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ORIGINAL ARTICLE

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Comparison of different drying methods for bergamot peel: Chemical and physicochemical properties

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Abstract: This study examines the effectiveness of seven drying methods applied to bergamot peels, encompassing hot air, microwave, infrared-assisted microwave, freeze, infrared, sun, and oven drying. All samples exhibited moisture content and water activity levels within the acceptable range for dry foods. All methods effectively transformed the peels into powdered form, yielding comparable results. Each method offers distinct advantages and disadvantages, and the choice of method should be based on the desired properties of the final product. The highest ascorbic acid content was found in freeze-dried and hot air-dried samples (>400 mg/100 g), whereas sun-dried samples had the lowest (89.58 mg/100 g). Infrared-dried samples exhibited the highest levels of total phenolics and flavonoids (193.40 and 530.14 mg/100 g, respectively), attributed to reactions induced by elevated temperatures. The total carotenoids were higher in freeze-dried samples (54.12 mg/100 g) compared to other drying methods (<27 mg/100 g). Microwave-dried samples had the highest 5-hydroxymethylfurfural content (73.06 mg/100 g), and freeze-dried samples had the highest naringin content (1568.70 mg/100 g). Although infrared drying had good particle density, porosity, and fluidity, freeze-drying was the most effective, retaining the highest levels of bioactive compounds. Among the methods studied, freeze-drying is recommended due to its superior ability to preserve bioactive compounds. Infrared and infrared-assisted drying methods were suitable for recovering phenolics from bergamot waste, offering lower energy consumption and practical preservation of physicochemical properties. This study emphasizes the importance of selecting the appropriate drying method to ensure high-quality dried food and producing value-added products from bergamot waste, contributing to sustainable agriculture and waste reduction.

KEYWORDS
Citrus bergamia, functional food

Practical Application: This study demonstrates that infrared and freeze-drying are the most effective methods for producing high-quality bergamot peel samples with enhanced antioxidant properties. These findings hold promising

implications for the food industry, offering a viable approach to preserve bergamot peels and their valuable attributes.

1 | INTRODUCTION

Citrus fruits and their byproducts are rich and renowned for their abundance of bioactive compounds and phytochemicals, including pectin, flavonoids, phenolics, antioxidants, and natural colorants (Andrade et al., 2022). Citrus peels, constituting a significant portion of the fruit's weight, have garnered attention due to their potential uses and associated health benefits. Nevertheless, the disposal of citrus peels, which result from fruit processing for juice and essential oil extraction, poses environmental challenges due to the risk of microbial spoilage (Ozcan et al., 2021; Wedamulla et al., 2022).

Three cultivars of bergamot (*Citrus bergamia*) are known: *Castagnaro*, *Fantastico,* and *Femminello* (Gioffrè et al., 2020; Giuffrè, 2019; Giuffrè & Nobile, 2020). The analysis of the total endocarp of bergamot, excluding the seeds, was sourced from the study conducted by Cuzzocrea and Centonze (1951). According to their findings, the composition per cent is as follows: water 87.57, total N 0.1151, protein 0.7194, ether extract 0.6282, alcohol extract expressed as glucose 3.6, ash 0.3657, and vitamin C presumably ranging from 44 to 83 mg/100 g. The composition of bergamot can vary based on the harvest date and cultivar (Gioffrè et al., 2020; Giuffrè, 2019; Giuffrè & Nobile, 2020). The proximate composition of bergamot was reported as follows by Cautela et al. (2019): 9.5% total soluble solids, 43 g/L acidity, 421 mg/L L-ascorbic acid, 562 mg/L pectins, 278 mg/L water-soluble pectins, 18 g/L sucrose, 13.1 g/L glucose, and 12.6 g/L fructose. A study conducted during fruit ripening found that vitamin C content decreased with ripening and showed a 50% decrease from October to March. In contrast, total flavonoids were characterized by the maximum content between December and January (Giuffrè, 2019). Sicari et al. (2016) found that the geographical area of production influenced color (L^* 1.83–7.60; a^* 0.05–0.29; b^* 0.60–4.82); pH (2.54–2.84); acidity (11.38–14.83 g/L); formol index (2.53–3.66); ascorbic acid (89.40–285.35 mg/100 mL); flavonoid content (51.11–148.15 mg/100 mL); and polyphenol content (180.53–233.36 mg/100 mL). Additionally, bergamot exhibits a total water-soluble solid of 7.93%, a pH of 2.77, an acidity of 2.56%, a vitamin C content of 20,335 mg/100 g, antioxidant activity of 79.23%, a total phenolic content of 53,745 mg/100 g, and total flavonoids of 8450 mg/100 g (Demircan et al., 2023; Giuffrè, 2019).

Bergamot distinguishes itself from other citrus fruits with its high content of flavonoids and glycosides such as neoeriocitrin, neohesperidin, naringin, rutin, and poncirin (Giuffrè & Nobile, 2020; Lamiquiz-Moneo et al., 2020). The phenolic compound composition of bergamot fruit varies across its different parts, namely, the fruit, peel, juice, flavedo, and albedo. Neoeriocitrin content is highest in the albedo part (435 mg/100 g), followed by the peel (331 mg/100 g). Naringin is abundant in the flavedo part (670 mg/100 g) and poncirin peaks in the juice (1870 mg/100 g). Neohesperidin is notably present in the juice (762 mg/100 g), whereas rutin is predominant in the flavedo part (89.7 mg/100 g). Among the flavonoids, hesperidin is minimal, with the highest concentration in the peel (2.6 mg/100 g). Diosmin is significantly present in the flavedo part (82.5 mg/100 g) (Nogata et al., 2006).

In contrast to its counterparts, bergamot is primarily used for extracting volatile oil from its peel, whereas several byproducts, including juice, peel, residual pulp, and seeds, are generated during the oil extraction process. Despite the richness of these byproducts in sugars, fibers, phenolics, and other valuable components, the peel, both outer and inner parts, is typically classified as waste. It contains distinctive *Citrus* flavanone rutinosides and neohesperidose from naringenin, eriodictyol, and hesperetin. Unique flavone *O*- and *C*-glycosides, previously unreported in orange and lemon peels, have also been detected (Baron et al., 2021). Russo et al. (2016), Mandalari et al. (2006), and Siano et al. (2023) conducted chemical analyses of the various parts of the bergamot fruit and found the peel to be the richest in bioactive molecules. Di Donna et al. (2011) found two new flavonoids in Bergamot albedo, that is, brutieridin and melitidin, which proved an anticholesterolemic effect in vitro. Bartella et al. (2022) applied nuclear magnetic resonance and high-resolution tandem mass spectrometry to bergamot juice and found peripolin, a new 3-hydroxy-3-methylglutaryl flavonoid.

The byproduct of bergamot essential oil production, Pastazzo, is industrially dried for pectin production and used as animal feed (Russo et al., 2016). Drying the peels may also offer an alternative for processing Pastazzo. Bergamot peels present an excellent raw material for isolating pure bioactive molecules in functional foods.

In a study on bergamot pomace (peels, pulp, and seeds), the authors investigated the effect of different extraction methods: conventional (by pressure), ultrasound-assisted, and microwave-assisted. They reported varying

total phenolic content (mg gallic acid/dry weight) as follows: 12–26.30 (conventional), 18.63–23.64 (ultrasound-assisted), and 9.53–19.44 (microwave-assisted). Similarly, the total flavonoid content (mg catechin/dry weight) exhibited variation: 2.67–5.72 (conventional), 6.02–7.80 (ultrasound-assisted), and 2.92–5.27 (microwave-assisted) (Gattuso et al., 2023). Maiuolo et al. (2023) conducted a high-pressure liquid chromatographic analysis. They found brutieridin, melitidin, naringin, neoeriocitrin, and neohesperidin as flavonoids in bergamot fiber, besides naringin, neoeriocitrin, and neohesperidin as significant components.

Bergamot peels are prone to rapid deterioration due to their high moisture content (up to 75%) (Marey & Shoughy, 2016; Rafiee et al., 2007). The drying process, a conventional method of food preservation, reduces moisture content, curbing microbial growth, chemical degradation reactions, and enzymatic activities (Ozdemir et al., 2018). Dried bergamot peel is recommended for various food products, including beverages, bakery items, dairy products, confectionery, and essential oil extraction (Farahmandfar et al., 2020).

Heat treatment is commonly employed for drying fruits and their byproducts, but prolonged exposure to high temperatures can lead to the loss of bioactive compounds. Different drying methods, with varying energy transmission, result in varying time required for drying and product quality (Ozcan et al., 2021).

Solar drying, an economical and low-cost method that uses solar energy is the traditional approach, but it has limitations, including uneven drying and dependence on weather conditions. Furthermore, products dried using this method are exposed to environmental pollution (Farahmandfar et al., 2020). Oven drying is slow due to moisture diffusion from the product's center to its surface, and high temperatures may not necessarily improve the product quality. Hot air drying, while cost-effective, negatively affects product quality even when low temperatures are used. Freeze-drying is optimal for preserving product quality but is energy-intensive, expensive, and time-consuming, with some volatile components lost due to water sublimation (Chua et al., 2019; Karam et al., 2016). Freeze-drying can lead to a significant loss of volatile compounds (Ma et al., 2023). These compounds are sensitive to heat and tend to evaporate during freeze sublimation, especially if they have higher vapor pressures than water (Chin et al., 2008). As a result, innovative drying methods have been explored to supplement these conventional techniques, with microwave and infrared drying offering shorter drying durations and faster drying rates (Kumar & Karim, 2019; Sakare et al., 2020; Tekgul & Baysal, 2018).

The choice of the most suitable drying method depends on the specific characteristics of the raw materials (Calín-Sánchez et al., 2020; Michalska et al., 2017). However, there is a paucity of published data regarding the chemical composition of dried bergamot peel using different drying techniques. This study aims to fill this gap by investigating the effects of various drying methods on the phenolic profile and physical–chemical properties of dried bergamot peels.

Previous research has explored the utilization of bergamot peel in various forms, including incorporation into chocolate, lyophilization for flavonoids and pectin extraction, and as an ingredient in biscuit formulations (Gabriele et al., 2017; Laganà et al., 2022; Mandalari et al., 2006). However, comprehensive published data on the chemical composition of dried bergamot peel through diverse drying techniques remain limited. Therefore, this study aims to contribute to understanding the potential applications of dried bergamot peels as a cost-effective source of phenolics in foods.

2 | MATERIALS AND METHODS

The bergamot fruit (*C. bergamia* Risso et Poiteau) used in this study was sourced from Tekirova-Antalya, Turkey, in February 2022. These bergamots, particularly their peels, were the same as those characterized in our prior research (Demircan et al., 2023). The trees, which belonged to the Italian-originated Castagnaro variety, were cultivated by experienced farmers in moderately sunny and lightly shaded areas. The trees were 3–5 m tall and approximately 40–50 years old. Organic fertilizers specific to citrus were regularly applied during the growth phase, and the trees received minimal irrigation. The soil where the trees were grown had a slightly acidic pH range of 5.5–6.5 and was well-drained.

Approximately 10 kg of fruit, separated from the stems and leaves, were carefully washed, halved, and then juiced in a laboratory fruit squeezer (Moulinex, FP519GB1). The flavedo of fruits was meticulously decorticated with a knife, cut into small pieces (approximately 1 × 1 cm, 4.3 kg) and stored at −20°C until use.

All chemicals used in this research were analytical or HPLC grade and procured from Sigma-Aldrich, Merck, and Carlo Erba.

2.1 | Drying process of bergamot peels with different methods

The drying process of bergamot peels encompassed a variety of methodologies. Initially, the peels underwent thawing at +4°C, followed by division into 100-g batches. The primary objective was to reduce the initial moisture

content of 77.6% to approximately 10% (wet basis). The employed drying techniques comprised the following: hot air oven drying at 60°C with an air velocity of 1.50 m/s for 204 min (Eksis, TK-LAB); microwave oven drying at 6 W/g for 10 min (Arcelik, MD 554); infrared-assisted microwave oven drying at 6 W/g for 10 min (Siemens); freeze-drying at a pre-freezing temperature of −20°C, condenser temperature of −53.6°C, heating plate temperature of 40°C, and a chamber pressure of 365 Pa for 24 h (TOPT-10D Shaanxi); infrared oven drying at 250°C for 53 min (UHF Necat Makina, INF-95,050) (utilizing a specially designed prototype with 18 flat electrically operated and temperature-controlled ceramic heaters equipped with Ceramicx ceramic heat sources, each with a maximum surface temperature of 500°C and 500 W power operating with the IR wavelength; the ceramic heaters were stabilized at 250°C before the drying process); sun drying for 48 h (in an open area on a cloth, with temperatures ranging from 17 ± 2°C at night and 28 ± 2°C during the day, a wind speed of 5 km/h, and a humidity of 47% ± 2% on the days when drying is done); and laboratory oven drying at 60°C for 300 min (Simsek Laborteknik). Each drying treatment was conducted in triplicate to ensure the accuracy and reliability of the results.

The process conditions (temperature and time) were determined through preliminary trials. Specifically, experiments conducted at higher temperatures using infrared, hot air, and oven drying methods resulted in low product quality (especially excessive browning and burning). In microwave drying, higher power levels cause adverse effects on the product (such as surface burning).

The moisture, ash, fat (analyzed using the Soxhlet extraction method), protein (determined through the Kjeldahl method with a nitrogen-to-protein conversion factor of 6.25), and carbohydrate content in dried peels were evaluated according to the methods outlined by Association of Official Analytical Chemists (1990), and the data is given in Table 1. According to the data provided in Table 1, the infrared-assisted microwave drying method stands out with the lowest ash content (3.17%). The freeze-drying method, on the other hand, attracts attention with the highest fat content (7.23%) and protein content (3.94%). In comparison, the microwave-drying method is characterized by the lowest fat content (3.94%) and moisture content (9.95%), on average. The infrared drying method, on the other hand, distinguishes itself with the highest carbohydrate content (79.39%). These results emphasize the factors that should be considered when evaluating the performance of different drying methods in preserving specific components.

The dried peels were ground into a powder using an electric grinder (Fakir, 41,001,921) and screened through a stainless steel sieve with a 1.0 mm aperture size (Retsch,

TABLE 1 The approximate chemical composition of dried bergamot peels (%).

Drying methods	Ash	Moisture	Fat	Protein	Carbohydrate
Hot air drying	3.27 ± 0.07^{e}	9.86 ± 0.09^{c}	4.73 ± 0.01^{e}	3.09 ± 0.04^{c}	79.25 ± 0.21^{a}
Microwave drying	4.09 ± 0.08^{b}	9.95 ± 0.03^{a}	3.94 ± 0.04^{f}	3.06 ± 0.04^{c}	78.96 ± 0.19^{b}
Infrared-assisted-microwave drying	3.17 ± 0.03^{f}	9.76 ± 0.06^{b}	5.67 ± 0.02^{d}	3.04 ± 0.03^{c}	78.36 ± 0.08^{c}
Freeze drying	3.32 ± 0.01^{e}	9.56 ± 0.06^{d}	7.23 ± 0.04^{a}	3.94 ± 0.02^{a}	75.95 ± 0.05^{e}
Infrared drying	3.98 ± 0.01^{c}	9.72 ± 0.03^{bc}	3.90 ± 0.01^{f}	3.01 ± 0.05^{c}	79.39 ± 0.02^{a}
Sun drying	3.74 ± 0.04^{d}	9.71 ± 0.01^{bc}	5.82 ± 0.02^{c}	3.50 ± 0.09^{b}	77.23 ± 0.16^{d}
Oven drying	4.51 ± 0.01^{a}	9.69 ± 0.04^{bc}	6.72 ± 0.02^{b}	3.06 ± 0.02^{c}	76.02 ± 0.09^{e}

Fisher Scientific Company) to achieve a homogenous sample mass for analysis. The powdered samples were then vacuum-sealed in polypropylene bags using a vacuum packaging machine (Audionvac, VMS 153) with a vacuum time of 30 s and stored at −20°C until used for further experiments.

The following analyses were performed on the results calculated based on 100 g of dried and powdered bergamot peel material prepared using different drying methods. All experiments were conducted in triplicate with three parallels.

2.2 | Drying yield and energy consumption of process

To determine the drying yield (%), the weight of the dried sample after the drying process (g) was divided by the weight of the fresh sample before drying (g), and the result was expressed as a percentage (Nguyen et al., 2016).

Energy consumption (kW h) is calculated using the following formula:

$$\text{Energy consumption (kW h)} = P \times t$$

where P is the power of the device used for drying (in kW), and t is the total time it takes to reduce the product moisture to ~10% during the process (in h). The required power value here is calculated by multiplying the voltage (V) and current (amps) of the device used (such as microwave dryer, freeze dryer, and infrared dryer) according to its specifications (Xu et al., 2006). On the other hand, energy consumption has yet to be calculated for sun-dried samples.

2.3 | Color

The color properties of peel powders were determined with a colorimeter (Konica Minolta, CR-400). Surface measurements of L^* (lightness), a^* (redness/greenness), and b^* (yellowness/blueness) were conducted on peel powders. The total color difference (ΔE^*), chroma (c^*), and hue angle ($H°$) were calculated with some modifications to the methodology outlined by Pathare et al. (2013).

2.4 | Moisture content

The moisture content (%) of the peel powders was determined gravimetrically by drying them in an oven (Simsek Laborteknik) at 105°C for 24 h (International Organization for Standardization, 2017).

2.5 | Water activity

The water activity of the peel powders was determined with a water activity meter (Aqualab, 4TE) at 25°C.

2.6 | Water holding capacity

Peel powder (0.5 g) was mixed with 10 mL of distilled water using a vortexer (Heidolph, D-91126) for 1 min. The resulting mixture was then heated at 60°C for 30 min in a water bath (ISOLAB Laborgerate, GmbH) and then cooled in an ice bath for 30 min and centrifugation (Hettich Zentrifugen, Universal 320R) at 11,228 *g* for 20 min at 25°C. Gravimetric analysis was used to determine the water holding capacity (g/g) by measuring the weight of the pellet left behind after removing the supernatant (upper phase), as per the methodology described by Anderson (1982).

2.7 | Water solubility

The powder was mixed in an orbital shaker (Heidolph, Unimax 2010) with distilled water (1:50, w/v) at 300 rpm for 1 h at room temperature and passed through coarse filter paper. The residues remaining on the filter paper were dried at 105°C for 2 h, and the solubility (%) was calculated gravimetrically (Chau et al., 2007).

2.8 | Browning index

Peel powder (0.5 g) and ethanol (15 mL, 75% (v/v)) vortexed for 1 min and kept in a water bath at 75°C for 2 h. The samples were filtered through coarse filter paper, and the absorbance was read at 420 nm using a UV–Vis spectrophotometer (Shimadzu, UV-1601) (Yu et al., 2020).

2.9 | Ascorbic acid content

Peel powder (1 g) was extracted with 30 mL of 0.8% (w/v) metaphosphoric acid, vortexed for 1 min, and centrifuged at 11,228 *g* for 10 min at 4°C. The supernatant was filtered through the Whatman No 4 filter paper, and the final volume was made up to 50 mL with 0.8% (w/v) metaphosphoric acid. The extract filtered through a 0.45 μm PTFE filter was analyzed using HPLC (Shimadzu, LC-20AD). Analysis was performed using a C18 column (Macherey-Nagel, EC 250/4.6 Nucleosil 300-5) at 25°C oven (Shimadzu, CTO-10ASVP) temperature, 15 min elution time, and 0.8 mL/min flow rate (isocratic). The

mobile phase was 0.8% (w/v) metaphosphoric acid. Photodiode array (PDA) detection (Shimadzu, SPD-M20A) was performed at 246 nm. Concentrations of ascorbic acid were determined using L-ascorbic acid (Sigma-Aldrich) calibration curves ($y = 50297x + 480{,}177$, $R^2 = 0.9993$) prepared by dissolving standard (0–1000 mg/100 g) in 0.8% metaphosphoric acid (Vikram et al., 2005).

2.10 | Total carotenoid content

Peel powder (1 g) was mixed with a 10 mL solution of hexane:acetone:ethanol (50:25:25, v/v) and agitated on an orbital shaker at 250 rpm for 10 min at room temperature. The mixture was centrifuged at 11,228 *g* for 5 min at 10°C, and the resulting supernatant was made up to 10 mL with the same solution. The absorbance was measured at 450 nm using a UV–Vis spectrophotometer. ß-Carotene was used as the standard for the calibration curve ($y = 0.001x + 0.0028$, $R^2 = 0.9955$), and the results were determined as ß-carotene equivalents (BCE) (De Ritter & Purcell, 1981).

2.11 | 5-Hydroxymethylfurfural content

Bergamot peel powder (0.5 g) and 10 mL of distilled water were vortexed for 1 min and kept in an ultrasonic water bath at 100% power, 35 kHz frequency, and 25°C for 20 min. Then, it was centrifugated at 20°C 11,228 *g* for 10 min, filtered through a 0.45 µm PTFE filter, and analyzed using HPLC. Analysis was performed using a C18 column (Macherey-Nagel, EC 250/4.6 Nucleosil 300-5) at 30°C oven (Shimadzu, CTO-10ASVP) temperature, 25 min elution time, and 1 mL/min flow rate. The mobile phase contained 99% acetonitrile (mobile phase A) and 0.2% (v/v) formic acid (mobile phase B). In analysis, gradient flow was used 0–10 min %5A, 10–25 min %5–80A. PDA detection (Shimadzu, SPD-M20A) was performed at 280 nm. Concentrations of 5-hydroxymethylfurfural (5-HMF) were determined using 5-HMF (Merck) calibration curves ($y = 78902x + 4E + 06$, $R^2 = 0.9926$) prepared by dissolving standard (0–1000 mg/100 g) in distilled water (International Federation of Fruit Juice Producers, 1985).

2.12 | Preparation of phenolic extracts

Peel powder (0.1 g) was vortexed with 10 mL of hot distilled water for 1 min and kept in a water bath at 95°C for 15 min. Then, the samples were filtered through coarse filter paper, and the prepared extract was used to analyze total phenolic content, total flavonoid content, antioxidant activity, and determination of individual phenolics (Papoutsis et al., 2016).

2.13 | Total phenolic content

Folin–Ciocalteu reagent (5 mL, 0.1 M) was added to 1 mL of powder extract, followed by 4 mL of 7.5% (v/v) Na_2CO_3, and vortexed for 2 min. The absorbances of the solutions incubated for 1 h at room temperature in the dark were measured at 760 nm using a UV–Vis spectrophotometer. Gallic acid was used as the standard for the calibration curve ($y = 0.0103x + 0.113$, $R^2 = 0.9959$), and the results were determined as gallic acid equivalents (GAE) (Vuong et al., 2013).

2.14 | Total flavonoid content

Powder extract (0.5 mL) was mixed with 2 mL of distilled water and 0.15 mL of 5% (w/v) $NaNO_2$ and left at room temperature for 6 min. Then, 0.15 mL of 10% (w/v) $AlCl_3$ was added and kept at room temperature for 6 min. Afterward, 2 mL of 4% (w/v) NaOH and 0.7 mL of distilled water were added to the mixture. The absorbance of the final solution was measured at 510 nm using a UV–Vis spectrophotometer after letting it stand at room temperature for an additional 15 min. Catechin was used as the standard for the calibration curve ($y = 0.0029x + 0.0809$, $R^2 = 0.9906$), and the results were determined as catechin equivalents (CE) (Zhishen et al., 1999).

2.15 | Antioxidant activity

A volume of 2.85 mL of 1.1×10^{-4} M 2,2-diphenyl-1-picrylhydrazyl solution was added to 0.15 mL of powder extract. The absorbance at 515 nm was measured using a UV–Vis spectrophotometer after incubating at room temperature for 30 min in the dark. Trolox was used as the standard for the calibration curve ($y = -0.0037x + 0.9963$, $R^2 = 0.9987$), and the results were determined as Trolox equivalents (TE) (Thaipong et al., 2006).

2.16 | Determination of individual phenolics

HPLC determined individual phenolic compounds in the extracts filtered through a 0.45 µm PTFE filter. Analysis was performed using a C18 column (Macherey-Nagel, EC 250/4.6 Nucleosil 300-5) at 30°C oven (Shimadzu, CTO-10ASVP) temperature, 40 min elution time, and 1 mL/min

flow rate. For the analysis, the mobile phase consisted of a mixture of water, acetonitrile, and formic acid in a ratio of 95:4:1 (v:v:v) (mobile phase A) and acetonitrile (mobile phase B). The gradient flow used during the analysis was as follows: 0 min, 5% B; 15 min, 20% B; 32 min, 5% B; 40 min, 5% B. PDA detection (Shimadzu, SPD-M20A) was performed at 280 nm. We used external standards to quantify the identified phenolic compounds, with each standard compound prepared separately by dissolving them (0–1000 mg/100 g) in 80% methanol. The concentrations of phenolics were determined using the calibration curves for each phenolic component (Papoutsis et al., 2017).

2.17 | Characterization of peel powders

In addition to the above analyses, the primary physical characteristics of the powders were assessed through examinations of hygroscopicity, caking degree (Cai & Corke, 2000), bulk density, tapped density, particle density, porosity (Jinapong et al., 2008), flowability (Carr, 1965), and cohesiveness (Hausner, 1967).

2.18 | Statistical analysis

The data obtained from the analysis were evaluated using a factorial analysis of variance, and the Duncan test was applied as a comparison. All statistical analyses were performed using SPSS 22.0 (SPSS Inc.) software, with differences considered $p < 0.05$ significant.

3 | RESULTS

3.1 | Yield and energy consumption

Bergamot peels were subjected to various drying methods, including hot air, microwave, infrared-assisted microwave, freeze-drying, infrared, sun, and oven drying. The resulting powders displayed a range of colors, from light yellow to dark yellow-light brown.

The process yields and energy consumption associated with different drying methods for bergamot peel powders are presented in Table 2. Process yield, calculated as the ratio of the final powder weight obtained after drying and grinding to the initial wet sample weight, was evaluated. The influence of these methods on the powder yield was consistent with previous studies (Nguyen et al., 2016). The microwave (22.22%), infrared-assisted microwave (23.52%), and infrared drying (23.90%) processes exhibited lower efficiency, primarily due to the formation of a surface crust during drying, leading to some powder loss during grinding and screening (Gopinathan et al., 2020). The quantity of product obtained after drying depends on the raw materials characteristics and drying equipment used. Although there were variations in process yield values among the methods, no statistically significant differences were observed for some of them.

Energy consumption is closely related to drying duration and the operational parameters of the equipment, such as power, current, and frequency. Among the evaluated drying methods, freeze-drying was identified as the most energy-intensive process, requiring 55.20 kW h, albeit resulting in high-quality final products. In contrast, hot air drying was associated with moderate energy consumption (17.00 kW h), whereas infrared and oven drying methods exhibited relatively lower energy consumption (7.95 and 4.50 kW h, respectively). Microwave and infrared-assisted microwave drying processes had minimal energy consumption (0.10 and 0.15 kW h, respectively) due to their shorter processing times. Notably, sun drying, utilizing solar energy, had negligible energy consumption. The energy consumption among all the methods is statistically significant. The observed differences in energy consumption among the methods were statistically significant.

In summary, freeze-drying, as noted by Papoutsis et al. (2017), had the highest energy consumption but yielded excellent product quality. Microwave and infrared-assisted microwave drying methods had the lowest energy consumption due to their shorter processing times, aligning with findings from Orphanides et al. (2013) and Nguyen et al. (2016), highlighting their drying time and energy consumption efficiency.

3.2 | Color coordinates (L^*, a^*, b^*) and browning index

Color is a predominant and crucial sensory quality attribute in dried products, often influenced by the heat treatments applied (Bozkir et al., 2021). The color values of bergamot peels subjected to various drying methods are provided in Table 3. L^*, representing lightness, and b^*, indicating yellowness, were the highest freeze-dried samples (89.53 and 29.97, respectively), whereas the lowest was observed in the microwave-dried samples (66.31 and 24.60, respectively). Freeze-drying, characterized by very low temperatures, prevents browning reactions, resulting in more stable color coordinates. Furthermore, freeze-drying is known to retain ß-carotene better, as evidenced by the high b^* value (Caparino et al., 2012; Rafiq et al., 2019). In contrast, the L^* and b^* values obtained in other drying methods are generally lower than in freeze-drying. This discrepancy can be attributed to the development of a darker crust in these methods, often associated with sugar

TABLE 2 Process yield (PY) and energy consumption (EC) of drying methods.

Drying methods	PY (%)	EC (kW h)
Hot air drying	25.17 ± 1.89^{ab}	17.00 ± 0.01^{b}
Microwave drying	22.22 ± 0.52^{b}	0.10 ± 0.01^{e}
Infrared-assisted-microwave drying	23.52 ± 1.41^{ab}	0.15 ± 0.01^{e}
Freeze drying	26.41 ± 0.23^{a}	55.20 ± 0.01^{a}
Infrared drying	23.90 ± 1.02^{ab}	7.95 ± 0.01^{c}
Sun drying	24.93 ± 0.96^{ab}	ns
Oven drying	25.09 ± 2.01^{ab}	4.50 ± 0.01^{d}

Note: The values in the table indicate mean ± standard deviation. Different letters in the same column show differences between sample groups according to the Duncan test ($p < 0.05$).
Abbreviation: ns: not subjected.

caramelization or the Maillard reaction, causing browning and the degradation of the carotene pigment during heat treatment (Rafiq et al., 2019). The opposite trend is observed in the a^* value, with the lowest value recorded in the freeze-dried samples (−3.38) and the highest in the microwave-dried samples (5.94). The a^* value reflects redness, which increases as the yellow crust color depends on high temperatures.

The ΔE^* value was lowest in the sun-dried samples (25.99), indicating that the color of these samples closely resembled that of the fresh product. This is attributed to the low product temperature during sun-drying. The c^* value was highest in the freeze-dried samples (30.16) and lowest in microwave-dried samples (25.31). Furthermore, the highest $H°$ value was observed in the hot air-dried samples (85.65), whereas the microwave-dried samples had the lowest $H°$ value (76.46). The lowest ΔE^* value in the sun-dried samples signifies that their color was closest to the fresh samples. This is due to the lower product temperature during sun-drying.

The browning index, an indicator of the brown color's purity resulting from enzymatic and nonenzymatic browning reactions (Farahmandfar et al., 2020), was highest in the microwave-dried samples (2.17) and lowest in the freeze-dried samples (0.97). Our findings align with data from various drying methods reported in the literature (Bozkir et al., 2021; Farahmandfar et al., 2020; Rafiq et al., 2019; Tekgul & Baysal, 2018). The difference between the methods regarding color values was statistically insignificant, except for the c^* value.

3.3 | Moisture content, water activity, water holding capacity, and water solubility

Moisture content, a crucial factor influencing physicochemical properties, and water activity values contributing to product stability are vital considerations for dried food products. The moisture content of bergamot peels subjected to various drying methods, as presented in Table 4, ranges from 9.56% to 9.95%, whereas water activity values range from 0.2626 to 0.4256.

The lowest moisture content and water activity were observed in the freeze-dried samples (9.56% and 0.2626%), whereas the highest values were found in the microwave-dried samples (9.95% and 0.4256%). Although there were variations among the methods, no statistically significant differences were identified. Freeze-drying directly removes water from the structure through sublimation, resulting in the lowest values. In contrast, microwave drying, known for achieving volumetric heating within a few centimeters beneath the sample's surface, tended to maintain relatively higher moisture content and water activity levels in bergamot peels than other drying methods. As demonstrated in Table 3, elevated moisture content has been linked to increased enzymatic degradation, contributing to the development of a darker color (Farahmandfar et al., 2020). These findings align with previous literature. Bozkir et al. (2021) reported moisture content ranging from 7.99% to 9.96% in orange peels, Ozcan et al. (2021) found values between 10.12% and 15.23% in lemon peels, and Sogi et al. (2013) reported values ranging from 5.19% to 6.77% in mango peels when using different drying methods. Tekgul and Baysal (2018) noted a minimum water activity value (0.42) for freeze-dried lemon peels.

Water holding capacity serves as an indicator of the powders' rehydration capability. Consistent with previous studies, the highest water-holding capacity was observed in freeze-dried samples (8.09%). This can be attributed to tissue damage and degradation during the freeze-drying process. A linear relationship exists between water-holding capacity, representing the affinity of the components in the dried powders for water, and water solubility. In this context, statistically significant differences were identified among the methods. Similar outcomes were reported by Sogi et al. (2013) in dried mango peels using various drying techniques.

TABLE 3 Color coordinates (L^*, a^*, b^*), total color difference (ΔE^*), chroma (c^*), hue angle ($H°$) values, and browning index (BI) of bergamot peel powders.

Drying methods	L^*	a^*	b^*	ΔE^*	c^*[1]	$H°$	BI (abs)
Hot air drying	80.02 ± 0.14^{b}	2.30 ± 0.21^{e}	29.92 ± 0.11^{a}	27.15 ± 0.15^{b}	30.01 ± 1.19	85.65 ± 1.13^{a}	0.99 ± 0.03^{c}
Microwave drying	66.31 ± 2.45^{c}	5.94 ± 0.28^{a}	24.60 ± 0.71^{d}	28.06 ± 1.91^{b}	25.31 ± 0.61	76.46 ± 1.68^{b}	2.17 ± 0.28^{a}
Infrared-assisted-microwave drying	66.74 ± 1.10^{c}	5.48 ± 0.51^{ab}	25.10 ± 0.83^{cd}	27.55 ± 2.78^{b}	25.69 ± 1.34	77.72 ± 2.54^{b}	2.09 ± 0.17^{a}
Freeze drying	89.53 ± 1.40^{a}	-3.38 ± 0.83^{f}	29.97 ± 0.24^{a}	34.01 ± 1.60^{a}	30.16 ± 2.16	-83.61 ± 0.14^{c}	0.97 ± 0.03^{c}
Infrared drying	67.91 ± 1.43^{c}	4.60 ± 0.27^{bc}	26.24 ± 1.81^{bcd}	26.47 ± 0.14^{b}	26.64 ± 2.50	80.10 ± 2.88^{ab}	2.00 ± 0.04^{a}
Sun drying	74.55 ± 5.61^{b}	3.72 ± 0.18^{cd}	28.34 ± 2.54^{abc}	25.99 ± 1.06^{b}	28.58 ± 1.40	82.56 ± 4.37^{ab}	1.51 ± 0.03^{b}
Oven drying	76.57 ± 2.89^{b}	3.55 ± 0.01^{d}	28.79 ± 1.11^{ab}	26.40 ± 2.84^{b}	29.01 ± 3.17	83.01 ± 3.01^{ab}	1.09 ± 0.01^{c}

Note: The values in the table indicate mean ± standard deviation. Different letters in the same column show differences between sample groups according to the Duncan test ($p < 0.05$).
[1]Statistically not different.

TABLE 4 Moisture content (MC), water activity (WA), water holding capacity (WHC), and water solubility (WS) of bergamot peel powders.

Drying methods	MC* (%)	WA*	WHC (g/g)	WS (%)
Hot air drying	9.66 ± 0.07	0.3645 ± 0.02	7.90 ± 0.23^{ab}	61.27 ± 1.03^{a}
Microwave drying	9.95 ± 0.71	0.4256 ± 0.02	6.26 ± 0.08^{b}	52.58 ± 0.15^{b}
Infrared-assisted-microwave drying	9.76 ± 0.44	0.4077 ± 0.01	6.80 ± 0.13^{ab}	52.90 ± 2.60^{b}
Freeze drying	9.56 ± 0.13	0.2626 ± 0.08	8.09 ± 1.26^{a}	61.51 ± 4.26^{a}
Infrared drying	9.72 ± 0.42	0.3967 ± 0.15	7.11 ± 1.23^{ab}	57.10 ± 2.39^{ab}
Sun drying	9.71 ± 0.08	0.3890 ± 0.04	7.38 ± 0.38^{ab}	57.37 ± 3.04^{ab}
Oven drying	9.69 ± 0.30	0.3729 ± 0.03	7.81 ± 0.04^{ab}	58.22 ± 0.61^{ab}

Note: The values in the table indicate mean ± standard deviation. Different letters in the same column show differences between sample groups according to the Duncan test ($p < 0.05$).
*Statistically not different.

3.4 | Ascorbic acid, antioxidant activity, total phenolic, flavonoid, carotenoid, and 5-hydroxymethylfurfural content

Table 5 provides data on the ascorbic acid, antioxidant activity, total phenolic, flavonoid, carotenoid, and 5-HMF contents of bergamot peels subjected to various drying methods. As ascorbic acid is sensitive to heat, drying can significantly influence its content. The highest ascorbic acid content was observed in freeze-dried samples (445.33 mg/100 g), whereas the lowest was found in the sun-dried samples (89.58 mg/100 g). Freeze drying, known for its minimal thermal treatment and oxygen-free conditions, preserves ascorbic acid content more effectively than sun drying, where exposure to oxygen occurs (Boateng & Yang, 2021; Yuste et al., 2020). Despite its minimal thermal treatment, sun-drying leads to a significant reduction in ascorbic acid content. The decline in ascorbic acid content during microwave and infrared drying can be attributed to oxidative and thermal degradation, in alignment with previous studies. Furthermore, long drying times, high temperatures, or exposure to oxygen during drying have been reported to decrease ascorbic acid content in lemon peels (Tekgul & Baysal, 2018). The drying temperature has also been reported to affect the ascorbic acid content in orange peels (Bozkir et al., 2021).

Different drying methods exerted varying effects on the antioxidant activity and total phenolic, flavonoid, and carotenoid contents of bergamot peels compared to ascorbic acid content (Table 5). Radical scavenging activity is attributed to phenolics, vitamin C, carotenoids, and flavonoid compounds (Li et al., 2006). The highest values for antioxidant activity and total phenolic, flavonoid, and carotenoid contents were observed in the freeze-dried samples (176.84 mg TE, 199.22 mg GAE, 550.55 mg CE, and 54.12 mg BCE per 100 mg, respectively). Freeze-drying consistently emerges as the method that best preserves the antioxidant, phenolic, flavonoid, and carotenoid properties, as reported in various studies (Farahmandfar et al., 2020; Gao et al., 2012; Orphanides et al., 2013; Rafiq et al., 2019; Sogi et al., 2013; Tekgul & Baysal, 2018). Thermal processing methods like microwaves, infrared, and infrared-assisted microwave drying retained relatively high values (excluding 5-HMF) despite the intense thermal processing due to their shorter drying times. High microwave powers were associated with shorter drying times and higher phenolic content (Ghanem et al., 2012). The use of microwaves results in fragile tissues, facilitating cell wall breakdown and increasing antioxidants' extractability. Heat treatment-induced damage to cell membranes, cell walls, and tissues in citrus peels enhances the solubility of phenolic compounds and the conversion of most

TABLE 5 Ascorbic acid (AA), antioxidant activity as Trolox equivalent (AOA), total phenolic (TPC) as gallic acid equivalent, total flavonoid as catechin equivalent (TFC), total carotenoid (TCC) as ß-carotene equivalent, and 5-hydroxymethylfurfural (5-HMF) content of bergamot peel powders (mg/100 g).

Drying methods	AA	AOA	TPC	TFC	TCC	5-HMF
Hot air drying	420.66 ± 7.06^{b}	127.38 ± 4.47^{bc}	163.69 ± 7.44^{de}	300.34 ± 14.33^{e}	25.22 ± 2.14^{bcd}	9.74 ± 0.61^{d}
Microwave drying	176.09 ± 4.85^{f}	138.19 ± 6.12^{b}	171.94 ± 2.53^{cd}	330.83 ± 41.14^{c}	26.82 ± 3.32^{bc}	73.06 ± 4.26^{a}
Infrared-assisted-microwave drying	192.84 ± 8.03^{e}	170.38 ± 5.85^{a}	182.72 ± 3.49^{bc}	400.72 ± 2.63^{b}	27.02 ± 3.37^{bc}	53.91 ± 5.02^{b}
Freeze drying	445.33 ± 5.92^{a}	176.84 ± 9.31^{a}	199.22 ± 0.84^{a}	550.55 ± 4.04^{a}	54.12 ± 1.44^{a}	0.07 ± 0.04^{e}
Infrared drying	277.51 ± 9.80^{d}	173.86 ± 4.54^{a}	193.40 ± 7.24^{ab}	530.14 ± 6.19^{a}	30.22 ± 6.95^{b}	36.38 ± 1.61^{c}
Sun drying	89.58 ± 2.40^{g}	103.05 ± 10.22^{d}	149.32 ± 9.99^{f}	223.48 ± 4.72^{d}	18.12 ± 1.17^{d}	1.12 ± 0.08^{e}
Oven drying	341.95 ± 3.11^{c}	116.57 ± 2.76^{cd}	153.69 ± 1.26^{ef}	234.52 ± 5.47^{d}	21.62 ± 1.19^{cd}	4.47 ± 0.08^{de}

Note: The values in the table indicate mean ± standard deviation. Different letters in the same column show differences between sample groups according to the Duncan test ($p < 0.05$).

phenolic compounds from a bound form to a free form (Ko et al., 2020; Tekgul & Baysal, 2018). Changes in the structure of flavonoids during drying generate low molecular weight phenolic compounds that impact antioxidant activity, whereas melanoidin-like pigments formed during the Maillard reaction enhance antioxidant activity (Turkmen et al., 2006). Nonenzymatic interconversion between phenolic compounds and other molecules generates new compounds that react with the Folin–Ciocalteu reagent, yielding high values in total phenol determination (Que et al., 2008).

Hot air and oven-dried samples (9.74 and 4.47 mg/100 g, respectively) displayed relatively low contents (excluding 5-HMF), with oven-dried samples having the lowest content of 5-HMF. The extended drying times in these methods might contribute to structural damage in compounds, resulting in lower values. Previous studies have reported a decrease in phenolic content in peels with increased drying time (Farahmandfar et al., 2020; M'hiri et al., 2017). Loss of carotenoids in lengthy drying processes is attributed to nonenzymatic browning and pigment degradation (Ghanam et al., 2017).

Sun-dried samples had the lowest values (1.12 mg/100 g) (excluding 5-HMF), aligning with findings by Ozcan et al. (2021), which reported that prolonged sun-drying led to decreased flavonoid compounds. The extended drying duration in sun drying triggers more enzymatic reactions, potentially reducing total phenolic content (Xing et al., 2017). Although low drying temperatures may favor the preservation of heat-sensitive antioxidant compounds, longer drying times may enhance enzymatic degradation (Farahmandfar et al., 2020).

The influence of various drying methods on 5-HMF contents differed from the effects on other compounds. The lowest 5-HMF contents (almost negligible and 1.12 mg/100 g) were observed in freeze-dried and sun-dried samples, as these methods do not involve thermal processing. In contrast, the more intense thermal processes in the infrared, infrared-assisted microwave, and microwave drying led to higher 5-HMF contents (36.38, 53.91, and 73.06 mg/100 g, respectively). Generally, samples with low L^* value and high browning index had higher 5-HMF content (Table 3). Heat treatment-induced formation of 5-HMF in persimmons was associated with browning, with samples exhibiting the lowest lightness values having the highest 5-HMF content (Kayacan et al., 2020). In dried fruit products, 5-HMF content typically falls within the range of 55–13,500 mg/100 g (Abraham et al., 2011), with our results aligning with current data. It has been reported that HMF content increases with temperature in thermal processes (Vardin & Yilmaz, 2018). The impact of drying processes on 5-HMF formation varies widely in the literature, mainly due to differences in raw material properties (Michalska et al., 2016; Tontul & Topuz, 2017).

Statistically significant differences were noted among samples for ascorbic acid, total phenolic content, and 5-HMF results. However, regarding antioxidant activity, statistically insignificant differences were observed among the infrared-assisted microwave, freeze, and infrared drying methods. Similarly, statistically insignificant differences were found between the freeze and infrared, sun drying and oven drying, and hot-air and microwave drying methods. For the total carotenoid content results, statistically insignificant differences were observed between the microwave and infrared-assisted microwave drying methods. Similar results were identified in the case of total flavonoid content, indicating statistically insignificant differences between the mentioned drying methods. Although the obtained data can be categorized as high or low concerning these aspects, Section 4 will provide a comprehensive conclusion, considering the statistical differences.

3.5 | Determination of individual phenolics

The amounts of individual phenolic compounds determined in the aqueous extracts of bergamot peels dried by different methods are given in Table 6. The HPLC chromatograms of the samples are shown in Figure 1.

Bergamot peel has been found to contain a variety of phenolics that are not present in other citrus peel residues, such as citrus flavanone rutinosides and neohesperidose derived from naringenin, eriodictyol, and hesperetin, as reported in the literature (Mandalari et al., 2006). Furthermore, Rafiq et al. (2019) have confirmed the existence of numerous phenolic compounds in citrus peels, indicating that they are present in high concentrations.

Ozcan et al. (2021) dried lemon and orange peels using different methods, identified the amounts of 16 different phenolic compounds, and reported that their contents were significantly affected by the drying methods. In our study, 12 phenolic compounds were identified in bergamot peels that were dried using HPLC.

The compounds gallic acid, *p*-coumaric acid, rutin, naringin, hesperidin, eriocitrin, limonin, neoeriocitrin, narirutin, neohesperidin, naringenin, and hesperetin were found in the freeze-dried, infrared-dried, infrared-assisted microwave-dried, microwave-dried, hot air-dried, oven-dried, and sun-dried samples, respectively, in descending order of concentration.

All identified compounds were present at their maximum levels in the freeze-dried samples because the process occurs under low temperatures and a vacuum.

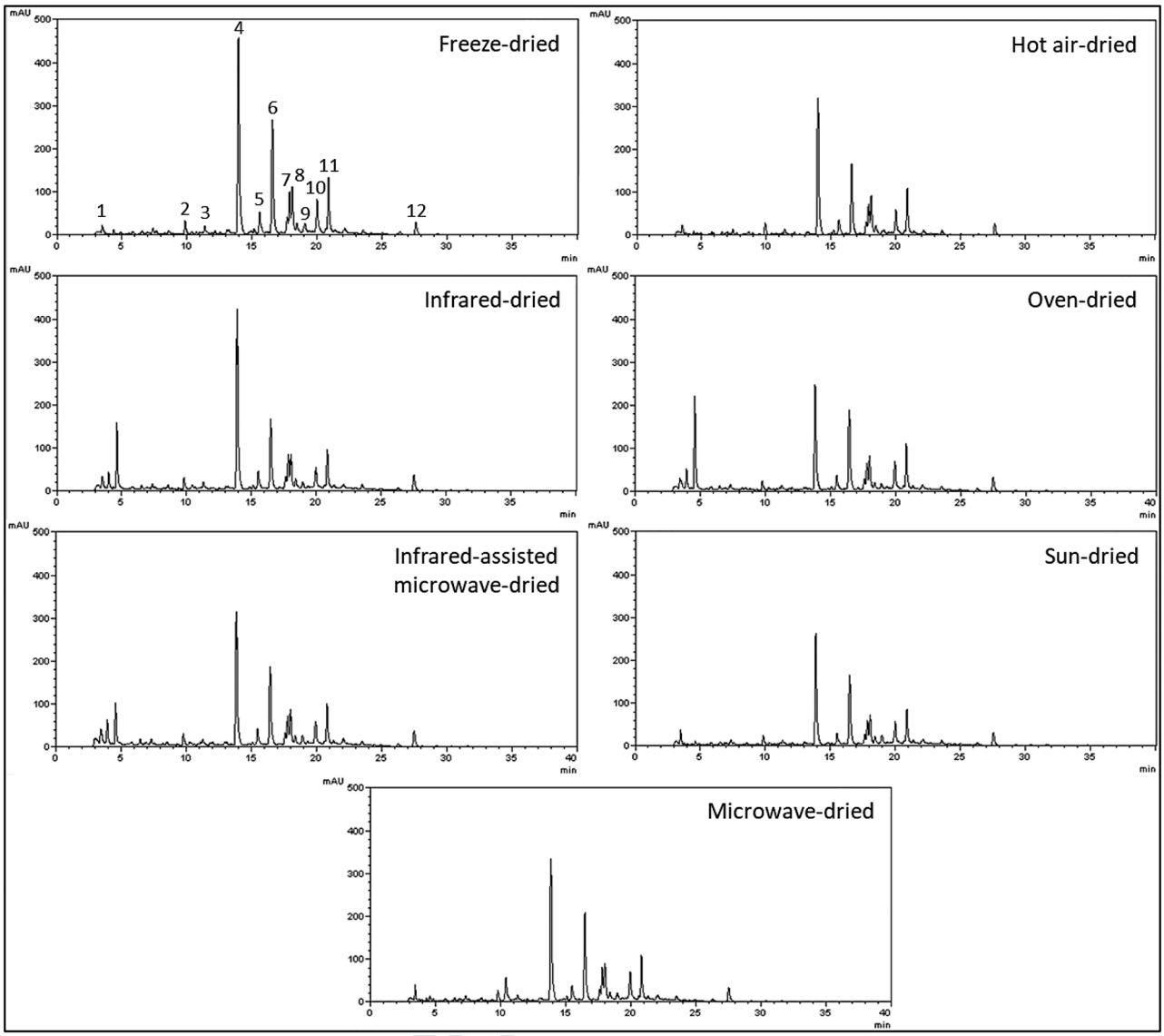

FIGURE 1 HPLC chromatograms of phenolic extracts from dried bergamot peels using different drying methods. Peak identifies as follows: gallic acid (1), *p*-coumaric acid (2), rutin (3), naringin (4), hesperidin (5), eriocitrin (6), limonin (7), neoeriocitrin (8), narirutin (9), neohesperidin (10), naringenin (11), and hesperetin (12).

The samples obtained by infrared drying, an intense but short-term thermal process, enabled the determination of high concentrations of phenolics. Similarly, Ozcan et al. (2021) reported that the phenolic compounds identified in infrared-dried lemon and orange peels were preserved at a higher level compared to oven and microwave drying. Xu et al. (2007) showed that the free fractions of phenolics increased after heat treatment, and Sun et al. (2015) reported that heat treatment could positively affect the number of phenolics.

The amounts of phenolic compounds identified in the infrared-assisted microwave and microwave-dried samples were similar, with no significant differences observed compared to the hot air and oven drying methods. Microwave is a high-energy but short-term thermal process so that the contents may be better preserved. The relatively low concentrations detected in the hot air-dried samples may be explained by the moderate temperatures (around 60°C) promoting the degradation of the phenolic compounds. On the other hand, as oxidation reactions take place more in the sun-drying process at lower temperatures but longer drying times, minimum values were determined in sun-dried samples.

For each drying method, the highest concentrations were determined for naringin (878.10–1568.70 mg/100 g), eriocitrin (575.80–910.60 mg/100 g), neoeriocitrin (41.40–403.10 mg/100 g), neohesperidin (158.20–260.30 mg/100 g), and naringenin (236.80–346.10 mg/100 g). In all drying methods, naringin, eriocitrin, neoeriocitrin, neohesperidin, and naringenin accounted for 82.05%–87.78% of

TABLE 6 Individual phenolics (mg/100 g) of bergamot peel powder on different drying methods.

Compound	Freeze drying	Infrared drying	Infrared-assisted microwave drying	Microwave drying	Hot air drying	Oven drying	Sun drying
Gallic acid	158.9 ± 1.8^{a}	127.4 ± 6.5^{b}	107.4 ± 0.2^{c}	87.5 ± 1.8^{d}	56.1 ± 8.5^{e}	47.2 ± 5.8^{f}	24.7 ± 0.2^{g}
p-Coumaric acid	120.7 ± 1.0^{a}	107.6 ± 1.4^{b}	88.1 ± 0.5^{c}	86.3 ± 6.4^{d}	74.6 ± 1.8^{e}	65.3 ± 4.8^{f}	6.9 ± 1.1^{g}
Rutin	44.5 ± 1.4^{a}	39.7 ± 2.3^{b}	38.4 ± 0.7^{c}	25.4 ± 5.4^{d}	20.3 ± 8.4^{e}	18.1 ± 1.4^{f}	9.1 ± 1.5^{g}
Naringin	1568.7 ± 1.8^{a}	1412.7 ± 6.5^{b}	1240.3 ± 1.2^{c}	1123.9 ± 0.3^{d}	1078.3 ± 6.7^{e}	945.2 ± 5.7^{f}	878.1 ± 5.1^{g}
Hesperidin	176.6 ± 2.0^{a}	145.3 ± 4.1^{b}	139.7 ± 4.6^{c}	131.1 ± 1.7^{d}	120.5 ± 5.2^{e}	116.1 ± 8.4^{f}	109.1 ± 4.1^{g}
Eriocitrin	910.6 ± 7.8^{a}	754.4 ± 2.0^{b}	721.4 ± 3.7^{c}	696.7 ± 1.1^{d}	647.4 ± 1.8^{e}	614.2 ± 5.6^{f}	575.8 ± 7.7^{g}
Limonin	143.3 ± 1.4^{a}	103.1 ± 5.4^{b}	91.7 ± 3.8^{c}	74.5 ± 0.8^{d}	37.4 ± 5.8^{e}	24.7 ± 1.4^{f}	10.3 ± 8.5^{g}
Neoeriocitrin	403.1 ± 2.4^{a}	374.1 ± 0.4^{b}	245.8 ± 4.1^{c}	191.0 ± 0.5^{d}	177.9 ± 7.7^{e}	100.5 ± 5.7^{f}	41.4 ± 4.1^{g}
Narirutin	98.9 ± 6.5^{a}	59.6 ± 0.9^{b}	44.8 ± 0.7^{c}	43.1 ± 0.4^{d}	25.4 ± 8.4^{e}	8.9 ± 2.4^{f}	5.6 ± 7.4^{g}
Neohesperidin	260.3 ± 8.4^{a}	247.4 ± 0.4^{b}	225.1 ± 0.8^{c}	210.5 ± 0.9^{d}	197.6 ± 1.5^{e}	170.4 ± 8.1^{f}	158.2 ± 5.8^{g}
Naringenin	346.1 ± 0.5^{a}	321.1 ± 5.7^{b}	301.7 ± 1.4^{c}	293.4 ± 7.4^{d}	288.2 ± 0.9^{e}	268.7 ± 6.2^{f}	236.8 ± 1.4^{g}
Hesperetin	20.3 ± 5.4^{a}	18.9 ± 3.6^{b}	15.9 ± 2.5^{c}	14.8 ± 5.1^{d}	13.5 ± 0.4^{e}	12.0 ± 0.2^{f}	6.1 ± 0.6^{g}

Note: The values in the table indicate mean ± standard deviation. Different letters in the same row show differences between sample groups according to the Duncan test ($p < 0.05$).

the total phenolics. Similarly, Mandalari et al. (2007) determined the significant phenolics in bergamot peels as naringin, neoeriocitrin, neohesperidin, and naringenin.

The individual phenolic contents belonging to different drying methods were also found to be statistically significant. Considering the results obtained and the available data in the literature, it was seen that the phenolic composition of citrus peels dried with different methods varies according to the type of raw material first and then the drying process parameters (Ozcan et al., 2021).

3.6 | Physical properties of dried bergamot peels

Table 7 presents the physical properties of bergamot peels dried using different methods. The bulk density of powders obtained by drying indicates the amount of product that can be packed in a specific volume, and the powder products' shape, size, and moisture content affect the bulk density. It was observed that different drying methods did not significantly affect bulk and tapped density. The freeze-dried samples had the lowest bulk density (0.35 g/mL), as reported by Tekgul and Baysal (2018) and Farahmandfar et al. (2020). Freeze-drying is a drying method that preserves the structure of samples with minimum shrinkage, resulting in products characterized by low bulk density, high porosity, and improved rehydration properties, as Xing et al. (2017) reported. Infrared-assisted microwave processing also resulted in a low bulk density (0.42 g/mL) due to the puffing phenomenon, which reduces the bulk density of dried products. The highest bulk density (0.51 g/mL) and the lowest porosity (42.74%) were determined in hot air-dried samples, as reported by previous studies (Farahmandfar et al., 2020; Horuz & Maskan, 2015), which could be attributed to the collapse of the plant's cell walls and substantial shrinkage during oven drying.

Tapped density is obtained by tapping or vibrating the powder until the minimum and constant volumes are reached. It depends on the porosity and shape of the particles, which determine flowability and packing density. The microwave, infrared, and infrared-assisted microwave processes resulted in relatively lower tapped densities (0.65–0.67 g/mL).

The Hausner ratio or compressibility index is the difference between apparent and tapped densities. Granules with high flowability and low intergranular friction are expected to have fewer packing flaws. Medium fluency (27.27%–33.33%) was determined in infrared, hot air, oven, and microwave-dried samples, and poor fluency (36.36%–50%) in the infrared-assisted microwave, freeze, and sun-

TABLE 7 Physical properties of dried bergamot peel powder.

Property	Hot air drying	Microwave drying	Infrared-assisted microwave drying	Freeze drying	Infrared drying	Sun drying	Oven drying
Bulk density (g/mL)	0.51 ± 0.03^{a}	0.45 ± 0.04^{ab}	0.42 ± 0.01^{ab}	0.35 ± 0.03^{b}	0.47 ± 0.07^{ab}	0.48 ± 0.06^{ab}	0.50 ± 0.11^{ab}
Tapped density* (g/mL)	0.72 ± 0.01^{a}	0.67 ± 0.03^{a}	0.66 ± 0.03^{a}	0.71 ± 0.18^{a}	0.65 ± 0.07^{a}	0.79 ± 0.03^{a}	0.71 ± 0.13^{a}
Particle density (g/mL)	1.26 ± 0.03^{b}	5.07 ± 0.04^{a}	5.09 ± 0.06^{a}	5.06 ± 0.07^{a}	1.01 ± 0.09^{c}	5.09 ± 0.04^{a}	5.04 ± 0.02^{a}
Porosity (%)	42.74 ± 1.19^{b}	86.72 ± 0.57^{a}	87.01 ± 0.27^{a}	86.06 ± 0.25^{a}	35.78 ± 3.96^{c}	84.39 ± 1.03^{a}	85.85 ± 2.40^{a}
Flowability (%)	30.00 ± 1.41^{de}	33.33 ± 2.66^{cd}	36.36 ± 2.52^{bc}	50.00 ± 1.77^{a}	27.27 ± 0.35^{e}	40.00 ± 3.04^{b}	30.00 ± 0.21^{de}
Cohesiveness	1.43 ± 0.06^{b}	1.50 ± 0.34^{b}	1.57 ± 0.10^{ab}	2.00 ± 0.37^{a}	1.37 ± 0.01^{b}	1.67 ± 0.09^{ab}	1.43 ± 0.04^{b}
Hygroscopicity (%)	15.07 ± 0.15^{abc}	14.39 ± 0.42^{bc}	13.94 ± 0.14^{bc}	18.51 ± 0.90^{a}	12.20 ± 2.76^{bc}	11.84 ± 1.36^{c}	15.88 ± 2.31^{ab}
Caking degree (%)	14.53 ± 0.17^{ab}	14.01 ± 0.75^{bc}	11.24 ± 0.08 cd	17.27 ± 2.63^{a}	10.96 ± 1.58^{d}	10.28 ± 0.14^{d}	16.10 ± 0.79^{ab}

Note: The values in the table indicate mean ± standard deviation. Different letters in the same row show differences between sample groups according to the Duncan test ($p < 0.05$).
*Statistically not different.

dried samples. The powders showed a high stickiness (1.37–2.00) in general.

Hygroscopicity refers to the ability of a powder to absorb water, and if a powder absorbs too much moisture, it may clump together during storage. The hygroscopicity was highest in freeze-dried (18.51%) and lowest in sun-dried (11.84%) samples. Freeze-dried products have high hygroscopicity due to their porous structure and low moisture content, whereas microwave-dried products have lower hygroscopicity due to their compact and skeletal structure. The freeze-dried samples also had the highest degree of caking (17.27%) due to their high hygroscopicity and low bulk density, but the values indicated slight caking. However, no statistically significant difference of significant magnitude was detected among these values. These results are consistent with previous literature (Gopinathan et al., 2020; Shuen et al., 2021).

Although specific drying methods may not show statistically significant differences within themselves based on the characterization analysis results, it is essential to interpret the lowest and highest values in this context.

4 | DISCUSSION

This study demonstrates the successful conversion of bergamot peel into powder using various drying methods, including hot air, microwave, infrared-assisted microwave, freeze-drying, infrared, solar, and oven drying. The drying method significantly influenced the powder's physical, chemical, and functional properties.

Energy consumption is an essential aspect to consider when comparing these drying methods. Among the methods evaluated, freeze-drying required the most energy (55.20 kW h), followed by hot air drying (17 kW h), infrared drying (7.95 kW h), oven drying (4.50 kW h), and infrared-assisted or non-assisted microwave drying (0.15 and 0.10 kW h). It is worth noting that microwave drying, despite its relatively short processing time, proved to be the most efficient method in terms of energy consumption, excluding sun drying.

The choice of drying method significantly affected the physical properties of the dried bergamot peel powders. All methods yielded powders with moisture content below 10%, and no significant differences were observed in moisture content and water activity, thanks to the adjustment of drying process parameters. The final moisture content ranged from 9.56% to 9.95%, and water activity values fell between 0.2626 and 0.4256, making the powders suitable for dried fruit products. Notably, freeze-dried samples exhibited the highest water holding capacity (8.09 g/g) and solubility (61.51%), making them an ideal choice for processing fruit byproducts.

Regarding the powder's physical properties, the bulk density, porosity, fluidity, and stickiness varied among the drying methods. Freeze-dried samples had the lowest bulk density (0.35 g/mL), highest flowability (50%), high porosity (86.06%), and high stickiness. Color properties also differed based on the drying method, with freeze-dried and hot air-dried samples having lighter and more yellow colors. In contrast, microwave-dried samples exhibited a higher browning index due to the intense thermal process. Sun-dried samples had the lowest color difference compared to the fresh sample, as they underwent no thermal processing.

Chemical properties of the powders were significantly affected by the drying method. Notably, microwave-dried samples had the highest 5-HMF content (73.06 mg/100 g) due to the intense thermal process, whereas freeze-dried and sun-dried samples had the lowest 5-HMF content (0.07 and 1.12 mg/100 g, respectively). Freeze-dried and infrared-dried samples contained higher levels of ascorbic acid, antioxidant activity, total phenolic, flavonoid, and carotenoid contents. Furthermore, the infrared drying method resulted in powders with high antioxidant activity and total phenolic, flavonoid, and carotenoid contents.

The choice of drying method should align with the desired properties of the final product. Freeze drying is advantageous in preserving high levels of bioactive compounds, color, solubility, and water-holding capacity, but its cost and energy intensity may limit large-scale production. Infrared or infrared-assisted drying methods are promising for recovering phenolics from bergamot waste due to their ability to maintain bioactive compounds, physicochemical properties, shorter processing times, and lower energy consumption than other methods. The selection of a drying method should consider factors such as product characteristics, available resources, and energy costs.

5 | CONCLUSION

This study has highlighted the significance of bergamot peel waste, a byproduct of essential oil extraction, which harbors valuable phenolic compounds that can be effectively reclaimed through diverse drying methods. The drying process not only diminishes moisture content but also retards the deterioration of active constituents within the peel, transforming it into a stable powdered state. The selection of an appropriate drying method should consider the distinctive impacts on the physicochemical attributes of bergamot peel powders tailored to specific applications. Although freeze-drying showcased optimal outcomes, its notable energy consumption and hygroscopic nature prompted consideration of infrared or infrared-assisted drying as viable alternatives.

The outcomes of this investigation convincingly establish the potential of dried bergamot peels as a valuable asset across a spectrum of industries. The inherent phenolics within these peels, endowed with antioxidant, anti-inflammatory, and antimicrobial properties, offer multifaceted utility in cosmetics, nutraceuticals, functional foods, and natural food preservatives, aligning closely with the overarching findings of this study. Ultimately, the successful conversion of bergamot peels into a powdered format unveils a promising avenue for harnessing this waste stream's latent value through our research's specific outcomes.

AUTHOR CONTRIBUTIONS

Bahar Demircan: Conceptualization; methodology; software; data curation; investigation; validation; formal analysis; supervision; visualization; project administration; resources; writing—original draft; writing—review and editing. **Yakup Sedat Velioglu**: Conceptualization; investigation; funding acquisition; writing—original draft; writing—review and editing; visualization; validation; methodology; software; formal analysis; project administration; resources; supervision; data curation. **Angelo Maria Giuffrè**: Conceptualization; methodology; software; data curation; supervision; resources; project administration; formal analysis; validation; visualization; investigation; writing—original draft; writing—review and editing.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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