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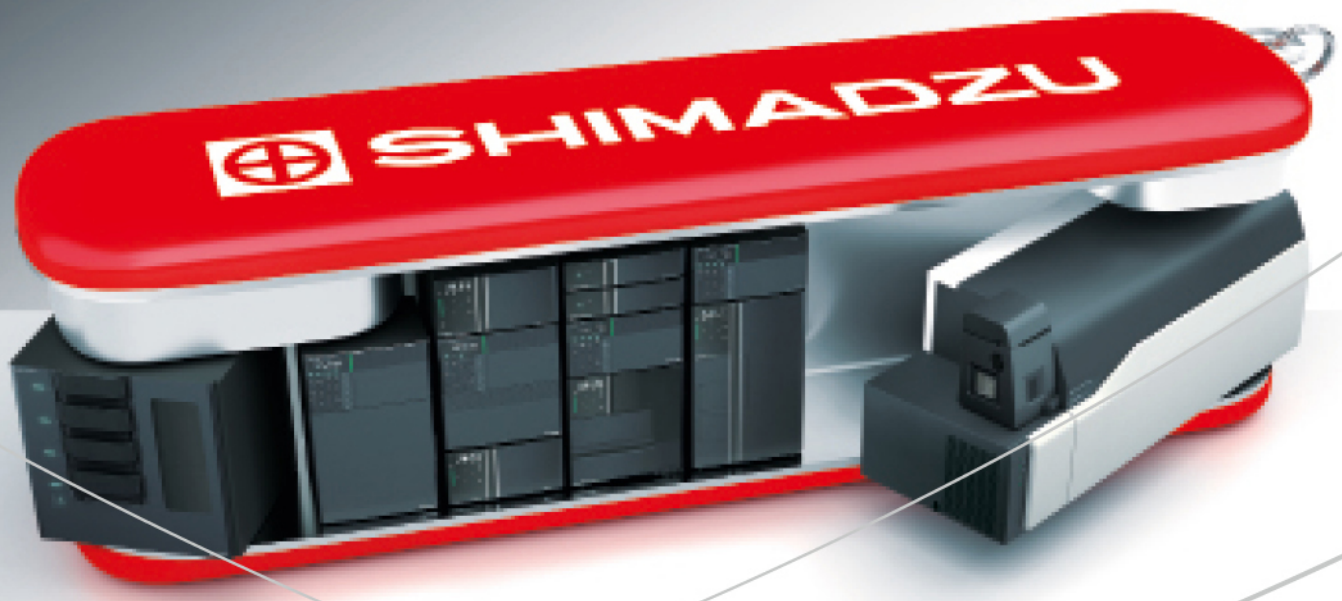

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ORIGINAL ARTICLE

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Bergamot juice powder with high bioactive properties: Spray-drying for the preservation of antioxidant activity and ultrasound-assisted extraction for enhanced phenolic compound extraction

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Abstract: The spray-drying process yielded functional bergamot juice powder with high antioxidant activity, phenolic content, and vitamin C content. Optimal drying conditions were determined as 10% maltodextrin concentration, 146.02°C inlet temperature, and 39.99% pump rate, preserving powder's bioactive properties. Under these drying conditions, bergamot juice powder exhibited an antioxidant activity of 62.2% DPPH scavenging activity, a total phenolic content of 3862.1 ppm, and a vitamin C content of 1385.9 ppm. The bergamot juice powder, with a water activity of 0.2, bulk density of 0.4 g/mL, tapped density of 0.5 g/mL, porosity of 89.6%, hygroscopicity of 8.6%, and cohesiveness of 37.2%, is highly suitable for further processing. High-pressure liquid chromatography analysis revealed the presence of major phenolic compounds in both fresh bergamot juice and spray-dried powder, although their concentrations were lower in the powder form. The major phenolics identified in the fruit juice were naringin (197.5 ppm), eriocitrin (105.9 ppm), neoeriocitrin (53.4 ppm), neohesperidin (68.8 ppm), and naringenin (119.8 ppm). However, in the powder form, the bitterness-associated compounds, naringin and neohesperidin, exhibited a significant reduction of 85.0% and 90.3%, respectively. Compared to dimethyl sulfoxide (48.4%), ethanol (37.9%), and distilled water (17.3%), ultrasound-assisted extraction with acetone solvent demonstrated the highest efficiency (61.7%) in obtaining phenolic compounds from bergamot juice powder. In conclusion, spray-drying is an effective method for obtaining functional bergamot juice powder, and ultrasound-assisted extraction can further enhance phenolic compound extraction efficiency. These findings have potential applications in the food, cosmetics, and pharmaceutical industries, with opportunities for further research in functional foods or nutraceuticals.

KEYWORDS
bergamot juice powder, bioactive properties, spray-drying, phenolics, powder quality

Practical Application: Spray-drying yields functional bergamot juice powder with high bioactive properties. Optimal drying conditions can be applied in industrial settings. Ultrasound-assisted extraction enhances phenolic compound extraction efficiency. Potential applications in food, cosmetics, and pharmaceutical industries.

1 | INTRODUCTION

Bergamot (*Citrus bergamia*) is a natural hybrid fruit obtained from sour orange and citron (Li et al., 2010; Nicolosi et al., 2000) and quite different from other citrus fruits with its rich content and an essential and wide variety of flavonoids (Di Donna et al., 2009, 2020; Giuffrè, 2019; Giuffrè & Nobile, 2020). These antioxidants are contained in the flavedo, albedo, and pulp (Gullo et al., 2020). Studies by other authors suggested sour orange as the most likely maternal parent and citron as a presumable paternal parent (Li et al., 2010). Bergamot is mainly produced in a coastal stripe of the Reggio Calabria province, South Italy, and in smaller quantities in the South of Turkey and some other Countries. Bergamot is the common name of the *C. bergamia* fruit and belongs to the Citrus genus Rutaceae family. It is a fruit that stands out with its unique appearance and strong aroma (Conidi et al., 2011).

Bergamot fruit has a very characteristic and intense odor, rich in terpenes, esters, and alcohol. Although bergamot has been known for several centuries, there are few studies on this fruit in the literature. Most of it focuses on essential oil, whereas research on bergamot juice is generally lacking. Bergamot is mainly used for the extraction of essential oil from flavedo (Gioffrè et al., 2020; Schipilliti et al., 2011; Verzera et al., 2000, 2003), which has antibacterial, antiseptic, neuroprotective, and anticancer effects and is widely used in the pharmaceutical, cosmetic, and food industries (Di Donna et al., 2009; Impellizzeri et al., 2015; Marotta et al., 2016; Navarra et al., 2015; Perna et al., 2019).

On the contrary, the juices obtained from the albedo and endocarp, the white tissue between the surface and the pulp, do not have significant industrial applications. Its disposal poses a severe problem due to its high economic costs and environmental pollution (Di Donna et al., 2011; Sicari & Poiana, 2017). Bergamot juice has a very bitter taste. It is difficult to drink but can be diluted with water (1/3) or added to other juice, such as apple juice; this beverage is already marketed in Italy. Nowadays, most of the bergamot juice is treated and discarded, but this treatment requires a specific expense (Conidi et al., 2011; Impellizzeri et al., 2015; Mollace et al., 2011). Although most of the bergamot juice is separated as a waste, it contains many phenolic compounds that positively affect human health. Bergamot juice has a high content of phenolic and other antioxidants and aromatic compounds. It is a natural source of flavonoids in terms of polyphenols (naringin, hesperidin, neohesperidin, and neoeriocitrin), which primarily protect human health (Conidi et al., 2011; Giuffrè, 2019; Pernice et al., 2009). Anticancer, antimicrobial, antiviral, and anti-inflammatory effects of flavonoids found in bergamot juice have been investigated by many researchers in recent years (Filocamo et al., 2015; Navarra et al., 2014; Russo et al., 2016), and thus, an increasing interest has emerged in the marketability of bergamot juice (Cautela et al., 2008, 2019; Gattuso et al., 2006; Giuffrè, 2019; Qiu et al., 2018; Russo et al., 2016).

In fact, because of the high flavonoid content, bergamot fruit was studied to use its extract to fortify biscuits (Laganà et al., 2022) and beer (Muscolo et al., 2022). In this context, the recovery of polyphenols from fruit juice can serve both sustainability and the production of a rich natural additive. Due to these crucial effects, natural phenols of fruit juice have the potential to be used as active substances in the pharmaceutical industry and as antioxidant and antimicrobial compounds in the food industry. In this context, the recovery of polyphenols from fruit juice can facilitate sustainability and the production of a valuable natural product for consumer demand. The production of fruit juice powders with high nutritional value and regular microstructure can meet this requirement in various industries.

Due to the difficulties of using and storing fruit juice in fresh form, using powder with high nutritional value and regular microstructure is much more advantageous (Fazaeli et al., 2012; Turchiuli et al., 2011). The demand for fruit and vegetable juice powders has increased significantly due to the multiple benefits of applying these products in various food formulations. Packaging, storage, and transportation expenses are less in juice powders with

high stability. Various drying techniques are available for powdering fruit juices on an industrial scale. Freeze drying is considered the most efficient in preserving nutrients in powder products. However, its application on an industrial scale could be more robust due to low efficiency, high instrumentation expenditures, and high energy consumption. Spray-drying is the most successful and economical technique to convert liquid foods into powder form, maintaining quality through rapid dehydration. Heat-sensitive and heat-resistant feeds can be spray-dried (Phisut, 2012). The choice of operating parameters is critical to obtain a high-quality product during spray-drying. Optimizing the parameters in the spray-drying process must be done because each parameter significantly affects the powder properties. Carrier type and concentration, feed flow rate, and inlet air temperature are the most critical parameters in this process. These parameter ranges vary considerably in the literature (Shishir & Chen, 2017). For this reason, drying conditions must be determined according to the raw material to obtain superior quality in the powder product.

The qualitative and quantitative extraction of phenolics as bioactive compounds from fruit juice powders obtained through suitable process parameters primarily depends on solvent selection. Extraction is the primary step in plant research that significantly impacts the final result. The primary purpose of all extraction methods is to separate the soluble phytochemicals from the insoluble part of the plant matrix (Suleria & Barrow, 2019; Tomsone et al., 2012). Various methods carry out the extraction of bioactive compounds in plant materials. In the last 50 years, more environment-friendly nontraditional methods have been developed due to reduced use of synthetic and organic chemicals, reduced processing time, and better yield and extract quality. Some of the most promising techniques are ultrasound-assisted extraction, enzyme-assisted extraction, microwave-assisted extraction, pulsed electric field-assisted extraction, supercritical fluid extraction, and pressurized fluid extraction. Some of these techniques are considered "green techniques" because they meet the standards set by the US Environmental Protection Agency. Among them, ultrasound-assisted extraction is an efficient and effective extraction technique for bioactive compound extraction from plants (Azmir et al., 2013). The efficiency of any extraction method mainly depends on the choice of solvent. The polarity of the targeted compound is the most critical factor for solvent selection. The molecular affinity between solvent and solute, mass transfer, use of cosolvent, environmental safety, toxicity values, and financial feasibility should also be considered in selecting a solvent for bioactive compound extraction. Different solvent systems are used for the extraction of polyphenols from plant material, and mixtures of acidified methanol, ethanol, acetone, ethyl acetate, and solvents with water have been widely reported in the literature (Annegowda et al., 2012; Haminiuk et al., 2011, 2014; Pushp et al., 2013; Ramful et al., 2011). The properties of extraction solvents significantly affect the total phenolic content (~25% variation) and antioxidant capacity (~30% variation) measured in fruits and vegetables (Tomsone et al., 2012).

As a result of our research, no studies were found in the literature on the optimization of spray-drying of bergamot juice and the characterization of bergamot juice powder obtained by this method. In this study, bergamot juice was first characterized, and then spray-drying conditions were optimized for antioxidant activity, total phenolic content, vitamin C content, and yield. The physicochemical properties and phenolic component profile of bergamot juice powder produced using optimum drying parameters were determined. Thus, it aims to determine whether fresh fruit juice's chemical contents are preserved in powder form. Moreover, the literature has yet to investigate the effect of different solvent systems on preparing extracts from bergamot juice powder. Therefore, this study aimed to pulverize bergamot juice in a spray-dryer and to determine the effects of using different solvents in extracts prepared from this powder on antioxidant activity, total phenolic substance, vitamin C, and phenolic component composition.

2 | MATERIALS AND METHODS

2.1 | Materials and reagents

Bergamot fruit (*C. bergamia* Risso et Poiteau) was procured from Tekirova–Antalya/Turkey (36°30′6″N–30°31′38″E) in February 2022. Trees were Italian-originated Castagnaro variety. The ripe fruit diameter was about 6–8 cm. Experienced farmers cultivated the trees and grew in moderately sunny and lightly shaded places. All trees were about 3–5 m tall and 40–50 years old. During the growth phase, citrus-specific organic fertilizers were added regularly, and trees were rarely watered. A slightly acidic soil having a pH of 5.5–6.5 was well drained.

Approximately 10 kg of fruit, separated from the stems and leaves, were washed, cut in half, and juiced in a laboratory fruit squeezer (Moulinex, FP519GB1, Ecully, France). The juice remaining in the pulp separated at this stage was recovered by centrifugation (Hettich Zentrifugen, Universal 320R, Tuttlingen, Germany). Juice (approximately 4.5 kg) was filtered through a strainer and stored in amber glass bottles at −20°C until use.

Maltodextrin (NutriDex-18) with 18–20 dextrose equivalents (DE) and ~140°C glass transition temperature used in the research were kindly donated by Durukan Confectionery, Ankara/Turkey. BUCHI B-290 Mini Spray Dryer (BUCHI Labortechnik AG, Flawil, Swtizerland)

TABLE 1 Design of experiment and results of dependent variables.

Run	Maltodextrin concentration (% w/w)	Inlet air temperature (°C)	Pump rate (%)	Process yield (%)	Antioxidant activity (inhibition%)	Total phenolic content (ppm GAE)	Vitamin C content (ppm AA)
1	20	180	40	83.81	47.23	2187.09	859.26
2	10	150	10	90.44	51.83	3587.61	1259.26
3	30	180	25	86.71	37.35	2316.29	740.74
4	10	120	25	87.96	56.36	3364.61	1309.89
5	20	150	25	89.31	45.42	2669.44	1007.65
6	30	120	25	81.4	44.33	1865.45	814.81
7	20	150	25	89.35	45.60	2742.73	1007.41
8	10	150	40	86.33	62.54	3710.91	1407.41
9	20	120	40	72.45	48.12	2635.77	888.89
10	20	180	10	89.58	42.41	2972.41	837.04
11	20	150	25	89.13	45.32	2668.32	950.21
12	10	180	25	93.69	53.86	2351.32	1296.29
13	30	150	10	91.16	46.01	3862.04	830.25
14	20	120	10	89.64	50.27	3049.20	962.96
15	30	150	40	72.95	38.63	2201.21	681.48

with 40 kg/h drying gas flow and 1 L H_2O/h evaporation capacity.

2,2-Diphenyl-1-picrylhydrazyl (DPPH, CAS No. 1898-66-4), gallic acid (CAS No. 149-91-7), L-ascorbic acid (CAS No. 50-81-7), and all high-pressure liquid chromatography (HPLC) standards (*p*-coumaric acid, CAS No 501-98-4; rutin hydrate, CAS No. 207671-50-9; naringin, CAS No. 10236-47-2; hesperidin, CAS No. 520-26-3; eriocitrin, CAS No. 13463-28-0; limonin, CAS No. 1180-71-8; neoeriocitrin, CAS No. 13241-32-2; narirutin, CAS No. 14259-46-2; neohesperidin, CAS No. 13241-33-3; naringenin, CAS No. 67604-48-2; and hesperetin, CAS No. 69097-99-0) were purchased from Sigma-Aldrich (St. Louis, MO, USA); all other analytical or HPLC grade chemicals were purchased from Sigma-Aldrich, Merck (Rahway, NJ, USA), and Carlo Erba (Emmindingen, Germany). Deionized water was prepared by Milli-Q system (Millipore, Burlington, MA, USA). The following steps were performed in three replications and three parallels.

2.2 | Experimental design of the spray-drying process

According to the Box–Behnken design, 15 experiments at 3-factor and 3-level with 3 center points were conducted. The experimental design combinations are shown in Table 1.

The experimental study order was completely randomized. In the experimental design, maltodextrin concentration (10%–30% w/w), inlet temperature (120–180°C), and pump rate (10%–40%) were independent variables; process yield, antioxidant activity, total phenolic content, and vitamin C content were dependent variables. The constant process parameters in the spray-dryer were used as 100% aspirator rate (35 m^3/h) and 40 mm airflow volume (667 L/h). The response variables as a function of process variables were expressed in the second-order polynomial in the following equation:

$$Y_k = \beta_{k0} + \sum_{i=1}^{n} \beta_{ki} x_i + \sum_{i=1}^{n} \beta_{kii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{kij} x_i x_j \quad (1)$$

where Y_k is the dependent variable, Y_1 is the process yield (%), Y_2 is the antioxidant activity (DPPH scavenging activity%), Y_3 is the total phenolic content (ppm gallic acid equivalent), Y_4 is the vitamin C content (ppm ascorbic acid); x_1 is the concentration of maltodextrin, x_2 is the inlet air temperature, x_3 is the pump rate; β_{k0} was the value of the fitted response at the center point of the design, and $\beta_{ki,kii,kij}$ are the regression coefficients, respectively.

Statistical significance was determined by the analysis of variance (ANOVA) at a 95% confidence level. The adequacy of the model was checked with R^2 values. The desired targets were (Y_1 is within range and all other responses max) selected for each variable and response.

2.3 | Preparing the feed for the spray-drying process

Maltodextrin was added to 50 mL of freshly squeezed fruit juice at the concentration determined in the

experimental design (Table 1) and mixed for 5 min at 500 rpm in a magnetic stirrer (Heidolph, MR Hei-Standard, Schwabach, Germany). The prepared maltodextrin feed mixture was diluted with water to the original soluble dry matter of the fruit juice (%7.93) and kept at +4°C for one night to hydrate the maltodextrin in the feed. The feed was brought to room temperature and used in the spray-dryer.

2.4 | Analysis of powders

The following analyses were performed in 3 replications-3 parallel to 15 different bergamot juice powders obtained with the experimental design in Table 1. The same analyses were performed on the fresh bergamot juice, and the protection levels of the chemical parameters determined in the final product were revealed.

2.4.1 | Process yield

The efficiency of the spray-drying process was calculated considering the total solids content of the maltodextrin-containing feed and the final dry powder weight (Saikia et al., 2015).

2.4.2 | Antioxidant activity and total phenolic content of powders

An amount of 1 g of the powder was dissolved in distilled water (DW) in an amount equal to the initial soluble dry matter of the fruit juice (7.93%). After vortexing (Heidolph, D-91126), it was used as a powder extract to analyze antioxidant activity and total phenolic content (Moo-Huchin et al., 2014).

For antioxidant activity, 100 μL of powder extract was added to 3.9 mL of DPPH (6×10^{-5} M) solution and vortexed vigorously. After being kept in the dark for 30 min, the absorbance of the sample was read at 515 nm with a UV–Vis spectrophotometer (Shimadzu, UV-1601, Kyoto, Japan), and the results were determined as the DPPH radical scavenging activity% (Mishra et al., 2014).

For the total phenolic content, 60 μL of powder extract, 300 μL of Folin–Ciocalteu reagent, and 750 μL of 20% (w/v) Na_2CO_3 solution were added to 4.75 mL of water. After incubation (Nuve, EN 400, Ankara, Turkey) of the vortexed solution at 40°C for 30 min, the absorbance was read at 765 nm with a UV–Vis spectrophotometer. Results are expressed as mg gallic acid equivalent (GAE)/kg using a gallic acid calibration curve (Mishra et al., 2014).

2.4.3 | Vitamin C content of powders

An amount of 1 g of the powder was completely dissolved in 6% (w/v) metaphosphoric acid solution in an amount equal to the initial soluble dry matter of the fruit juice (7.93%) by mixing in the orbital shaker (Heidolph, Unimax 2010) at slow speed. Then, the supernatant obtained by centrifugation at 5000 rpm at 20°C for 10 min was titrated with a fixed 0.025% (w/v) 2,6-dichlorophenolindophenol solution until a light pink color was obtained. The results are expressed as mg ascorbic acid (AA)/kg (Tareen et al., 2015).

2.5 | Determination of optimum parameters for spray-drying of bergamot juice

Response surface methodology (RSM) was used for optimizing the spray-drying parameters. The data analysis was performed by a design expert (Version: 11.0, Stat-Ease Inc.).

2.6 | Physicochemical properties of spray-dried bergamot juice powder

The following analyses were performed in three replications-three parallel to spray-dried bergamot juice powder.

2.6.1 | Antioxidant activity, total phenolic content, and vitamin C content

It is made as specified in Sections 2.4.2 and 2.4.3.

2.6.2 | pH and titrable acidity

A 1 g aliquot of powder was dissolved in 10 mL of DW, and the pH was measured at 27°C using a pH meter (WTW, InoLab 720, Xylem, Weilheim, Germany). Titratable acidity was determined by the titration of the aqueous mixture of the powder against 0.1 N NaOH with the phenolphthalein indicator, and the acidity (%) was determined as citric acid equivalent (Saikia et al., 2015).

2.6.3 | Water activity

The water activity of the powder was determined with a water activity meter (Aqualab, 4TE) at 25°C (Saikia et al., 2015).

2.6.4 | Color

*L**, *a**, and *b** color values of the powder were determined with a colorimeter (Konica Minolta, CR-400, Tokyo, Japan) (Saikia et al., 2015).

2.6.5 | Moisture content

The powder's moisture content (%) was determined gravimetrically by drying approximately 1 g of the sample in an oven (Simsek Laborteknik, Ankara, Turkey) at 105°C to constant weight.

2.6.6 | Hygroscopicity

A 1 g aliquot of the powder was placed in preweighed glass Petri dishes and kept in a desiccator at 25°C containing saturated NaCl solution (75% relative humidity) for 7 days. At the end of the period, the sample cups were weighed, and the hygroscopicity was expressed as g moisture/100 g solids (Saikia et al., 2015).

2.6.7 | Caking degree

After determining the hygroscopicity, the wet sample was dried in an oven at 70°C, and after cooling, it was weighed and sieved through a 500 μm mesh diameter sieve (Retsch, Thermo Fisher Scientific, Waltham, MA, USA). The weight of the powder remaining on the sieve was determined, and the degree of caking (%) was calculated by the ratio of the initial powder amount to the powder remaining on the sieve (Goula & Adamopoulos, 2008).

2.6.8 | Bulk and tapped densities

The powder was weighed into a graduated cylinder, and the volume-to-mass read ratio calculated the mass density (ρ_{bulk}). For the tapped density (ρ_{tapped}), the volume was calculated with the volume read due to the maximum compression of the sample, and the results were expressed in g/mL units (Jinapong et al., 2008).

2.6.9 | Particle density

An amount of 1 g of powder was shaken with 5 mL of petroleum ether until all powder particles were suspended, and the remaining powder particles were remixed with another 1 mL of petroleum ether. Particle density ($\rho_{particle}$) was calculated as the powder weight ratio to volume difference as g/mL units (Jinapong et al., 2008).

2.6.10 | Porosity

The porosity (%) was determined by the ratio of the $\rho_{particle}$ and ρ_{tapped} difference to the $\rho_{particle}$ of the powder (Jinapong et al., 2008).

2.6.11 | Flowability and cohesiveness

The flowability (Carr index, CI) and cohesiveness (Hausner ratio, HR) of the powder were calculated from the ρ_{bulk} and ρ_{tapped} values of the powder (Jinapong et al., 2008).

2.6.12 | Individual phenolics by high-pressure liquid chromatography

Spray-dried bergamot juice powder was diluted with ultrapure water to the juice's initial amount of soluble dry matter (%7.93). The aqueous powder extract was filtered through a 0.45 μm PTFE filter and injected into HPLC (Shimadzu, LC-20AD) with conditions specified by Papoutsis et al. (2017) to quantify the major phenolic compounds.

Analysis was performed using a C18 column (Macherey-Nagel, EC 250/4.6 Nucleosil 300-5, Dueren, Germany) at 30°C oven (Shimadzu, CTO-10ASVP) temperature, 40 min elution time and 1 mL/min flow rate. The mobile phase contained water:acetonitrile:formic acid, 95:4:1 (v:v:v) (Mobile Phase A) and acetonitrile (Mobile Phase B). In analysis, gradient flow was used 0 min 5% B; 15 min, 20% B; 32 min, 5% B; 40 min, 5% B. Photodiode array (PDA) detection (Shimadzu, SPD-M20A) was performed at 280 nm. Components were determined according to their retention times and peak areas. Concentrations were determined using calibration curves prepared by dissolving standard phenolic compounds in 80% methanol. The performance parameters of the method for conducting this analysis with high accuracy and precision are given in Table 2.

2.7 | Physicochemical properties of fresh bergamot juice

Antioxidant activity, total phenolic content, vitamin C content, moisture, pH, titratable acidity, color, and the determination of individual phenolics analysis of bergamot juice powder was also performed on fresh bergamot juice by the methods mentioned in Section 2.5. In addition, the

TABLE 2 Performance characteristics of method.

Phenolic compound	LR (μg/mL)	*r*	Equation	LOD	LOQ	*R* (%)
Gallic acid	5–50	0.9970	$Y = 0.63978x - 0.16370$	0.57	1.72	85.72
p-Coumaric acid	5–20	0.9964	$Y = 0.11825x + 0.13812$	1.12	3.38	84.03
Rutin	1–20	0.9977	$Y = 0.14121x + 0.50531$	2.10	6.37	92.20
Naringin	10–500	0.9965	$Y = 5.13052x + 1.09523$	0.03	0.08	96.48
Hesperidin	5–50	0.9967	$Y = 0.70504x - 0.16485$	0.94	2.84	72.87
Eriocitrin	10–500	0.9647	$Y = 4.85498x + 1.78924$	0.17	0.51	86.11
Limonin	5–100	0.9972	$Y = 1.05789x - 0.04857$	3.09	9.36	83.52
Neoeriocitrin	20–200	0.9795	$Y = 2.26881x + 0.97825$	0.35	1.06	70.50
Narirutin	0–20	0.9804	$Y = 0.10689x + 0.10258$	4.32	13.10	88.64
Neohesperidin	5–100	0.9872	$Y = 1.95148x - 0.75896$	1.66	5.02	87.93
Naringenin	10–500	0.9886	$Y = 4.90026x + 3.03525$	0.50	1.51	90.16
Hesperetin	0–10	0.9911	$Y = 0.05676x - 0.57587$	5.81	17.62	91.32

Abbreviations: LOD, limit of detection; LOQ, limit of quantification; LR, linear range; *r*, correlation coefficient; *R*, recovery in sample.

juice's total water-soluble solids (%) were determined with a digital refractometer (Atago, RX-5000, Tokyo, Japan).

2.8 | Ultrasound-assisted extraction of phenolics from the bergamot juice powder

The powder (1 g) is diluted (w/v) with a solvent acetone (ACTN), dimethyl sulfoxide (DMSO), and ethanol (EtOH) at 50%, 60%, 70%, 80%, 90%, and 100% concentration to equal the initial water-soluble solids content of juice (7.93%). The mixture was vortexed for 30 s and placed in capped glass tubes. Ultrasound-assisted extraction (ISOLAB Laborgerate, GmbH, Eschau, Germany) was performed for 30 min at 53 kHz, 100% power, and 20°C (our preliminary trials determined process parameters). Then, it was centrifuged at 5000 rpm at 20°C for 10 min, and the supernatants obtained were taken into amber-colored vials and stored at −20°C until use. The following analyzes were performed on the prepared extracts to determine the optimum concentration for each solvent.

2.8.1 | Antioxidant activity, total phenolic, and vitamin C content of powder extracts

The antioxidant activity, total phenolic content, and vitamin C content analyses of these extracts prepared with different solvents at different concentrations were performed as described in the Sections 2.4.2 and 2.4.3. As a result, the concentration that maximized these contents was determined for each solvent, and extracts prepared with these optimal concentrations of solvents were used in subsequent stages.

2.8.2 | Individual phenolics of extracts by high-pressure liquid chromatography

The individual phenolic compounds of the extracts prepared with three different solvents and DW as a control sample with determined optimal concentrations were identified as described in Section 2.6.12.

2.9 | Statistical analysis

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

3 | RESULTS

To improve the properties of the powder obtained by spray-drying bergamot juice, the processing conditions were optimized with a Box–Behnken design. The results of response variables (process yield, antioxidant activity, total phenolic, and vitamin C content) measured for each set of the experimental run are presented in Table 1.

Process yield was determined as 72.45%–93.69%, antioxidant activity 37.35%–62.54%, DPPH scavenging activity, total phenolic content 1865.45–3862.05 ppm GAE, and

TABLE 3 Analysis of variance (ANOVA) of quadratic model terms of dependent variables.

	Sum of Squares	df	Mean square	*F*-value	*p*-Value	Inference
Process yield						
Model	546.18	9	60.69	1736.88	<0.0001	Significant
Residual	0.1747	5	0.0349			
Lack of fit	0.1486	3	0.0495	3.80	0.2153	Not significant
Pure error	0.0261	2	0.0130			
Antioxidant activity						
Model	603.68	9	67.08	973.06	<0.0001	Significant
Residual	0.3447	5	0.0689			
Lack of fit	0.3030	3	0.1010	4.85	0.1757	Not significant
Pure error	0.0417	2	0.0208			
Total phenolic content						
Model	5.065E + 06	9	5.627E + 05	95.95	<0.0001	Significant
Residual	29,323.78	5	5864.76			
Lack of fit	25,687.31	3	8562.44	4.71	0.1801	Not significant
Pure error	3636.47	2	1818.24			
Vitamin C content						
Model	7.056E + 05	9	78,398.85	88.02	<0.0001	Significant
Residual	4453.61	5	890.72			
Lack of fit	2263.63	3	754.54	0.6891	0.6376	Not significant
Pure error	2189.98	2	1094.99			

vitamin C content 681.48–1407.41 ppm AA in spray-dried bergamot juice powders produced with different process conditions.

In the response, variables x_1, x_2, x_3, x_1x_2, x_1x_3, x_2x_3, ${x_1}^2$, ${x_2}^2$, and ${x_3}^2$ are determined as significant model terms in quadratic models. The ANOVA results of the second-degree model terms of the dependent variables are provided in Table 3. Here, the *F* and *p*-values associated with the model are used to determine statistically significant factor effects. The *F*-value represents the ratio of the variance between factors. In this study, high *F*-values have shown that there is a significant difference between factors. However, even when the *F*-value is high, the *p*-value should also be considered. The *p*-value indicates the reliability of the hypothesis test. If the *p*-value is less than 0.05, the hypothesis test is statistically significant and cannot be rejected. In this study, *p*-values lower than 0.0001 have shown that the established hypothesis is significant. Overall, a high *F*-value and a low *p*-value obtained from the Box–Behnken experimental design indicate significant differences between factors. This means that both the terms used in the model are essential, and the differences between these terms are significant.

On the other hand, the lack of fair value in the ANOVA results for model terms should be insignificant, indicating model adequacy. As seen in Table 3, the lack of fit was determined to be insignificant for each dependent variable. The significant model and insignificant lack of fit indicate the model's accuracy. In this case, optimum conditions can be determined with high accuracy and precision.

In support of this, the adjusted coefficient of determination (R^2), adjusted R^2 (AdjR^2), and predictive R^2 (PredR^2) values for all dependent variable models were determined to be higher than 0.90. The polynomial equation obtained from the experiments and its corresponding coefficients is provided in Table 4. This equation allows for the accurate calculation of process efficiency, antioxidant activity, total phenolic content, and vitamin C content even under conditions not used in the experiments.

3.1 | Effects of maltodextrin concentration

This research determined that all dependent variables decreased with an increase in maltodextrin concentration, provided that other independent variables were considered constant.

Higher maltodextrin concentration increased the viscosity of the feed, resulting in slower drying and wet powder formation in the final product. Thus, the process efficiency is reduced. Tonon et al. (2008) reported a similar finding in dried acai powder.

TABLE 4 Coefficients table.

	Intercept	x_1 [a]	x_2 [a]	x_3 [a]	x_1x_2	x_1x_3	x_2x_3	x_1^2	x_2^2	x_3^2
Process yield	89.26	**−3.27**	**2.79**	**−5.66**	*−0.11*	**−3.52**	**2.86**	−0.23	**−1.58**	**−3.81**
p-Values		**<0.0001**	**<0.0001**	**<0.0001**	*0.2760*	**<0.0001**	**<0.0001**	0.0639	**<0.0001**	**<0.0001**
Antioxidant activity	45.44	**−7.28**	**−2.28**	**0.75**	**−1.12**	**−4.52**	**1.74**	**2.63**	*−0.11*	**1.67**
p-Values		**<0.0001**	**<0.0001**	**0.0005**	**0.0004**	**<0.0001**	**<0.0001**	**<0.0001**	*0.4640*	**<0.0001**
Total phenolics	2693.49	**−346.18**	**−135.99**	**−342.03**	**366.03**	**−446.03**	−92.97	**205.12**	**−424.20**	**441.82**
p-Values		**<0.0001**	**0.0040**	**<0.0001**	**0.0002**	**<0.0001**	0.0595	**0.0036**	**0.0001**	**0.0001**
Vitamin C content	988.42	**−275.70**	**−30.40**	*−6.56*	*−15.12*	**−74.23**	*24.07*	**104.79**	**−52.78**	**−48.61**
p-Values		**<0.0001**	**0.0345**	*0.5614*	*0.3575*	**0.0042**	*0.1676*	**0.0011**	**0.0193**	**0.0260**

Note: Those with $p < 0.05$ **are in bold**; $0.05 \leq p < 0.1$ are normal; $p \geq 0.1$ *are in italics*.

[a] x_1 is the maltodextrin concentration, x_2 is the inlet air temperature, and x_3 is the pump rate.

Adding maltodextrin at increasing concentrations caused a percent decrease in antioxidant and phenolic compounds in the feed. Similarly, a decrease in vitamin C in the feed was observed. Maltodextrin does not have antioxidant activity, and when added in high amounts to the feed, it dilutes the feed, thus reducing the antioxidant activity and phenolic content (Sánchez-Madrigal et al., 2019). Mishra et al. (2014) reported that increasing maltodextrin concentration decreased the total phenolic content and antioxidant activity in fruit juice powder.

3.2 | Effects of inlet air temperature

It was determined that all dependent variables decreased with the increase in inlet air temperature, provided that other independent variables were considered constant.

High inlet air temperature causes high outlet air temperature. Thus, a crust forms on the particle surface, and water diffusion becomes difficult. As the final product remains moist, the powder obtained is reduced. Similar results have been reported in the literature (Ávila et al., 2014).

Drying at high temperatures reduced the content of heat-sensitive components in the feed. The content of heat-sensitive compounds (polyphenols, antioxidants, pigments, vitamins, etc.) decreases with increasing temperature due to thermal and oxidative degradation (Pernice et al., 2009). This has been proved in different heat-sensitive compounds, such as betacyanin, anthocyanin, vitamin C, total phenolic content, and antioxidant activity (Mishra et al., 2014; Murali et al., 2015; Tonon et al., 2008).

3.3 | Effects of the pump rate

All dependent variables decreased with the increased pump rate, provided that other independent variables were considered constant.

When the pump speed increases, the feed is given to the system faster (Shishir & Chen, 2017). The high feed flow rate does not allow sufficient interaction time between the feed and the hot air, and as there is less heat–mass transfer at the shorter contact time, it is difficult for the droplets to dry out, and wet particles are formed. In addition, in larger droplets formed at a high feed flow rate, the water in the droplet evaporates less because the contact time between the droplet and the air is reduced. In other words, as moisture remains in the final product, product yield has decreased. Many researchers have also reported that increased feed flow rate has a negative effect on process yield (Fazaeli et al., 2016; Tonon et al., 2008).

At a high feed flow rate, as the amount of recovered powder is low, the content of antioxidants, total phenolic, and vitamin C in the product also decreased.

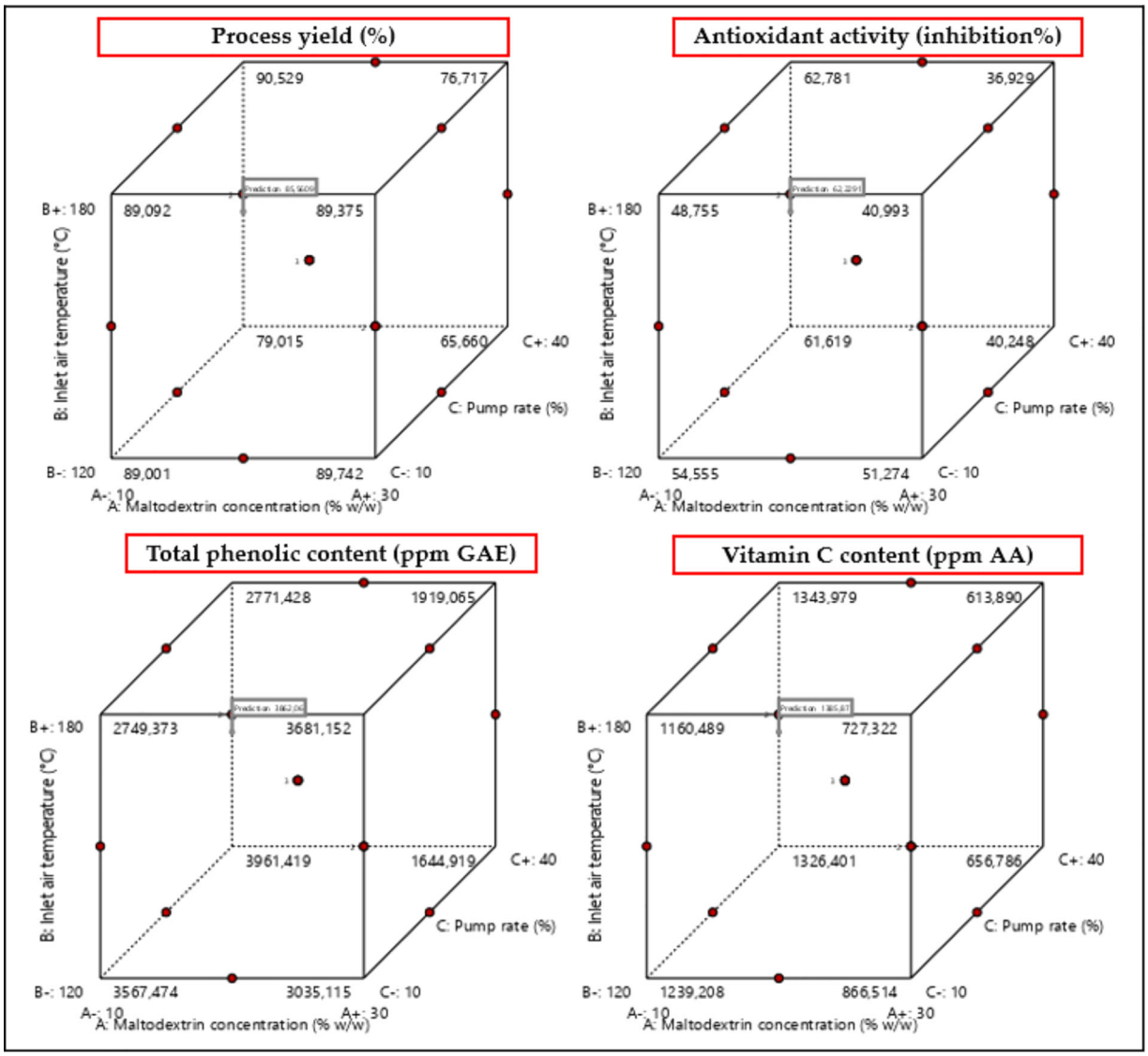

FIGURE 1 Cube graphs of optimization.

3.4 | Optimum spray-drying conditions for the production of bergamot juice powder

The choice of operating parameters is critical to obtain a high-quality product during spray-drying. The effects of inlet temperature (especially 120–180°C) and maltodextrin concentration (especially 7%–20%) variables on powder properties have been extensively investigated in Box–Behnken design optimization as an RSM design (Shishir & Chen, 2017).

The cube graphs of the optimization are given in Figure 1. The response surface has been optimized to achieve maximum efficiency at the intersection point of the optimal values for each factor. Optimization criteria x_1, x_2, x_3, and Y_1 "in range" Y_2, $_3$, $_4$ were determined at the same level of importance as "maximize." Optimum processing parameters were obtained as 10% maltodextrin concentration, 146.02°C inlet temperature, and 39.99% pump rate. This study's desirability value of 0.986 indicates that expectations were met mainly, and an approximate desired outcome was achieved. The value is close to 1, the highest possible desirability score.

Thus, these processing conditions were determined as the optimum levels of the independent variables in the experimental plan. Under these conditions, the analysis results were determined as 85.56% process yield, 62.23% antioxidant activity, 3862.06 ppm GAE total phenolic content, and 1385.87 ppm AA vitamin C content.

In order to verify the results, another drying process was carried out with optimum conditions. Analyzes were made on the powder product obtained here, and it was determined that the results were more than 87.13% similar to the values obtained in the optimization.

TABLE 5 Physicochemical properties of fresh bergamot juice and its powder.

	Unit	Bergamot juice powder	Fresh bergamot juice
Antioxidant activity	DPPH% scavenging act.	62.23 ± 4.42^{b}	79.23 ± 5.30^{a}
Total phenolics	ppm GAE	3862.06 ± 64.52^{b}	5374.54 ± 35.74^{a}
Vitamin C	ppm AA	1385.87 ± 51.02^{b}	2033.58 ± 22.67^{a}
Moisture content	%	4.57 ± 0.15^{b}	89.29 ± 0.61^{a}
Dry matter	%	96.43 ± 0.47^{a}	10.71 ± 0.35^{b}
pH		2.75 ± 0.01^{a}	2.77 ± 0.01^{a}
Titrable acidity	Citric acid %	2.56 ± 0.03^{a}	2.56 ± 0.01^{a}
L^*		43.51 ± 1.14^{b}	94.30 ± 0.87^{a}
a^*		-1.35 ± 0.58^{a}	-1.61 ± 0.17^{b}
b^*		10.94 ± 0.87^{a}	8.10 ± 0.94^{b}
Total soluble solids	%	Ns	7.93 ± 0.01
Water activity		0.190.03	ns
Bulk density	g/mL	0.42 ± 0.01	ns
Tapped density	g/mL	0.52 ± 0.04	ns
Particle density	g/mL	5.00 ± 0.02	ns
Porosity	%	89.60 ± 0.24	ns
Flowability (CI)	%	20.00 ± 1.12	ns
Cohesiveness (HR)		1.25 ± 0.57	ns
Hygroscopicity	g moisture/100 g	8.58 ± 1.16	ns
Caking degree	%	37.21 ± 2.54	ns
Gallic acid	ppm	7.49 ± 0.02^{b}	49.76 ± 0.05^{a}
p-Coumaric acid	ppm	2.29 ± 0.15^{b}	18.01 ± 0.49^{a}
Rutin	ppm	1.49 ± 0.25^{b}	16.36 ± 0.97^{a}
Naringin	ppm	29.64 ± 0.31^{b}	197.50 ± 0.10^{a}
Hesperidin	ppm	5.64 ± 0.74^{b}	28.24 ± 0.05^{a}
Eriocitrin	ppm	17.19 ± 0.01^{b}	105.90 ± 0.55^{a}
Limonin	ppm	6.70 ± 0.15^{b}	37.43 ± 0.40^{a}
Neoeriocitrin	ppm	8.64 ± 0.85^{b}	53.38 ± 0.31^{a}
Narirutin	ppm	0.68 ± 0.04^{b}	10.35 ± 0.25^{a}
Neohesperidin	ppm	6.65 ± 0.03^{b}	68.77 ± 0.07^{a}
Naringenin	ppm	14.23 ± 0.10^{b}	119.80 ± 0.03^{a}
Hesperetin	ppm	0.12 ± 0.07^{b}	1.17 ± 0.02^{a}

Note: Different letters in the same line show differences between sample groups according to the Duncan test ($p < 0.05$).
Abbreviations: CI, Carr index; HR, Hausner ratio; ns, not subjected.

3.5 | Physicochemical properties of bergamot juice powder obtained under optimum spray-drying conditions and fresh bergamot juice

The physicochemical properties of the spray-dried product mainly depend on the type and concentration of the carrier agent, the feed flow rate, and the inlet temperature. Some physicochemical properties of fresh bergamot juice and bergamot juice powder obtained with optimum conditions of the spray-drying process (10% maltodextrin concentration, 146.02°C inlet temperature, and 39.99% pump rate) are given in Table 5.

The content of total soluble solids expressed as Brix did not exceed 10% in the laboratory and industrial juices (Cautela et al., 2019), so the Brix value of freshly squeezed juice (7.93%) is appropriate.

The moisture content of spray-dried powders is usually <5%, and water activity is <0.3. These values indicate that the final product is microbiologically and chemically safe and can remain stable throughout its shelf life (Bicudo et al., 2015). The moisture content of the bergamot juice powder was 4.57%, and the water activity was 0.19.

Hygroscopicity is defined as the ability of food powder to absorb environmental moisture. It is desirable to produce a food powder with low hygroscopicity (<20%) as high

hygroscopicity means it tends to absorb more water and cause stickiness (Nurhadi et al., 2012). The hygroscopicity of the bergamot juice powder was found to be 8.58 g moisture/100 g dry matter. In this regard, the degree of caking was determined as 37.21%.

Solubility, which is the ability of powders to form solutions or suspensions in water, is the most crucial criterion for evaluating the behavior of powder in an aqueous solution. Especially when the obtained powder is used as an additive in the production of different products, higher solubility is required (Cavia-Saiz et al., 2010). The 100% solubility of bergamot juice powder is related to the recovery of water-soluble solids in the feed solution by spray-drying. In addition, maltodextrin added to the structure also provides water solubility.

The color properties of food powders generally vary according to the raw material. The product's color is affected by the carrier material, its higher concentration, natural color, and nonenzymatic browning reactions of sugars at high drying temperatures (Tontul & Topuz, 2017). The L^* value was 43.51, a^* value −1.35, and b^* value 10.94 in bergamot juice powder, and these values reflect the original colors of bergamot juice.

Powders with smooth and even surfaces have greater bulk density. High bulk density is desirable to reduce shipping and packaging costs (Tontul & Topuz, 2017). The ρ_{bulk} value of the bergamot juice powder was 0.42, the ρ_{tapped} value was 0.50, and the $\rho_{particle}$ value was 5 g/mL. Additionally, bulk density influences other powder properties, such as flowability (Murali et al., 2015). Our results determined the bergamot juice powder's porosity as 89.60%. Due to the nature of the bergamot juice powder, a good level of fluidity and an average stickiness were detected; this finding can be attributed to the polysaccharide structure of maltodextrin in the final product.

The percentages of preservation of antioxidant activity and total phenolic and vitamin C content in bergamot juice powder obtained by spray-drying fresh bergamot juice were determined as 78.54%, 71.86%, and 68.15%, respectively. Powder and fresh juice have the same pH and titratable acidity levels. Our results are similar to values of 2.54–2.97 reported by Sicari et al. (2016) and Sicari and Pellicanò (2016). These results show that the spray-drying method successfully produces bergamot powder with the optimum process conditions.

3.6 | Individual phenolics of bergamot juice powder obtained under optimum spray-drying conditions and fresh bergamot juice

The amounts of major phenolic compounds in fresh bergamot juice and spray-dried bergamot juice powder at optimum conditions (10% maltodextrin concentration, 146.02°C inlet temperature, and 39.99% pump rate) are given in Table 5. In addition, HPLC chromatograms of the aqueous extract of bergamot juice powder are also given in Figure 2.

The amounts (mg/kg) of phenolic compounds in bergamot juice have been indicated differently by many researchers; naringin 43–296, naringenin 0.5–122, eriocitrin 10–151, neoeriocitrin 29–296, neohesperidin 58–236, hesperidin 10–172, narirutin 4–9, rutin 8–20, hesperetin 1–10, and limonin in bergamot juice are in the range of 1–22 (Cautela et al., 2008; Di Donna et al., 2011; Gardana et al., 2008; Multari et al., 2020; Nicolosi et al., 2000; Nurhadi et al., 2012; Sicari & Pellicanò, 2016; Sicari & Poiana, 2017; Sicari et al., 2016). Our results are similar to the data in the literature; the differences are related to the characteristics of bergamot, such as species, variety, harvest time, maturity level, and chemical composition. Based on the amounts given above, a wide range of data has been reported in the literature. In the literature, the major phenolic components of bergamot juice have been determined by many researchers as naringin, neohesperidin, eriocitrin, and neoeriocitrin.

In our results, naringin, neohesperidin, eriocitrin, and neoeriocitrin constituted 60.22% of the total determined phenolic compounds (12 units) in bergamot juice and 61.66% in bergamot juice powder. Thus, major phenolic components in fresh bergamot juice and bergamot juice powder were confirmed.

It was determined that the bergamot juice powder produced by the spray-drying process did not have bitterness in the bergamot juice. This is associated with the presence of naringin and neohesperidin. Naringin is a flavonon glycoside that is even more bitter than quinine and responsible for citrus fruits' distinctive taste. The aglycone part of this compound is naringenin (Alam et al., 2014). Naringin is hydrolyzed to naringenin, and all naringin can be debited with complete hydrolysis at a high temperature.

On the other hand, neohesperidin, an isomer of hesperidin, also causes bitterness, but this bitterness is 10% of naringin (Cavia-Saiz et al., 2010). Our results determined that naringin was reduced by 84.99% and neohesperidin by 90.33% in bergamot juice powder compared to fresh bergamot juice. As the temperature level used in the spray-dryer damaged the structure of naringin and neohesperidin, most of the bitterness disappeared.

These results show that, despite high temperatures, major phenolic compounds can be preserved at a certain level in converting bergamot juice into powder form by spray-drying with optimum process parameters.

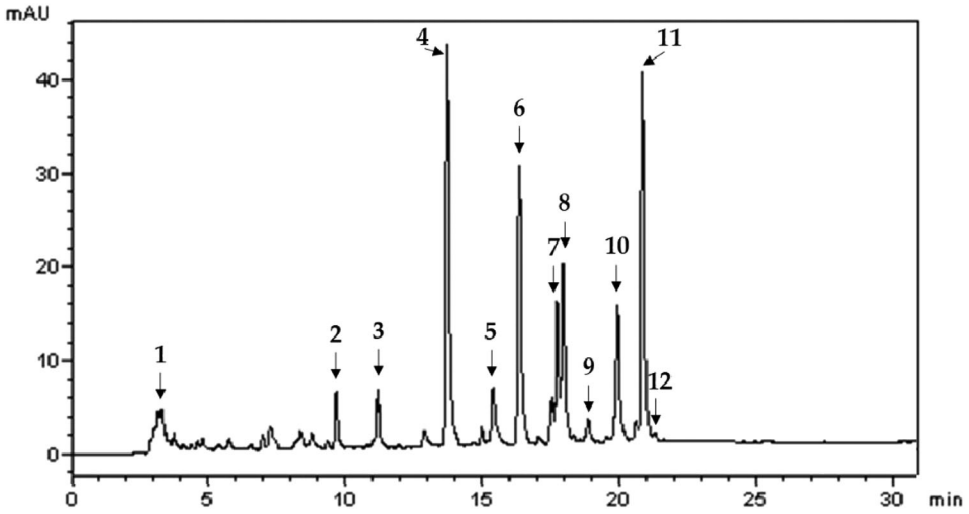

FIGURE 2 Chromatogram of spray-dried bergamot juice powder at 280 nm. Here: (1) gallic acid, (2) *p*-coumaric acid, (3) rutin, (4) naringin, (5) hesperidin, (6) eriocitrin, (7) limonin, (8) neoeriocitrin, (9) narirutin, (10) neohesperidin, (11) naringenin, and (12) hesperetin peaks.

3.7 | Antioxidant activity, total phenolic content, and vitamin C content of bergamot juice powder extracts prepared with different solvents at different concentrations

Phenolic compounds from bergamot juice powder were extracted using an ultrasonic-assisted extraction method. Various solvents, including ACTN, DMSO, and EtOH at different concentrations, were used to prepare the extract from the plant source, which is the most critical parameter affecting the chemical composition of the final product in this research. The reason for preparing extracts with different solvents is that different solvents have different polarity characteristics. The distribution of positive and negative charges in the molecule determines the polarity of a solvent. Some solvents are more suitable for dissolving polar molecules, whereas others are more suitable for dissolving nonpolar molecules. Therefore, using different solvents can extract different components and allow for the selective extraction of components.

The antioxidant activity, total phenolic content, and vitamin C content of extracts prepared with different solvents at various concentrations are given in Table 6.

The antioxidant activity, total phenolic content, and vitamin C content of extracts prepared with ACTN, DMSO, and EtOH at concentrations ranging from 50% to 100% are as follows:

- For ACTN, the antioxidant activity ranged from 72.74% to 8.36%, the total phenolic content ranged from 4898.52 to 1242.83 ppm GAE, and the vitamin C content ranged from 1734.04 to 1142.70 ppm AA.
- For DMSO, the antioxidant activity ranged from 54.44% to 70.54%, the total phenolic content ranged from 2854.47 to 4785.10 ppm GAE, and the vitamin C content ranged from 1201.07 to 1699.57 ppm AA.
- For EtOH, the antioxidant activity ranged from 67.47% to 27.92%, the total phenolic content ranged from 4412.66 to 1380.29 ppm GAE, and the vitamin C content ranged from 1537.84 to 1038.29 ppm AA.

As the water content in the solvent increased, the antioxidant activity, total phenolic content, and vitamin C content increased for ACTN and EtOH, whereas these values decreased for DMSO.

3.8 | Determination of the optimum concentration for each solvent

According to the results of the antioxidant activity, total phenolic and vitamin C content of the extracts prepared with solvents at different concentrations, the highest values were obtained with 50% ACTN, 50% EtOH, and 100% DMSO solvents, with the maximum values achieved with ACTN solvent.

The antioxidant activity and total phenolic and vitamin C content of the extracts prepared with solvents at optimum concentrations and DW are given in Table 7. Based on the analysis, the highest values were observed in ACTN, DMSO, EtOH, and DW extracts, respectively. Polyphenols are bioactive compounds that have the potential to replace synthetic antioxidants due to their free radical scavenging and inhibition of oxidation reactions (Navarra et al.,

TABLE 6 Analysis results of extracts.

Solvent concentration	Acetone	Dimethyl sulfoxide	Ethanol
Antioxidant activity (DPPH% radical scavenging activity)			
50%	72.74 ± 1.43^{aA}	54.44 ± 6.13^{cB}	67.47 ± 3.98^{aA}
60%	67.44 ± 4.22^{bA}	60.36 ± 3.34^{bA}	64.60 ± 5.28^{aA}
70%	65.63 ± 2.52^{bcA}	62.03 ± 2.58^{bA}	63.44 ± 1.92^{aA}
80%	62.93 ± 2.59^{cA}	63.19 ± 0.49^{bA}	62.80 ± 2.91^{aA}
90%	41.05 ± 1.40^{dB}	63.96 ± 0.62^{bA}	43.62 ± 4.22^{bB}
100%	8.36 ± 0.38^{eC}	70.54 ± 1.88^{aA}	27.92 ± 0.93^{cB}
Total phenolic content (ppm GAE)			
50%	4898.52 ± 2.63^{aA}	2854.47 ± 4.68^{eC}	4412.66 ± 8.61^{aB}
60%	3072.10 ± 2.26^{bB}	2877.38 ± 9.35^{deC}	3980.47 ± 14.57^{bA}
70%	3060.65 ± 30.73^{bB}	2934.65 ± 39.94^{dC}	3716.01 ± 2.50^{cA}
80%	2591.01 ± 35.61^{cC}	3003.38 ± 11.61^{cB}	3479.56 ± 35.10^{dA}
90%	2216.47 ± 45.61^{dC}	3429.31 ± 1.72^{bA}	2399.74 ± 20.14^{eB}
100%	1242.83 ± 30.00^{eC}	4785.10 ± 73.69^{aA}	1380.29 ± 21.64^{fB}
Vitamin C content (ppm AA)			
50%	1734.04 ± 5.09^{aA}	1201.07 ± 13.95^{dC}	1537.84 ± 16.59^{aB}
60%	1234.14 ± 14.70^{bC}	1287.41 ± 25.11^{cB}	1480.47 ± 11.20^{bA}
70%	1209.26 ± 11.44^{cC}	1303.65 ± 4.79^{cB}	1369.01 ± 6.75^{cA}
80%	1197.14 ± 15.54^{cC}	1487.38 ± 7.91^{bA}	1279.56 ± 7.62^{dB}
90%	1150.32 ± 9.57^{dC}	1503.31 ± 4.07^{bA}	1199.74 ± 11.14^{eB}
100%	1142.70 ± 3.25^{dB}	1699.57 ± 28.72^{aA}	1038.29 ± 5.62^{fC}

Note: Different letters in the same column (a) and line (A) show differences between sample groups according to the Duncan test ($p < 0.05$).

TABLE 7 Results of extracts prepared with optimum concentrations of solvents and distilled water.

Solvent	Antioxidant activity (DPPH% radical scavenging activity)	Total phenolic content (ppm GAE)	Vitamin C content (ppm AA)
50% acetone	72.74 ± 1.43^{a}	4898.52 ± 2.63^{a}	1734.04 ± 5.09^{a}
Dimethyl sulfoxide	70.54 ± 1.88^{ab}	4785.10 ± 13.69^{b}	1699.57 ± 8.72^{b}
50% ethanol	67.47 ± 3.98^{bc}	4412.66 ± 8.61^{c}	1537.84 ± 6.59^{c}
Distilled water	65.02 ± 2.50^{c}	4105.74 ± 1.82^{d}	1425.17 ± 5.85^{d}

Note: Different letters in the same column show differences between sample groups according to the Duncan test ($p < 0.05$).

2015). In bergamot juice powder extracts, higher phenolic content provided higher antioxidant activity.

Generally, water, ethanol, methanol, chloroform, dichloromethanol, ether, and acetone are used to extract bioactive compounds from plant materials (Annegowda et al., 2012; Azmir et al., 2013; Suleria & Barrow, 2019). The application of mixtures of acidified methanol, ethanol, acetone, ethyl acetate, and other solvents with water has been widely reported in the literature (Annegowda et al., 2012; Haminiuk et al., 2011, 2014; Prasad et al., 2011; Ramful et al., 2011).

The main objective of this research was to extract phenolic compounds. According to the results, ACTN was the most effective solvent, whereas DW solvent was the least effective. The higher effectiveness of ACTN as a solvent indicates that phenolic compounds are better dissolved and more efficiently extracted by ACTN. ACTN, due to its lower polarity, was considered a more suitable solvent for increasing the solubility of phenolic compounds and extracting them. On the other hand, more polar solvents, such as DMSO, EtOH, and DW, were found to be less effective in dissolving phenolic compounds. The high to low polarity ranking is DMSO, EtOH, and ACTN.

Properties of extraction solvents significantly affect the total phenolic content (~ca. 25% variation) and antioxidant capacity (~ca. 30% variation) in fruits and vegetables. Solvent polarity is the most critical parameter in this context; the higher the polarity, the better the solubility of phenolic compounds. The highest extract yield can be obtained

TABLE 8 Phenolics of the extracts (ppm).

	50% acetone	Dimethyl sulfoxide	50% ethanol	Distilled water	Fresh juice
Gallic acid	34.75 ± 0.99^{b}	27.64 ± 0.46^{c}	18.67 ± 1.09^{d}	9.10 ± 0.11^{e}	49.76 ± 1.26^{a}
p-Coumaric acid	11.24 ± 0.39^{b}	10.15 ± 0.13^{c}	8.56 ± 0.41^{d}	4.45 ± 0.04^{e}	18.01 ± 0.16^{a}
Rutin	7.42 ± 0.02^{b}	4.48 ± 0.08^{c}	3.37 ± 0.07^{d}	2.89 ± 0.09^{e}	16.36 ± 0.06^{a}
Naringin	127.27 ± 0.86^{b}	97.82 ± 0.43^{c}	73.69 ± 1.00^{d}	33.24 ± 0.04^{e}	197.50 ± 0.10^{a}
Hesperidin	20.26 ± 0.99^{b}	12.47 ± 0.60^{c}	9.20 ± 0.20^{d}	7.20 ± 0.20^{e}	28.24 ± 0.24^{a}
Eriocitrin	58.63 ± 0.78^{b}	48.26 ± 0.85^{c}	44.06 ± 0.06^{d}	20.25 ± 0.25^{e}	105.90 ± 0.90^{a}
Limonin	27.11 ± 0.01^{b}	21.85 ± 1.57^{c}	17.39 ± 0.39^{d}	8.99 ± 0.99^{e}	37.43 ± 0.43^{a}
Neoeriocitrin	36.16 ± 0.38^{b}	27.01 ± 0.13^{c}	19.29 ± 0.29^{d}	10.24 ± 0.24^{e}	53.38 ± 0.38^{a}
Narirutin	5.46 ± 0.50^{b}	3.07 ± 0.50^{c}	2.16 ± 0.16^{d}	1.14 ± 0.14^{e}	10.35 ± 0.35^{a}
Neohesperidin	41.37 ± 0.69^{b}	33.16 ± 2.91^{c}	25.73 ± 0.73^{d}	7.98 ± 0.98^{e}	68.77 ± 0.77^{a}
Naringenin	65.28 ± 0.76^{b}	55.68 ± 1.68^{c}	45.54 ± 0.54^{d}	16.74 ± 0.74^{e}	119.80 ± 0.80^{a}
Hesperetin	0.80 ± 0.06^{b}	0.66 ± 0.25^{b}	0.36 ± 0.06^{c}	0.14 ± 0.10^{c}	1.17 ± 0.17^{a}

Note: Different letters in the same line show differences between sample groups according to the Duncan test ($p < 0.05$).

with polar alcohol-based solvents. Adding water to ethanol increases the extraction rate, but very high water content also increases the extraction of other compounds and, therefore, can lower the phenols concentrations in the extracts. In literature, it has been reported that the phenolic content of extracts obtained with an aqueous acetone solution is higher than ethanol extracts (Tomsone et al., 2012).

It was reported that antioxidant activity in bergamot juice was in the range of 22%–65% inhibition, total phenolic content was 752–17,100 ppm GAE, and vitamin C content was 125–2853 ppm (Cassano et al., 2013; Conidi et al., 2011; Hashemi & Jafarpour, 2020; Sicari & Pellicanò, 2016; Sicari et al., 2016). The data in the literature for bergamot juice are different, and our results are by most values. These discrepancies among literature and concerning present results may be ascribed to the phenolic fruit content that depends on many factors, such as genetic differences, degree of maturity at harvest, and environmental conditions.

3.9 | Individual phenolics of bergamot juice powder extracts

The amounts of individual phenolic compounds determined by HPLC for the four extracts (ACTN, DMSO, EtOH, and DW) given in Table 7 are provided in Table 8. In addition, the values for fresh bergamot juice from Table 5 have also been included in this table for easy comparison.

HPLC chromatograms of tested extracts and fresh bergamot juice are given in Figure 3. All identified compounds were 706.66, 435.73, 342.27, 268.03, and 122.36 ppm for fresh juice, ACTN extract, DMSO extract, EtOH extract, and DW extract, respectively. The preservation percentages of phenolic compounds in fresh fruit juice are 61.66% with ACTN, 48.43% with DMSO, 37.93% with EtOH, and 17.31% with DW. The retention rates of phenolic components in fresh bergamot juice in powder extracts are given in Figure 4. Here it appears all phenolics which can be extracted with ACTN in higher amounts.

The most abundant components in bergamot juices are naringin, neoeriocitrin, and neohesperidin, as reported by Calabrò et al. (2004), Dugo et al. (2005), Gattuso et al. (2006), and Nogata et al. (2006). Sicari et al. (2016) reported the amounts of eriocitrin, naringin, neoeriocitrin, and neohesperidin in the juices of bergamot fruits collected from different geographical areas of the same Province (Reggio Calabria, Italy) 6.36–14.45, 163.18–295.73, 158.59–296.77, and 110.79–244.82 ppm, respectively. Similarly, Cautela et al. (2019) found 114–402 mg/L neoeriocitrin, 18–37 mg/L narirutin, 97–394 mg/L naringin, 66–279 mg/L neohesperidin, and 20–65 mg/L limonin in different bergamot juices.

On the other hand, Sicari and Pellicanò (2016) reported the amounts of gallic acid, rutin, narirutin, neohesperidin, hesperidin, and narirutin in the juice of different varieties of bergamot as 1.10–1.86, 20.1–30.6, 1.33–3.41, 231.2–554.5, 33.5–37.5, and 148–182 mg/mL, respectively.

In a study conducted in the Reggio Calabria province (South Italy) (Giuffrè & Nobile, 2020), the authors reported the amounts of total flavonoids in the juice of different bergamot cultivars: Castagnaro cv. (362 mg/L), Fantastico cv. (460 mg/L), and Femminello cv. (520 mg/L), whereas the quantity of flavonoids was highest in the so-called cloudy juice (Giuffrè, 2019) of Castagnaro cv. (4660 mg/L), Fantastico cv. (6787 mg/L), and Femminello cv. (8438 mg/L). Naringin prevailed in both the juice and cloudy juice of all cultivars (32.69%–37.64% for Castagnaro cv, 26.82%–32.80% for Fantastico cv., and 31.90%–36.66% for Femminello cv. (Giuffrè, 2019)).

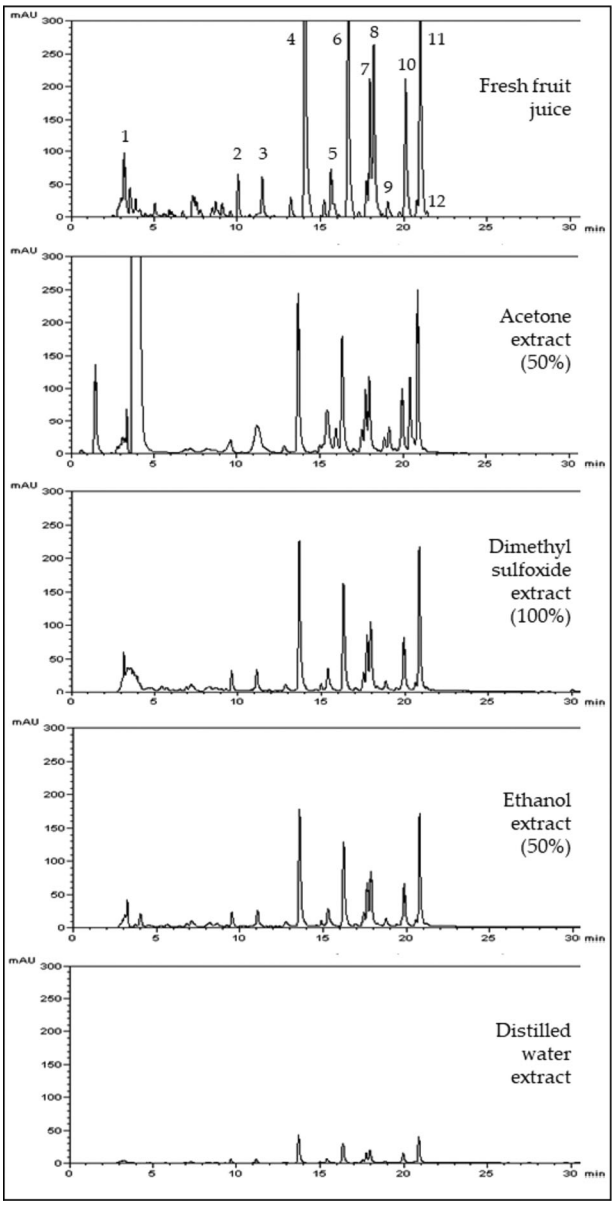

FIGURE 3 Chromatogram of spray-dried bergamot juice powder extracts at 280 nm. Here: (1) gallic acid, (2) *p*-coumaric acid, (3) rutin, (4) naringin, (5) hesperidin, (6) eriocitrin, (7) limonin, (8) neoeriocitrin, (9) narirutin, (10) neohesperidin, (11) naringenin, and (12) hesperetin peaks.

Our research determined the major phenolic compounds as naringin, eriocitrin, neoeriocitrin, and neohesperidin in both fresh bergamot juice and extracts. The major phenolic compounds in fresh juice, ACTN, DMSO, EtOH, and DW extracts are 425.55, 263.42, 206.26, 162.78, and 71.71 ppm, respectively. Thus, the extractability of major phenolics with each solvent was determined.

Compared to fresh bergamot juice, all phenolic compounds were determined in ACTN, DMSO, EtOH, and DW extract, respectively, from most to least. The results show that 50% ACTN is the most efficient solvent for extracting phenolic compounds in bergamot juice powder.

4 | DISCUSSION

If the relationship between all tested parameters is considered in detail, each factor affects the final product differently.

This research found that inlet temperature, carrier agent concentration, and pump rate were effective on tested chemical properties of bergamot juice powder in the spray-drying process. Finally, mathematical modeling determined optimum drying conditions as 10% maltodextrin concentration, 146.02°C inlet temperature, and 39.99% pump rate. In the spray-drying process with optimum parameters, antioxidant activity, total phenolic, and vitamin C content of fresh bergamot juice can be preserved at over 68.15% despite heat treatment.

The moisture content of bergamot juice powder was determined as 4.57% and water activity as 0.19. This shows that bergamot juice powder can be stored microbiologically, safely, and stably. The pH and acidity of bergamot juice powder were found to be 2.75 and 2.56, respectively, the same as fresh fruit juice. The natural acidity of the fruit is preserved. Compared to juice, the powder is darker and more yellow, although dilution achieves the original color of the juice. Bulk tapped and bergamot juice powder particle densities are suitable for use and storage. The powder form is porous due to its lightness, resulting in good flowability and low stickiness. As 100 g of bergamot juice powder can recover 8.58 g of moisture and has a caking degree of 37.21%, it has low hygroscopicity, which prevents stickiness in powder form.

This study revealed that major phenolic compounds, including gallic acid, *p*-coumaric acid, rutin, naringin, hesperidin, eriocitrin, limonin, neoeriocitrin, narirutin, neohesperidin, naringenin, and hesperetin, were present in fresh bergamot juice at varying concentrations. However, these compounds were also detected in bergamot juice powder, albeit at lower levels. Notably, the bitterness-associated compounds naringin and neohesperidin were significantly reduced in the powder, indicating that the bitterness of bergamot juice was effectively eliminated during the processing into powder form.

This study used ultrasound-assisted extraction to obtain phenolic compounds from spray-dried bergamot juice powder using different solvents, including ACTN, DMSO, EtOH, and DW. The choice of solvent is a critical factor that can significantly influence the chemical composition of the extracted product. The efficiency of the extraction process was evaluated at different concentrations (50%–100%) for each solvent. The extracts' antioxidant activity,

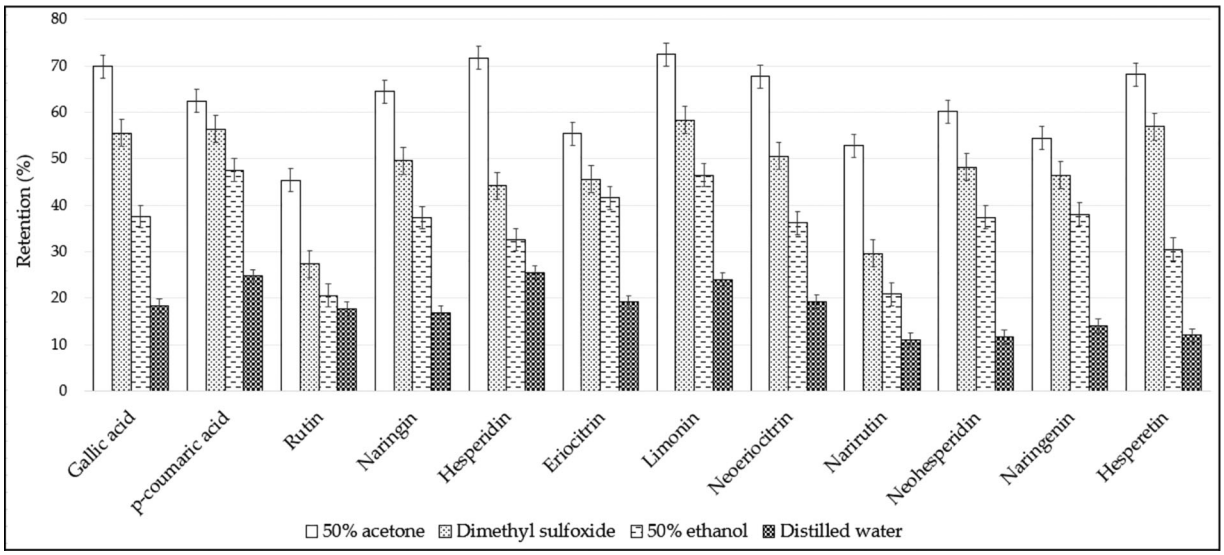

FIGURE 4 Retention (%) of individual phenolics of fresh juice with different solvents.

total phenolic content, and vitamin C content were determined for each solvent and concentration. The results showed that the extraction efficiency varied depending on the solvent and concentration.

Based on the antioxidant activity, total phenolic content, and vitamin C content, the solvents' effectiveness differed. ACTN and EtOH showed increased extraction efficiency with increasing water content in the solution, whereas DMSO showed decreased efficiency with increasing water content. The maximum extraction efficiency was achieved with a 50% concentration of ACTN, 100% DMSO, and 50% concentration of EtOH.

HPLC analysis was performed to determine the phenolic compounds in the extracts obtained with the optimal solvent and concentration for each solvent. The results were compared with the phenolic composition of fresh bergamot juice. The total amount of individual phenolic components in the extracts was lower than that in fresh juice, with ACTN providing the highest extraction efficiency among all the solvents. Naringin, eriocitrin, neoeriocitrin, and neohesperidin were identified as the major phenolic compounds in fresh bergamot juice and the extracts, comprising a significant proportion of the total phenolic content.

This study demonstrated that phenolic compounds could be effectively extracted from bergamot juice powder using ultrasound-assisted extraction with different solvents. ACTN was the most efficient solvent in terms of extraction efficiency, followed by DMSO, EtOH, and DW. The major phenolic compounds in fresh juice and extracts were naringin, eriocitrin, neoeriocitrin, and neohesperidin. Further research can be conducted to explore the potential applications of these extracts in functional foods or nutraceuticals, considering their antioxidant properties and phenolic content.

5 | CONCLUSION

In conclusion, the spray-drying process offers several advantages for obtaining functional bergamot juice powder with high antioxidant activity, total phenolic content, and vitamin C content. Due to its bioactive properties, this powder can be utilized in various industries, including food, cosmetics, and pharmaceuticals. Unlike bergamot juice, which is challenging to store for long periods while maintaining chemical, physical, and microbiological stability, spray-drying provides a viable solution for prolonging its shelf life.

Furthermore, the choice of solvent for polyphenol extraction from plant materials significantly affects the extraction efficiency. Ultrasound-assisted extraction has been demonstrated as an effective and efficient technique for obtaining bioactive compounds from plants. In this study, bergamot juice was first spray-dried to obtain the powder, and ultrasound-assisted extraction of polyphenols was performed using different solvents, including ACTN, EtOH, DMSO, and DW. The results indicated that the highest antioxidant activity, total phenolic content, vitamin C content, and individual phenolic components were obtained with solvents at 50% for ACTN, 100% for DMSO, and 50% for EtOH. ACTN was the most effective solvent, whereas DW was the least effective.

Extracts obtained from different parts of bergamot fruits have the potential to be used as natural additives in the production of functional and healthy foods due to

their rich polyphenol content. Moreover, the drying process significantly reduced bitterness in the bergamot juice extracts, further enhancing their potential for use in food applications.

In summary, combining spray-drying and ultrasound-assisted extraction techniques offers a promising approach for obtaining functional bergamot juice powder with enhanced bioactive properties. Further research and application of these methods in different industries are warranted.

AUTHOR CONTRIBUTIONS

Bahar Demircan: Conceptualization; Methodology; Software; Formal analysis; Investigation; Data curation; Writing—original draft; Validation. **Yakup Sedat Velioglu**: Conceptualization; Methodology; Investigation; Supervision; Funding acquisition; Project administration; Resources; Writing—review and editing; Validation. **Angelo Maria Giuffrè**: Conceptualization; Investigation; Resources; Data curation; Writing—review and editing; Visualization; Supervision; Project administration; Validation.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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