation of close to 54% of the lycopene content (12 mg/kg) compared to the fresh sample (25.9 mg/kg DW) (Fig. 2). In contrast, microwave (MW) drying resulted in higher lycopene retention in the TP (44.1–53.2

mg/kg) (Fig. 2). This difference could be due to the bulk heating phe-nomenon, promoted by electromagnetic waves in the microwave, that appreciably reduces the drying time, leading to less degradation of thermolabile compounds (Bhatkar et al., 2021). Similar results were observed with the Spiral Flash dryer, where the reduced drying time could explain the higher lycopene retention (55.6 mg/kg) (Fig. 2).

When observing the impact of the type of drying technology on the lycopene content of the seed-rich fraction (F2), the drying technologies, with the exception of the Spiral Flash, resulted in an increase in the lycopene quantified (Fig. 2). The high stability of lycopene in seeds could be related with the protective effect of the seed matrix composi-tion with high fat content (Rajan et al., 2022) against degradation during dehydration. In the literature, different quantities of lycopene
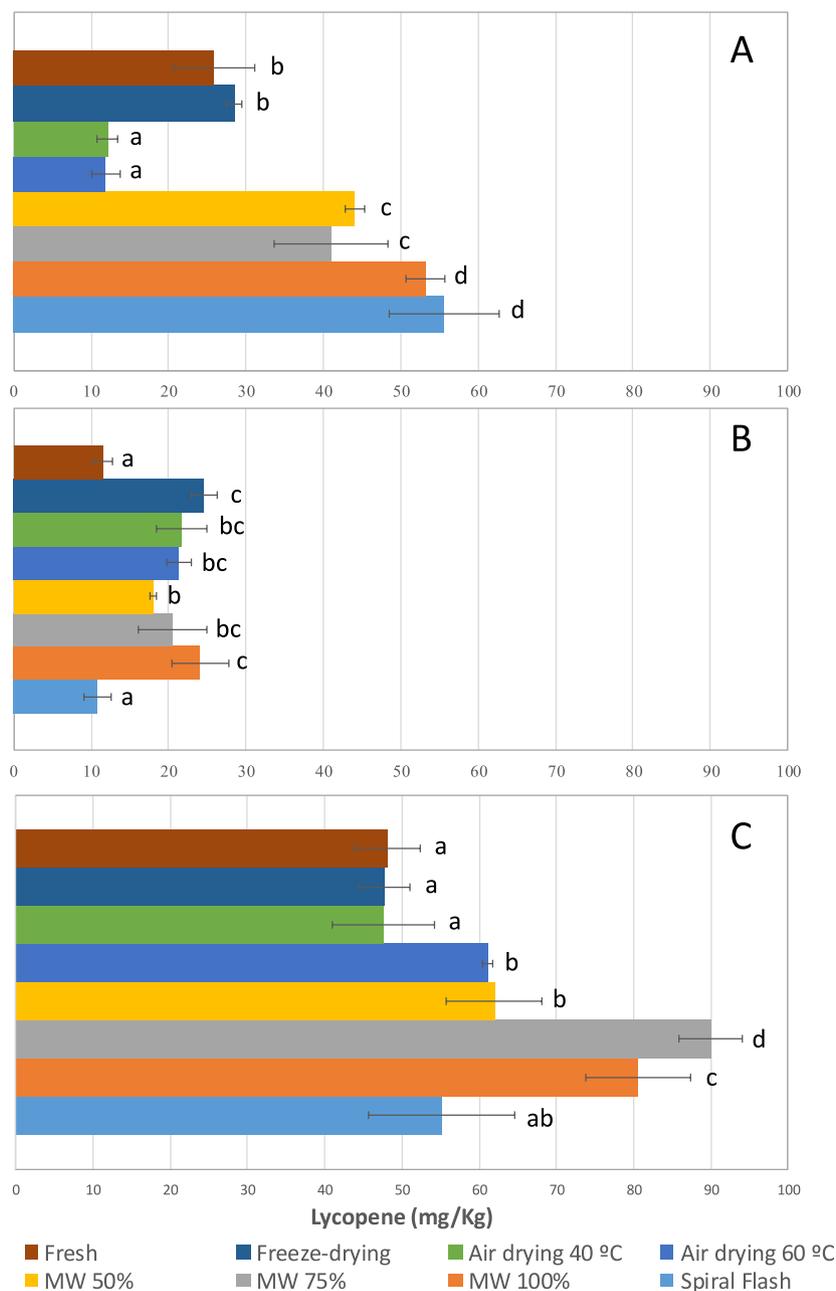
Fig. 2. Content of lycopene (expressed in mg/Kg sample) in each type of tomato by-product (A: Tomato pomace; B: Seed-rich fraction; C: Peel-rich fraction) and comparison between the dehydration methods. Different letters in the same tomato by-product present a significant difference between dehydration methods (P < 0.05).

have been reported in tomato seeds, from 16 to 167 mg/kg (Kumar et al., 2021; Silva et al., 2019).

Regarding the peel-rich tomato fraction (F5), the Spiral Flash, air drying (60 °C) and especially the microwave increased the lycopene quantified compared with the fresh sample (Fig. 2). The use of microwaves with higher powers (75% and 100%) undoubtedly resulted in better lycopene extraction from the peel matrix, with an average increase in its content, resulting in values between 89.9 and 80.7 mg/kg, respectively (Fig. 2). The impact of heat during short periods of time (microwave, Spiral Flash and hot-air at 60 °C) probably facilitates lycopene extraction from the tomato peel. In fact, the lycopene in the tomato peel is mainly in the bound form (Zuorro et al., 2013). Thus, previous studies have reported that heat processing is necessary to break down the membranes and cell walls and release lycopene from the insoluble part of the tomato, which may increase its bio-accessibility (Jayathunge et al., 2017). By contrast, heat treatment over a long period produces color degradation in the tomato peel as a consequence of lycopene degradation (Bakir et al., 2023).

3.3. Effect of drying processes on soluble dietary fiber content

Dietary fiber has attracted attention for many beneficial effects, such as increasing satiety, preventing colon cancer, lowering the risk of cardiovascular disease and reducing blood sugar (Fabek et al., 2014). Dietary fiber is either insoluble or soluble according to whether it can be dissolved in water (Arora et al., 2016). Soluble dietary fiber includes oligosaccharides, pectin and β-glucans. In our study, the soluble dietary fiber contents of the different fractions of the tomato by-products is shown in Table 4. The highest concentration of soluble dietary fiber was detected in the seed-rich fraction (F2) with a concentration ranging from 121.59 to 136.58 g/kg (average about 13%) compared with the peel-rich fraction (F5) whose concentration ranged from 64.10 to 74.24 g/kg (an average of around 7%). Regarding the dehydration methods, no significant differences were observed in the soluble dietary fiber content for each of the fractions studied (TP, F2 and F5) (Table 4), which indicates a good stability of the tomato soluble fiber during the dehydration process, regardless of the drying technology. Similarly to what was observed in the present study, previous studies have reported that the dietary fiber of tomato peel is around 8.9% soluble and 48.5% insoluble (Li et al., 2018). Another study by Grassino et al. (2016) applied conventional extraction and ultrasound-assisted extraction to obtain pectin from tomato waste, at around 15.1–35.7 g/kg, with a high extraction rate (73%). Therefore, the results of our study reveal the potential of tomato by-products, mainly the seed-rich fractions, as a source of soluble dietary fiber with commercial interest for functional food formulations.

4. Conclusion

The current study responds to the challenges of the tomato processing industry, since they have allowed the identification of dehydration technologies for tomato waste obtaining stable products that maintain their highly perishable bioactive compounds. The microwave drying technology showed the best results in the retention of the main flavonoid naringenin in tomato by-products, probably related with the increase in the porosity of the sample that favour the naringenin extractability. Moreover, the Spiral Flash air drying, which combines the advantages of flash drying and fluidized bed, revealed a great potential for application to the dehydration of tomato by-products on an industrial-scale, retaining the heat-sensitive phenolic compounds. In this system, the filtered hot air (140-160 °C) is pushed into the drying chamber by a fan generating a highly turbulent airflow that allowed the drying of the tomato pomace in a short time (20–60 s) in which the product does not exceed 45 °C. In contrast to the results observed for the flavonoids, the tomato phenolic acids (hydroxycinnamic and hydroxybenzoic acids) were more sensitive to the drying techniques studied.

Table 4
Soluble dietary fiber content in tomato by-products obtained from the different drying processes. The results are expressed as mg/kg sample and in % of the total weight.

Drying process*	Soluble fiber (g/kg)(%)					
	Tomato Pomace (TP)		Seed-rich fraction (F2)		Peel-rich fraction (F5)	
Freeze-drying	106.56	(10.6%)	131.39	(13.1%)	67.34	(6.7%)
Air drying 40 °C	112.06	(11.2%)	136.58	(13.7%)	71.63	(7.1%)
Air drying 60 °C	105.45	(10.5%)	128.77	(12.8%)	74.24	(7.4%)
Spiral Flash	101.33	(10.1%)	136.08	(13.6%)	72.28	(7.2%)
MW 50% 3 MG	99.51	(9.9%)	129.43	(12.9%)	64.10	(6.4%)
MW 75% 3 MG	100.56	(10.6%)	128.66	(12.8%)	69.66	(6.9%)
MW 100% 3 MG	106.26	(10.6%)	121.59	(12.1%)	67.52	(6.7%)

MW: Microwave. MG: 3 magnetrons, each with a power of 1000 W.
*No significant differences (P < 0.05) were observed between drying processes.

Surprisingly, freeze-drying resulted in a significant reduction in the phenolic acid concentration when compared to raw TP. The low-molecular-weight phenolic acids in plants are covalently bound to other compounds (i.e. pectin, cellulose, and proteins) and structural changes may occur during drying processes. Therefore, the low temperature conditions applied during freeze-drying would not be enough to liberate the phenolic acids from the tomato samples. Regarding lycopene, the use of microwaves with higher powers resulted in better lycopene retention in tomato by-products. The content of soluble dietary fiber was not affected by different dehydration technologies. The present study reinforces the interest in tomato by-products as a source of naringenin, a flavonoid with important nutraceutical properties. In addition, this study reveals the potential of peel-rich and seed-rich tomato fractions as sources of lycopene and soluble dietary fiber, respectively.

CRediT authorship contribution statement

Bianca Souza da Costa: Research, Methodology, Validation, Formal analysis, Writing – original draft. **Marta Oliván García:** Research, Formal analysis, Writing – review & editing. **Germán Soldevilla Muro:** Project administration, Supervision, Funding acquisition, Writing – review & editing. **Maria-Jose Motilva:** Conceptualization, Methodology, Writing – original draft, Supervision, Project administration, All the authors have read and approved the final manuscript.

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.lwt.2023.114644.

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