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Broccoli pomace: effect of drying methods and temperature on the grinding process and physicochemical properties**

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Abstract. The objective of this study was to evaluate the impact of different drying methods and temperatures on the physicochemical properties of broccoli pomace. The broccoli juice by-product was subjected to contact-drying at temperatures of 40°C (with microwave assistance at 50 W), 60, and 80°C as well as freeze-drying at hotplate temperatures of 20, 40, and 60°C, and then ground into powders. The drying kinetics of the pomace was assessed, along with measurements of water activity, moisture content, grinding efficiency, and energy consumption of pulverizing. Additional evaluations included powder color coordinates, total polyphenol content (determined using Folin-Ciocalteu reagent), antioxidant activity (measured using DPPH and ABTS assays), and a phytochemical analysis using LC-MS/MS. The results indicated that lyophilized broccoli pomace exhibited superior grindability, compared to contact-dried broccoli pomace. The freeze-dried powders were significantly brighter and greener than those obtained through contact-drying. Contact-drying, including microwave-assisted drying, resulted in increased phenolic compound content and enhanced antiradical activity, in comparison to freeze-drying. The phytochemical analysis revealed the highest concentrations of quinic acid and fumaric acid, along with trace amounts of aconitic acid, protocatechuic acid, piceid, coumarin, and astragalin. Based on the antioxidant properties and total polyphenol content, contact drying at 80°C was identified as the optimal method for drying broccoli pomace.

Keywords: by-product, broccoli waste, antioxidant activity, phenolic profile, freeze-drying

1. INTRODUCTION

Broccoli, a vegetable that has gained popularity worldwide, has seen a remarkable surge in production, escalating from an estimated 3.5 million metric tons in 1961 to 26.1 million metric tons in 2022 (FAOSTAT, 2024). This growing interest can be attributed (combined data for broccoli and cauliflower) to the numerous potential health benefits associated with broccoli consumption, stemming from its well-established antioxidant and chemopreventive properties (Gasmi *et al*., 2023). Various parts of the broccoli plant exhibit significant nutritional content, serving as a rich source of both protein and dietary fiber (Berndtsson *et al*., 2020; Li *et al*., 2022). Additionally, broccoli contains a diverse range of minerals, including magnesium, potassium, calcium, zinc, selenium, and iron, as well as essential vitamins such as vitamin C, vitamin K, vitamin E, and B vitamins (Nagraj *et al*., 2020). Moreover, all components of the plant, including florets, leaves, stalks, roots, seeds, and sprouts, serve as reservoirs of phenolic compounds and metabolites derived from glucosinolates, such as sulforaphane and indole-3-carbinol (Li *et al*., 2022).

The utilization of broccoli by-products carries considerable significance within the domains of sustainable agriculture, waste management, and closed-loop economy (Costa-Pérez *et al*., 2022). It is estimated that agricultural

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and industrial residues stemming from broccoli production, encompassing unconsumed parts, leaves, and stalks, account for 60-75 % of the total vegetable volume (Petkowicz and Williams, 2020). This underscores the urgent necessity for innovative strategies in addressing broccoli waste sustainably. Consequently, contemporary trends are increasingly focusing on leveraging broccoli waste as a constituent in feed formulations (de Evan *et al*., 2020; Quintero-Herrera *et al*., 2021), functional food applications (Krupa-Kozak *et al*., 2021; Salas-Millán *et al*., 2022), and the extraction of bioactive compounds (Cao *et al*., 2023). One of the products derived from broccoli is juice, packaged separately or intended as an addition to fruit juices (Sánchez-Vega *et al*., 2020), with pomace as the by-product.

Due to the high moisture content present in vegetable pomace and, consequently, the associated microbiological degradation (Abano *et al*., 2019), it is imperative to employ preservation methods to mitigate deterioration while safeguarding active compounds. Among various preservation techniques, drying is crucial for extending the shelf life of food products while maintaining their nutritional and functional properties. Various drying techniques, such as sun or shade drying, hot air drying, freeze-drying, microwave-assisted drying, spray drying, and vacuum drying, each uniquely affect the physicochemical properties of the material, including its moisture content, color, texture, and concentrations of individual bioactive compounds (Farahmandfar *et al*., 2020; Petikirige *et al*., 2022). Convective drying utilizes heated air to remove moisture from food products through moisture exchange with the air in the drying chamber (Calín-Sánchez *et al*., 2020). While this method offers such benefits as extended shelf life, operational simplicity, and cost-effectiveness, it also has drawbacks, including prolonged drying times, risk of oxidation, potential development of off-flavors, and crust formation due to high temperatures. Freeze-drying, or lyophilization, is an energy-intensive and time-consuming process that removes water through sublimation, preserving color, aroma, flavor, and nutritional content and producing high-quality products with excellent reconstitution properties and long shelf life (Bogusz *et al*., 2024). Microwave drying enhances drying efficiency and product quality by using microwave energy for effective heat transfer, reducing energy consumption and greenhouse gas emissions, while also improving water resorption, minimizing shrinkage, and increasing porosity in the dried product (Taghinezhad *et al*., 2023).

Despite these established effects, the impact of these drying techniques and their conditions on broccoli pomace remains largely unstudied. Therefore, this study aimed to explore the physicochemical properties of broccoli pomace (BP) focusing on the influence of drying methods and temperatures. The investigation encompassed analyses of drying kinetics, comminution susceptibility, energy consumption, particle size distribution, water activity and content, color, antioxidant activity, and the phenolic compound profile of the BP. This comprehensive examination sheds light on effective strategies for maximizing the utilization of broccoli by-products while maintaining their nutritional and functional properties.

2. MATERIALS AND METHODS

2.1. Raw material

Fresh broccoli at the consumer maturity stage was obtained from a local market. Juice was extracted from broccoli inflorescences using a twin-screw juicer (Angel 5500, Angel Juicers, South Korea). The raw material used for the study was the by-product of juice production, known as pomace.

2.2. Reagents

Analytical grade reagents including methanol (99.8%), ABTS (2,2′-azino-bis-(3-ethylbenzothiazoline-6-sulfonic acid), potassium persulfate, DPPH (1,1-diphenyl-2-picrylhydrazyl), gallic acid, sodium bicarbonate, Folin-Ciocalteu phenol reagent, and standard phenolic compounds for LC-MS/MS were obtained from Sigma (Sigma-Aldrich GmbH, Germany).

2.3. Contact-drying and freeze-drying conditions

Fresh BP (100 g samples rolled out to a thickness of 6 mm) was contact-dried in a convection dryer (Promis-Tech, Poland) at 40°C (with microwave assistance at 50 W), 60°C, and 80°C (±1°C). For the contact-drying process, the material, which was in the form of a pulp with moisture content of 59.18 ± 0.28%, was placed on plastic (polypropylene) trays (2 mm thickness). These trays were in direct contact with the drying agent. Metal trays were excluded due to the use of microwaves during drying at 40°C. Additionally, freeze-drying was performed using an Alpha 1-4 lyophilizer (Martin Christ Gefriertrocknungsanlagen GmbH, Germany) using plate heating at 20, 40, and 60°C (±2°C) under a pressure of 100 Pa. The freeze-dryer was modified to integrate the heating system with an LDC-1M driver (Martin Christ Gefriertrocknungsanlagen GmbH, Germany) and an electronic balance connected to the plates for mass recording. This system, which allows monitoring mass changes throughout the drying process with an accuracy of ±0.1 g, ensures precise measurement of sample mass (Rudy, 2009). The raw material designated for lyophilization was previously frozen in a freezing chamber (GTL-4905, Liebherr, Sweden) at -30°C. Based on the changes in mass during drying, the process was terminated once the final moisture content (wet basis) of the pomace reached between 5.1-5.4%. Additionally, after the drying process, the moisture content of the material was determined following the methodology described in Chapter 2.5.

2.4. Mathematical modeling of drying processes

During freeze-drying and contact-drying, changes in the weight of the drying material were continuously recorded at 5 min intervals. Drawing from the mass loss measurements recorded during the experiment, drying curves were derived as functions of the moisture ratio (*MR*) with respect to time (Kouhila *et al*., 2020). *MR* was defined based on equation:

$$MR = \frac{M_t - M_e}{M_o - M_e}, \quad (1)$$

where: M_t denotes the moisture content at time *t*, M_e represents the equilibrium moisture content, and M_o is the initial moisture content (Motevali *et al*., 2013). Since the value of M_e is negligible, compared to M_t and M_o, it was omitted from the equation, resulting in M_t / M_o. Subsequently, seven models commonly found in the literature and described in Table 1 were tested to select the most suitable model for describing the freeze-drying and contact-drying of BP.

Table 1. Mathematical drying models used for the freeze-drying and contact-drying of broccoli pomace

No.	Model	
1	Newton (Chabane *et al*., 2023)	$MR = \exp(-k\,\tau)$
2	Page (Page, 1949)	$MR = \exp(-k\,\tau^n)$
3	Henderson and Pabis (1961)	$MR = a \exp(-k\,\tau)$
4	Logarythmic (Isa *et al*., 2021)	$MR = a \exp(-k\,\tau) + b$
5	Wang and Singh (1978)	$MR = 1 + a\,\tau + b\,\tau^2$
6	Logistic (Kohli *et al*., 2022)	$MR = b\,((1 + a \exp(k\,\tau))^{-1}$
7	Midilli (2002)	$MR = a \exp(-k\,\tau^n) + b\,\tau$

k – drying coefficient (min^{-1}), τ – time (min), *n* – exponent, *a*, *b* – equation coefficients.

2.5. Water activity and moisture content of dried BP

The water activity of 2 g of dried BP was measured at a temperature of 22°C using a LabMaster (Novasina AG, CH-8853 Lachen, Switzerland). The moisture content was determined by drying at 105°C (SLN 53 STD/TOP+, POL-EKO-APARATURA, Poland) until the samples reached a stable mass. The moisture content (wet basis) was calculated as the difference in the mass of the sample before and after drying divided by the mass of the sample before drying.

2.6. Grinding energy and particle size analysis

The dried BP was ground using a knife mill GRINDOMIX GM-200 with a power of 1000 W at 10 000 rpm (Retsch, Germany). It was equipped with a computer system VC870 Interface 4.2.6. for recording and analyzing energy consumption during comminution (VOLTCRAFT®, Germany) (Dziki *et al*., 2023). Energy consumption during grinding for one minute was determined using a digital multimeter VC 870 (VOLTCRAFT®, Germany). The specific grinding energy was determined by the ratio of the grinding energy to the mass of the material being reduced in size, while the grinding efficiency index was determined by dividing the energy used for grinding by the surface area of the milled material (measured using the Malvern Mastersizer 3000 (Malvern Instruments Ltd., UK)) (Hassoon and Dziki, 2018). The aim of the study was evaluate the influence of quinoa seed moisture content (10, 12, 14, 16 and 18%).

The particle size distribution of the powder from BP was measured using laser light scattering with a laser particle size analyzer (Malvern Mastersizer 3000, Malvern Instruments Ltd., UK). A 5 g powdered sample was placed in the inlet chamber, and the particle size was measured using the laser diffraction method. For each sample, particle size distribution parameters d_{10}, d_{50}, and d_{90} were assessed, corresponding to particles with cumulative volumes of 10, 50, and 90%, respectively. Span, regarded as the size dispersion index, was calculated as the quotient of the difference between d_{90} and d_{10} divided by d_{50} (Dziki *et al*., 2020b).

2.7. Color coordinates

The color coordinates of the BP powders, including brightness (*L**), redness (*a**), and yellowness (*b**), were determined using a colorimeter NR20XE (Shenzhen Threenh Technology Co., China). When positive, the *a** coordinate denotes the transition from red to green, while negative values denote the transition from green to red (Lisiecka and Wójtowicz, 2021). Analogously, positive values of the *b** coordinate signify the transition from yellow to blue, whereas negative values signify the transition from blue to yellow.

2.8. Antioxidant activity and total phenolic content

One gram of ground BP was extracted in 5 ml of a methanol:water (1:1, v/v) mixture for thirty minutes, with stirring using a Multi Bio RS-24 rotator (Biosan Sia, Latvia). Subsequently, the samples were centrifuged for 5 min at 5000 rpm (LC8 3500 Benchmark, USA). The supernatant was collected, and the extraction procedure was repeated two more times, after which the extracts obtained were

combined and stored in darkness at 20°C. The method described by Lisiecka *et al*. (2021) was used to determine the total phenolic content, which was then quantified and expressed as milligrams of gallic acid equivalent (GAE) per gram of dry matter. The antioxidant activities against DPPH and ABTS radicals were evaluated following the protocol outlined by Krajewska *et al*. (2024). The antiradical activity was quantified as the EC_{50} index, representing the concentration of the extract required to achieve a 50% antioxidant effect (Liu Haonan *et al*., 2021).

2.9. Quantitative analysis of phytochemicals by LC–MS/MS

One gram of powdered plant material was introduced into a 15 ml centrifuge tube along with 10 ml of methanol and then underwent ultrasonic extraction for 3 h before being centrifuged. Afterward, the top solvent layer was filtered through a 0.2 µm string filter. The analysis of phytochemicals (aconitic acid, acacetin, apigenin, amentoflavone, astragalin, caffeic acid, catechin, chrysin, chlorogenic acid, cosmosiin, coumarin, cynarin, cyranoside, daidzein, daidzin, ellagic acid, epicatechin, epicatechin gallate, epigallocatechin, epigallocatechin gallate, ferulic acid, fisetin, fumaric acid, gallic acid, genistein, genistin, gentisic acid, hesperetin, hesperidin, isoquercitrin, kaempferol, luteolin, miquelianin, naringenin, nicotiflorin, o-coumaric acid, p-coumaric acid, piceid, protocatechuic acid, protocatechuic aldehyde, quercetin, quercitrin, quinic acid, rosmarinic acid, rutin, salicylic acid, sinapic acid, syringic acid, syringic aldehyde, tannic acid, vanillic acid, vanillin) in the methanolic extract of BP was performed with the UPLC-MS/MS technique, which had been previously established and verified (Yilmaz, 2020). The chromatography conditions were described by Krajewska *et al*. (2024).

2.10. Statistical analysis of data

All experiments were conducted in triplicate. To assess the data, one-way analysis of variance (ANOVA) was used, and statistical differences between the means were calculated using Tukey's test. All tests were conducted at a significance level of $\alpha = 0.05$. During the analysis of drying kinetics regression, the coefficient of determination (R^2), root mean square error (*RMSE*), and values of the Chi-square test (χ^2) were determined using the following formulas:

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(MR_{i,p}-MR_{ie})^2}{N}}, \quad (2)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{i,p}-MR_{ie})^2}{N-n}, \quad (3)$$

where: $MR_{i,p}$ represents the predicted reduced water content value, while $MR_{i,e}$ stands for the experimental reduced water content value. N denotes the total count of measurements, while n represents the quantity of parameters within the model equation. Statistica 13.0 software (StatSoft Inc., USA) was used to perform the calculations.

3. RESULTS AND DISCUSSION

3.1. Drying results

The initial moisture content in the pomace was 59.18 ± 0.28%, and the final water content for all samples was 5.26 ± 0.15%, corresponding to an average level of water activity in the tested samples of 0.210 ± 0.009% (Table S1). Figures 1A and 1B present a comparative analysis of experimental and predicted data pertaining (according to the Page model) to contact-drying and freeze-drying. The shortest drying duration for BP was observed in microwave-assisted contact-drying, specifically 120 min. Elevated microwave power correlates with heightened drying velocity and improved moisture diffusion, thereby curtailing the processing duration (Tepe and Tepe, 2020). Conversely, BP required the longest drying period during freeze-drying at the lowest temperature of 20°C (385 min). Generally, freeze-drying is acknowledged as a more energy-intensive procedure relative to conventional drying (Duan *et al*., 2016), which surpasses air-drying in duration (Zhang *et al*., 2023), owing to inadequate internal heat transfer within the product and low operational pressure during freeze-drying (Oyinloye and Yoon, 2020). Nevertheless, our experimentation involved heating the plates upon which the raw material was positioned. The adoption of freeze-drying resulted in a reduction of the drying time by over 41% (from 360 to 210 min), in comparison to contact-drying at an identical temperature of 60°C. The shorter freeze-drying time, compared to contact drying, in our study may be attributed to the direct contact of the bottom layer of the material with the metal heating plate. In contrast, during contact-drying, the material was placed on plastic trays, which have lower thermal conductivity. An increase in the freeze-drying temperature from 20 to 40°C led to a 16% reduction in time, while a reduction of 35% was noted at the rise from 40 to 60°C. Similarly, in a study on freeze-drying mushrooms (*Cordyceps militaris*), augmenting the temperature of heating plates from 40 to 70°C reduced the processing time by 37% (Wu *et al*., 2019). Amplifying temperatures during the secondary phase of freeze-drying (desorption drying) accelerates the drying course by necessitating additional energy to eliminate residual water content (Nowak and Jakubczyk, 2020). The selection of an optimal drying time necessitates a trade-off among drying duration, energy conservation, and the physical and chemical attributes of the targeted product.

Examining the mathematical facets of drying kinetics not only enhances the efficiency and cost-effectiveness of drying procedures but also improves product quality (Ross *et al*., 2020). The outcomes of regression analyses for the seven models utilized to characterize the contact-drying and freeze-drying kinetics of BP are delineated in

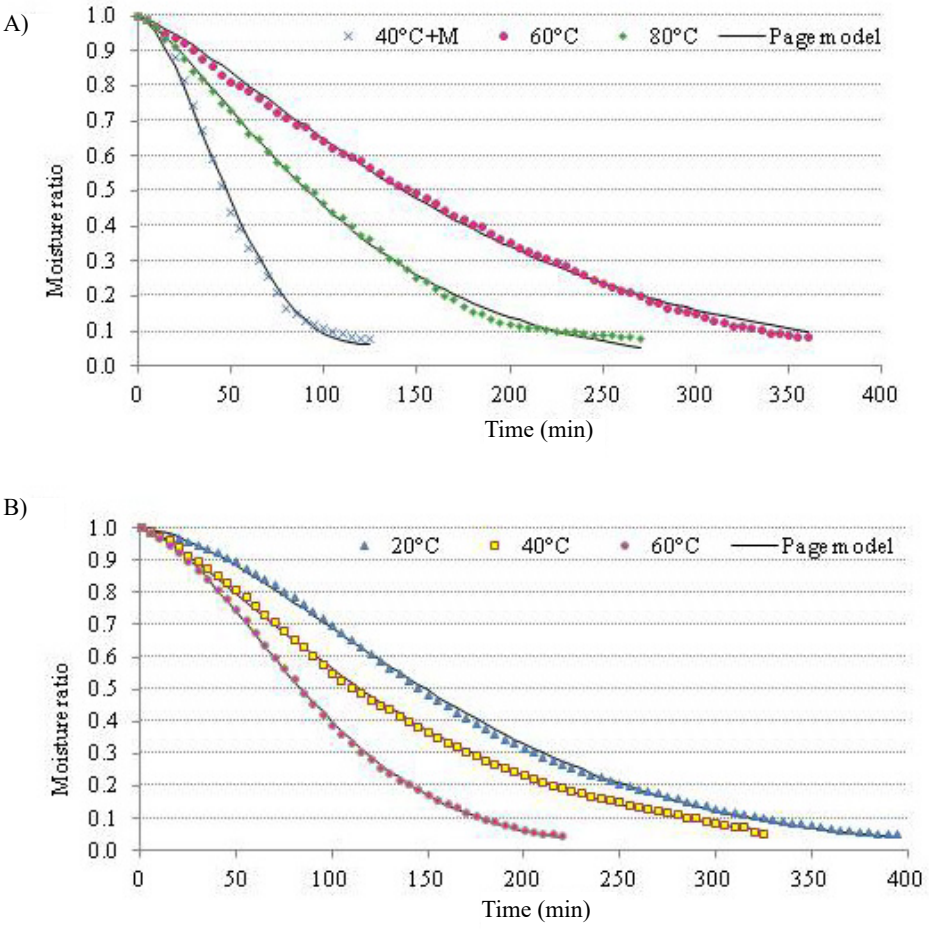

Fig. 1. Contact-drying curves (A) and freeze-drying (B) curves (experimental and predicted data according to the Page model).

Tables 2 and 3, while the coefficients of the equations are provided in Tables 4 and 5. Regarding contact-drying, a satisfactory fit was observed between all analyzed models and the experimental data. The most optimal fit within this drying method was identified for the Page model, yielding R^2 values ranging from 0.0994 to 0.0998, and *RMSE* ranging from 0.001 to 0.003. Concerning freeze-drying, a good fit was noted for six models, whereas the Logistic model exhibited lower R^2 values and higher *RMSE*, with insignificant differences between the coefficients ($p < 0.05$). Once again, the Page model emerged as the most suitable fit for BP freeze-drying, showing an R^2 value of 0.999 and an *RMSE* of 0.001. In the context of air-drying of eggplant (Akpinar and Bicer, 2005), freeze-drying alone and in combination with microwave vacuum drying of button mushrooms (Pei *et al*., 2014), or air-drying and freeze-drying of broccoli sprouts, the Page model also exhibited the best fit (Dziki *et al*., 2020a).

3.2. Particle size distribution and milling energy consumption

Food byproducts, such as BP, can undergo conversion into the powder form to enhance their utility in various food processing applications, including functional food enrichment, and use as colorants, natural flavorings, raw materials for food paste, or as inks for 3D food printers (Ahmed *et al*., 2020). The particle size of the additive plays a crucial role in determining texture properties, water distribution, protein structure, and sensory attributes of the final product (Zhang *et al*., 2019). Generally, freeze-drying made the BP more susceptible to grinding, compared to pomace dried by the contact method. It was evidenced by parameters such as d_{10}, d_{50}, and d_{90} (Table 6). At 60°C, the freeze-dried material exhibited a particle size distribution wherein 10% of particles possessed diameters equal to or smaller than 39.13 µm (d_{10}) and equal to or larger than 352.00 µm (d_{90}). BP subjected to contact-drying at the same temperature displayed a particle size distribution where 10% of particles had diameters equal to or smaller than 63.70 nm (d_{10}) and equal to or larger than 445.67 nm (d_{90}). During freeze-drying, water is removed through sublimation, which can result in the formation of porous and brittle material. These structural properties make the material more susceptible to fragmentation, potentially resulting in smaller particle sizes. Contact-drying, on the other hand, can produce more compact particles that are more difficult to break down into smaller fragments. Ahmed *et al*.

Table 2. Coefficient values, R^2, and root mean square error (*RMSE*) for the models describing the contact-drying process of BP

Model	Sample								
	40°C+MA			60°C			80°C		
	R^2	*RMSE*	χ^2	R^2	*RMSE*	χ^2	R^2	*RMSE*	χ^2
1	0.925	0.042	1.774×10^{-3}	0.973	0.018	3.162×10^{-4}	0.973	0.017	3.202×10^{-4}
2	0.994	0.003	1.027×10^{-5}	0.998	0.001	2.408×10^{-6}	0.998	0.001	1.477×10^{-6}
3	0.960	0.023	5.076×10^{-4}	0.984	0.011	1.121×10^{-4}	0.985	0.023	5.508×10^{-4}
4	0.976	0.013	1.788×10^{-4}	0.999	0.006	3.320×10^{-7}	0.993	0.004	1.847×10^{-5}
5	0.966	0.019	3.689×10^{-4}	0.999	0.001	2.508×10^{-7}	0.995	0.030	1.054×10^{-5}
6	0.844	0.088	7.730×10^{-3}	0.901	0.066	4.392×10^{-3}	0.893	0.087	7.656×10^{-3}
7	0.579	0.238	5.642×10^{-2}	0.803	0.132	1.735×10^{-2}	0.728	0.177	3.137×10^{-2}

Models: 1 – Newton, 2 – Page, 3 – Handerson and Pabis, 4 – Logarythmic, 5 – Wang and Singh, 6 – Logistic, 7 – Midilli, MA – microwaves assistance 50 W, R^2 – the determination coefficient, *RMSE* – mean-square error; χ^2 – chi-quadrate test.

Table 3. Coefficient values, R^2, and root mean square error (*RMSE*) for the models describing the freeze-drying process of BP

Model	Sample								
	20°C			40°C			60°C		
	R^2	*RMSE*	χ^2	R^2	*RMSE*	χ^2	R^2	*RMSE*	χ^2
1	0.941	0.053	2.786×10^{-3}	0.937	0.019	3.686×10^{-4}	0.939	0.042	1.774×10^{-3}
2	0.999	0.001	5.753×10^{-7}	0.999	0.001	1.807×10^{-7}	0.999	0.001	6.972×10^{-8}
3	0.971	0.026	6.723×10^{-4}	0.988	0.008	6.567×10^{-5}	0.966	0.023	5.333×10^{-4}
4	0.989	0.001	9.217×10^{-5}	0.996	0.003	8.615×10^{-6}	0.989	0.007	5.461×10^{-5}
5	0.994	0.015	2.270×10^{-4}	0.994	0.004	1.548×10^{-5}	0.984	0.011	1.133×10^{-4}
6	0.860	0.125	1.572×10^{-2}	0.898	0.071	5.083×10^{-3}	0.852	0.101	1.029×10^{-2}
7	0.825	0.157	2.461×10^{-2}	0.769	0.162	2.637×10^{-2}	0.738	0.179	3.220×10^{-2}

Explanations as in Table 2.

(2020) also reported that flour derived from freeze-dried green bananas (-47 to -50°C, 36 h) contained finer particles than flour obtained from tray-dried bananas (55°C, 15 h). The drying temperature of the raw material also influenced the particle size distribution of the powders. In the case of contact-drying, the elevation of temperature from 60 to 80°C led to a reduction in particle size, as evidenced by the decline in the values of d_{10} and d_{50} parameters. Moreover, the particle diameter of powder contact-dried at the lowest temperature with microwave assistance, wherein 10% of the total particle volume was situated, was lower than that of material contact-dried at 60°C, suggesting the presence of smaller particles. However, the d_{90} value of the microwave-assisted contact-dried pomace indicated the presence of the largest particles among the tested samples, possibly implying that the low temperature used during contact-drying yields inferior fragmentation. Concerning the freeze-dried material, the values of parameters d_{50} and d_{90} exhibited a decrease with the increase in the temperature of the heating plates. Higher drying temperatures can cause greater material shrinkage and structural changes, such as loosening or hardening, which may reduce particle size. Additionally, elevated temperatures accelerate water evaporation, yielding more uniform and smaller particles during grinding, particularly in contact drying. Specific surface area was notably higher for the freeze-dried powder at 60°C, compared to powder processed *via* contact-drying at the same temperature. Furthermore, as the temperature increased, specific surface area showed an incremental trend from 60 to 80°C in the case of contact-drying as well as with the incorporation of microwaves, and from 40 to 60°C in the case of freeze-drying. Both the drying method and temperature exerted a marginal effect on Span.

The majority of energy expended in the grinding process dissipates as heat, with only a fraction of the input energy, ranging from 0.06 to 1%, contributing to actual material size reduction (Pandurangappa *et al*., 2013). Energy consumption during shredding is contingent upon several variables, including the moisture content and hardness of the material, pre-treatment methods, material feed rate, and machine parameters (Mohd Rozalli *et al*., 2015). In our study, the moisture content of the material subjected to grinding remained at a similar level (5.26 ± 0.15%, Table S1), indicating that the differences in the grinding process were most likely due to the drying method and conditions. Freeze-drying, compared to traditional drying methods,

Table 4. Values of parameters in the models describing contact-drying process of BP

Drying conditions	Model	Coefficient			
		a	*k* (min^{-1})	*n*	*b*
40°C+MA	1		0.016889		
	2		0.001009	1.689149	
	3	1.176104	0.019906		
	4	1.395851	0.012752		-0.271588
	5	-0.012573			0.000038
	6	-0.999984	-0.000001		0.000020
	7	0.959900	17.30690	-49.0871	0.008500
60°C	1		0.005265		
	2		0.001055	1.307141	
	3	1.083323	0.005765		
	4	1.421000	0.003160		-1.396970
	5	-0.003971			0.000004
	6	-0.999989	-0.000001		0.000012
	7	0.921800	4.360400	18.89060	-0.002600
80°C	1		0.008439		
	2		0.001697	1.331499	
	3	1.103883	0.009354		
	4	1.21553	0.00694		-1.15198
	5	-0.006549			0.000011
	6	-0.999985	-0.000001		0.000018
	7	0.878000	1.675700	-20.13770	-0.003600

a, *b* – equation coefficients, *k* – drying coefficient (min^{-1}), *n* – exponent. Other explanations as in Table 2.

results in a dried product with a less compact structure that is more easily ground than the product obtained through traditional methods, such as convective or contact drying. In broad terms, the choice of the drying technique and temperature significantly influenced the energy intensity of the BP grinding process (Table 6). Raw material subjected to contact-drying with microwave assistance at 40°C exhibited the highest energy requirement for milling, as evidenced by the specific grinding energy parameter. However, a comparative analysis between samples subjected to contact-drying and those freeze-dried at 60°C revealed no substantial disparity in the energy demand for milling the material. Nevertheless, the grinding efficiency index values demonstrated an enhanced efficiency in the shredding process during freeze-drying, compared to contact-drying, particularly at elevated temperatures. The freeze-dried BP at a hotplate temperature of 60°C exhibited the highest grinding efficiency index value, whereas those subjected to contact-drying at temperatures of 40°C (with microwave assistance) and 60°C recorded the lowest values. A similar trend, albeit more pronounced, was observed in a preceding investigation concerning pear pomace. Freeze-dried pear pomace necessitated significantly less energy for grinding, and its grinding efficiency surpassed that of contact-dried pomace (Krajewska *et al*., 2024). Consequently, the energy intensity of the size reduction process is notably contingent upon the drying method employed for the plant material, with the specific attributes of the raw material further modulating the intensity of this relationship.

3.3. Color of BP powders

The color of BP powders exhibited variation contingent upon the drying method employed (Table 7). The raw materials were notably brighter and more greenish, as indicated by the higher values of the *L** parameter and the lower values of the *a** parameter when pomace was freeze-dried, compared to contact-dried pomace. Vargas *et al*. (2022) reached analogous conclusions, observing an augmentation in luminosity and a reduction in redness in lyophilized broccoli powders relative to their air-dried counterparts. Furthermore, the yellowness of the ground freeze-dried BP at 60°C was more intense, compared to the yellowness of the contact-dried powder at the same temperature.

The temperature of contact-drying also influenced the color parameters of powdered BP. Products dried at 40°C with microwave assistance appeared the darkest among all analyzed samples. Furthermore, both the redness and yellowness of contact-dried powders significantly increased with the rise in the drying temperature from 60 to 80°C as well as with the application of 40°C and microwave assistance. Considering that microwave-assisted drying leads to an increase in material temperature (Liu Haili *et al*., 2021),

Table 5. Values of parameters in the models describing FD process of BP

Temperature of heating plates	Model	Coefficient			
		a	*k* (min^{-1})	*n*	*b*
20°C	1		0.005379		
	2		0.000266	1.572706	
	3	1.614128	0.006291		
	4	1.412090	0.003772		-0.312904
	5	-0.003930			0.000004
	6	-0.999986	0.000001		0.000018
	7	0.94750	25.75690	-39.78740	-0.002700
40°C	1		0.006668		
	2		0.001253	1.331117	
	3	1.111288	0.007460		
	4	1.234482	0.005452		-0.164899
	5	-0.005151			0.000006
	6	-0.999985	0.000001		0.000018
	7	0.897000	15.57350	-32.48400	0.003000
60°C	1		0.009603		
	2		0.000564	1.605911	
	3	1.155486	0.011135		
	4	1.472220	0.006139		-0.385072
	5	-0.006915			-0.000011
	6	-0.999976	-0.000001		0.000029
	7	0.950400	2.940800	-18.92900	-0.004800

a, *b* – equation coefficients, *k* – drying coefficient (min^{-1}), *n* – exponent. Other explanations as in Table 2.

Table 6. Parameters describing grinding process of BP depending on drying method and conditions

DC (°C)	SA (m^2 kg^{-1})	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	Span	E_s (kJ kg^{-1})	E_f (m^2 kJ^{-1})
Contact-drying							
40+MA	54.85±2.77^{b}	55.50±3.70^{c}	211.67±5.13^{d}	473.33±18.15^{d}	1.98±0.04^{ab}	11.14±0.50^{c}	4.93±0.23^{ab}
60	46.98±0.31^{a}	63.70±0.78^{d}	208.00±2.65^{d}	445.67±7.02^{cd}	1.84±0.03^{a}	10.17±0.21^{ab}	4.62±0.10^{a}
80	55.94±0.50^{b}	53.50±0.78^{bc}	194.00±1.73^{c}	445.00±5.29^{cd}	2.02±0.04^{b}	10.29±0.32^{b}	5.44±0.17^{c}
Freeze-drying							
20	52.90±3.51^{ab}	50.80±1.05^{b}	185.33±1.15^{b}	417.33±14.01^{bc}	1.98±0.06^{ab}	9.95±0.13^{ab}	5.32±0.07^{bc}
40	53.79±4.76^{ab}	54.53±0.72^{bc}	179.67±2.52^{b}	393.00±19.29^{b}	1.89±0.11^{ab}	9.48±0.19^{a}	5.68±0.12^{c}
60	68.16±0.74^{c}	39.13±0.23^{a}	155.00±0.01^{a}	352.00±1.73^{a}	2.02±0.01^{b}	9.63±0.22^{ab}	7.08±0.16^{d}

DC – drying conditions, SA – specific surface area; d_{10}, d_{50}, d_{90} – express the 10th, 50th, and 90th percentiles of the overall volume, assuming the particles demonstrate a spherical shape; Span – the size dispersion index; E_s – specific grinding energy; E_f – shredding efficiency index; MA – microwaves assistance 50 W; the data is presented as mean ± SD; means marked with different letter superscripts indicate significant differences ($\alpha = 0.05$).

Table 7. Color coordinates of BP powders depending on drying method and conditions

Method	DC (°C)	*L**	*a**	*b**
Contact-drying	40+MA	55.45±0.14^{a}	4.47±0.16^{c}	24.39±0.19^{b}
	60	56.58±0.46^{b}	1.54±0.19^{b}	23.07±0.24^{a}
	80	56.59±0.26^{b}	5.51±0.24^{d}	25.54±0.68^{c}
Freeze-drying	20	62.31±0.21^{c}	-6.57±0.33^{a}	23.12±0.40^{a}
	40	62.76±0.30^{c}	-6.50±0.15^{a}	22.98±0.74^{a}
	60	62.11±0.43^{c}	-6.74±0.38^{a}	24.00±0.20^{b}

DC – drying conditions, *L** – lightness, *a** – redness, *b** – yellowness; MA – microwaves assistance 50 W; the values are presented as mean ± SD, and means labeled with different letter superscripts indicate significant differences (α = 0.05).

Table 8. Antioxidant activity of BP powders depending on drying method and conditions

Drying method	DC (°C)	DPPH	ABTS	TP
Contact-drying	40 +MA	38.39±0.48^{b}	55.97±0.32^{c}	2.65±0.01^{d}
	60	39.84±0.20^{c}	54.33±1.20^{c}	2.54±0.02^{c}
	80	36.03±0.06^{a}	46.96±1.16^{a}	3.24±0.02^{e}
Freeze-drying	20	42.11±0.52^{d}	50.07±0.96^{b}	1.84±0.05^{b}
	40	45.43±0.39^{e}	54.01±0.17^{c}	1.54±0.05^{a}
	60	46.57±0.35^{f}	58.79±1.24^{d}	1.53±0.02^{a}

DC – drying conditions; TP – total phenolic content expressed as milligrams of gallic acid equivalent (GAE) g^{-1} DW; DPPH – the capacity to neutralize DPPH radicals expressed as EC_{50} (mg DW ml^{-1}); ABTS – the capacity to neutralize DPPH radicals expressed as EC_{50} (mg DW ml^{-1}); MA – microwaves assistance 50 W; the values are presented as mean ± SD, and means labeled with different letter superscripts indicate significant differences (α = 0.05).

it can be hypothesized that the elevation of temperature in contact-drying of BP leads to an increase in the values of *a** and *b** coordinates. Mahn *et al*. (2011) also found that an increase in air temperature during drying results in a decrease in greenness. Moreover, temperature generally did not affect the color of lyophilized BP powders. Only yellow tones increased significantly when the highest lyophilization temperature was applied.

3.4. Total phenolic content and antiradical activity against DPPH and ABTS

Both the method and temperature of BP drying influenced the total phenolic compound content (TP) and antioxidant activity of the extracts (Table 8). Pomace dried through contact-drying exhibited higher TP and stronger antioxidant activity against DPPH and ABTS, compared to lyophilized samples. The TP of contact-dried pomace at 80°C was approximately 2.1 times higher than that of freeze-dried material at the highest temperature. While freeze-drying is generally regarded as a superior fixation method, compared to air-drying, in terms of preserving the antioxidant activity of plant material (Yap *et al*., 2020), some raw materials demonstrate higher antioxidant activity in hot-air-dried forms than in freeze-dried ones. For instance, hot-air-dried pumpkin at 70°C Que *et al*. (2008) exhibited 4.6 times higher TP than freeze-dried pumpkin, which also translated into antiradical activity against DPPH. Similarly, in the case of lemon pomace, higher TP was observed for air-dried material at 110 and 90°C, compared to freeze-dried material, with TP not significantly different only when compared with that air-dried at 70°C. In the case of BP powder, the enhanced antioxidant activity of contact-dried materials may stem from the prolonged exposure of the dried material to high temperatures, leading to cell wall rupture and increased free phenolic content (Jiang *et al*., 2019). Unlike the studies mentioned above, our study involved heating the plates during lyophilization. However, it can be assumed that this resulted in a significantly lower temperature at the top layer of the material not in contact with the heating plate, compared to contact-drying. Furthermore, air-drying at 60°C is more effective in polyphenol oxidase inactivation than freeze-drying (Li *et al*., 2018). Polyphenol oxidase catalyzes the conversion of phenolic compounds to quinones, leading to enzymatic

browning and phenol loss reactions. Another probable explanation for the heightened antioxidant activity of contact-dried pomace, compared to freeze-dried pomace, is the Maillard reaction, a non-enzymatic browning reaction between the carbonyl groups of sugars and the amino groups of amino acids or proteins (Murata, 2021), which intensifies in the presence of more oxygen and moisture during hot air drying (Fante and Noreña, 2015) and destruction of the cellular structure of the material, resulting in the release of chemical compounds, such as amino acids and reducing sugars (Yang *et al*., 2019). Maillard reaction products exhibit antioxidant properties in DPPH and ABTS assays (Kim *et al*., 2017). Additional evidence of the increased formation of Maillard reaction products in contact-dried BP powders, compared to freeze-dried BP ones, is the lower brightness, hence the lower value of the *L** parameter (Table 7). Given the above, the selection of the drying method is difficult to predict based on data from other fruits and vegetables and should be supported by experimental data specific to the particular raw material.

The antioxidant activity of BP powder was also influenced by the drying temperature. BP subjected to contact-drying at 80°C exhibited the highest TP, and correspondingly, the highest antioxidant activity against DPPH, as indicated by the EC_{50} parameter. Considering that the use of microwaves elevates the drying temperature, it can be inferred that, in hot air drying, both TP and antioxidant activity in the DPPH assay increase with rising temperature. Similar findings were reported by López *et al*. (2010), who observed enhanced quality of blueberries in terms of TP and DPPH activity at higher temperatures (80-90°C) during air-drying. In the ABTS assay, the most potent activity was detected for BP powders freeze-dried at a hotplate temperature of 20°C. Since the DPPH and ABTS assays operate based on different antioxidant mechanisms, their results do not necessarily converge (Mareček *et al*., 2017). Concerning freeze-drying, the most robust antioxidant activity was observed for BP dried at the lowest hotplate temperature. However, in the case of contact-drying, the pomace dried at the highest temperature (80°C) exhibited the highest TP and was most active against both DPPH and ABTS.

A significant negative correlation was identified between TP and antioxidant activity against DPPH, expressed as EC_{50} ($r = -0.9698$, $p = 0.000$), while no significant correlation was observed between TP and EC_{50} in ABTS ($r = -0.035$, $p = 0.888$) and between antioxidant activity against DPPH and ABTS ($r = 0.1371$, $p = 0.588$). The absence of a significant correlation between the results of the antioxidant tests may stem from the utilization of two distinct action mechanisms against different radicals (Rubio-Senent *et al*., 2013). Additionally, the differential solubility of BP extracts in distinct systems and the stereoselectivity of the radicals might impact their ability to react and quench individual radicals (Srividya Nayak, 2015).

3.5. Quantitative analysis of phytochemicals

A total of 10 phytochemicals were detected in BP powder among the 53 compounds tested (section 2.9.), which are presented in Table 9. The predominant substances were quinic acid and fumaric acid. Quinic acid, widely distributed in various plant parts, exhibits antioxidant, anti-cancer, anti-diabetic, or anti-microbial activities (Benali *et al*., 2022). Fumaric acid and its esters find extensive applications in the food, feed, pharmaceutical, and biomedical industries, serving as agents to enhance the sensory attributes of food and as antibacterial and antioxidant agents utilized in drug production for conditions like psoriasis or multiple sclerosis (Ilica *et al*., 2019). A study by Shi *et al*. (2019) also reported significant quantities of quinic acid in powder derived from freeze-dried BP (8.2 mg chlorogenic acid equivalents g^{-1} DW), while its content in our investigation ranged between 5.21 and 7.47 mg g^{-1} DW. These disparities could stem from the specific broccoli cultivar analyzed, harvesting timing, storage conditions, and the particulars of the drying process. In our study, the freeze-drying duration did not exceed 385 min (Fig. 1B), which was facilitated by the application of heated plates beneath the raw material, whereas the cited study involved a five-day drying period. Furthermore, the choice of solvent and extraction temperature can influence the quantification of individual phenolic compounds (Bucić-Kojić *et al*., 2009; Roselló-Soto *et al*., 2019). In contrast, our work did not detect quantifiable amounts of chlorogenic acid in BP powder, likely attributable to divergent methods for juice separation from pomace and subsequent extraction. The rise in quinic acid content may inversely correlate with chlorogenic acid levels in the sample, as elevated temperatures degrade chlorogenic acid into quinic acid (Milić *et al*., 1968). Throughout the investigation, the quinic acid content remained relatively stable; however, a marked increase was observed specifically with freeze-drying at the lowest temperature. Similarly, in the research by Adamczak *et al*. (2009), quinic acid content was substantially higher in freeze-dried cranberry fruit (without heated plates), compared to air-dried fruit at 35-40°C. Moreover, trace amounts of aconitic acid, protocatechuic acid, piceid, coumarin, and astragalin were identified in BP powder. Additionally, trace levels of syringic aldehyde were detected in contact-dried and freeze-dried material at all temperatures, but not in contact-dried samples with microwaves or at 60°C. Similarly, trace amounts of salicylic acid were found in contact-dried BP and in freeze-dried samples at 40°C. Moreover, acacetin was only detected in lyophilisates dried at hotplate temperatures of 20 and 60°C.

4. CONCLUSIONS

The shortest drying time for broccoli pomace was achieved with contact-drying at 40°C assisted by microwaves. The lyophilization of the raw material using hot plates required less time than contact drying at the same

Table 9. Identified phytochemicals in BP powder utilizing LC–MS/MS screening (mg g^{-1} DW)

Analytes	Contact-drying			Freeze-drying		
	40°C +MA	60°C	80°C	20°C	40°C	60°C
QA	5.21±0.11^{a}	5.28±0.48^{a}	5.69±0.51^{a}	7.47±0.75^{b}	5.90±0.41^{a}	6.24±0.31^{ab}
FA	0.171±0.010^{c}	0.146±0.009^{ab}	0.211±0.011^{d}	0.142±0.003^{a}	0.165±0.003^{bc}	0.163±0.007^{abc}
AAC	0.00500±0.00005^{c}	0.00700±0.00014^{d}	0.00800±0.00016^{e}	0.00200±0.00002^{a}	0.00300±0.00015^{b}	0.00300±0.00018^{b}
PA	0.00200±0.00008^{a}	0.00200±0.00002^{a}	0.00200±0.00016^{a}	0.00400±0.00036^{b}	0.00700±0.00063^{d}	0.00600±0.00012^{c}
SA	nd	nd	0.00300±0.00003^{a}	0.00400±0.00024^{b}	0.00300±0.00015^{a}	0.00300±0.00015^{a}
P	0.00200±0.00002^{a}	0.00200±0.00006^{a}	0.00200±0.00010^{a}	0.00700±0.00035^{b}	0.00200±0.00004^{a}	0.00200±0.00010^{a}
C	0.0330±0.0030^{c}	0.0330±0.0017^{c}	0.0310±0.0006^{bc}	0.0190±0.0021^{a}	0.0310±0.0009^{bc}	0.0280±0.0000^{b}
SAA	0.00200±0.00002^{b}	0.00400±0.00012^{c}	0.00400±0.00016^{c}	nd	0.00100±0.00001^{a}	nd
AS	0.0070±0.0003^{a}	0.0200±0.0008^{c}	0.0140±0.0001^{b}	0.0130±0.0013^{b}	0.0070±0.0001^{a}	0.0070±0.0002^{a}
ACA	nd	nd	nd	0.00200±0.00002^{b}	nd	0.00100±0.00002^{a}

QA – quinic acid, FA – fumaric acid, AAC – aconitic acid, PA – protocatechuic acid, SA – syringic aldehyde, P – piceid, C – coumarin, SAA – salicylic acid, AS – astragalin, ACA – acacetin, MA – microwaves assistance 50 W, nd – not detected, the values are presented as mean ± SD, and means labeled with different letter superscripts indicate significant differences (α = 0.05).

temperature (60°C). The Page model best described the freeze-drying and contact-drying processes of broccoli pomace.

The freeze-dried broccoli pomace was more susceptible to grinding than the contact-dried one. The lyophilized powders exhibited a markedly brighter and greener hue than powders derived from contact-dried broccoli pomace. While the temperature used during contact-drying significantly affected the color parameters, its impact was marginal during freeze-drying.

The contact-drying of broccoli pomace, including microwave-assisted drying, increased the content of phenolic compounds and antiradical activity, compared to freeze-drying. The vegetable powders demonstrated a diverse phytochemical profile, being most abundant in quinic and fumaric acids, and containing trace amounts of aconitic acid, protocatechuic acid, piceid, coumarin, and astragalin.

Considering the antioxidant properties and total polyphenol content, contact-drying at 80°C was found to be the optimal method for drying broccoli pomace.

Conflicts of Interest: The Authors do not declare any conflict of interest.

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