






## PAPER

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# Production of chokeberry pulp powder by convective and freeze-drying foam-mat techniques: effects on physicochemical properties, bioactive content, and antioxidant activity†

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Chokeberry has gained popularity due to its high anthocyanin content. However, its high-water content shortens its shelf life, leading to potential waste, while its high pectin content limits its application as a food ingredient. A viable approach is using mature fruits to produce bioactive-rich pulp powder as a functional food ingredient. This study investigated the production of chokeberry fruit pulp powder by foam-mat drying, for the first time, via freeze and convective drying at different temperatures (50, 60, and 70 °C) and evaluate it in terms of powder properties, bioactive content, and antioxidant activity. Regarding the color of the produced powder, the lowest redness and highest yellowness values were observed in the freeze-dried powder. Bulk density, tap density, and the Carr index were found to be higher in convectively dried products. Freeze drying produced a powder with the highest bioactive content, including phenolic ( $41.14 \pm 0.22 \text{ mg g}^{-1}$ ), flavonoid ( $6.03 \pm 0.12 \text{ mg g}^{-1}$ ), and anthocyanin ( $4.42 \pm 0.27 \text{ mg g}^{-1}$ ) compounds, as well as the strongest antioxidant activity. Although convective drying reduced bioactive content, drying at 50 °C preserved key properties, making it a cost-effective alternative. The two-term model at 50 °C best described the drying kinetics, further supporting CD 50 °C as an efficient option with favorable powder characteristics.

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## Sustainability spotlight

Post-harvest loss of perishable fruits like chokeberry contributes to global food waste and underutilization of nutrient-rich crops. This study offers a sustainable solution by transforming mature chokeberries into functional powders using foam-mat drying with freeze and convective methods. The resulting powders retain high levels of bioactives and antioxidant activity, supporting health-oriented food systems. By valorizing excess produce into shelf-stable, health-promoting ingredients, this work supports UN SDG 3 (Good Health and Well-being), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action) through waste reduction, efficient resource use, and food system resilience.

## 1 Introduction

Black chokeberry (*Aronia melanocarpa* L.) is a perennial shrub belonging to the Rosaceae family.<sup>1</sup> The fruit, which grows in

clusters, reaches maturity in late August and September. While there are varying accounts of the chokeberry's origin, it is believed to have originated in eastern North America before spreading to Eastern Europe and Russia.<sup>2</sup> Chokeberry is one of the richest natural sources of polyphenols, including phenolic acids, flavonoids, and anthocyanins.<sup>1</sup> The total phenolic content of chokeberry fruit can reach 24.7 g/100 g dry mass (dm),<sup>3</sup> this is greater than that of other berries such as blueberries (1.69 g/100 g dw),<sup>4</sup> strawberries (2.2 g/100 g dm)<sup>5</sup> and blackberries (1.74–3.57 g/100 g dm).<sup>6</sup> Analysis of dried chokeberry fruit powder has shown that it contains high levels of cyanidin-3-O-galactoside (8286 mg/100 g dm) and cyanidin-3-O-arabinoside (3329 mg/100 g dm), which are considered the most abundant polyphenols in chokeberry and belong to the anthocyanin class.<sup>3</sup>

Due to its exceptionally high antioxidant capacity, chokeberry consumption helps reduce oxidative stress and

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inflammation associated with chronic diseases<sup>7</sup> as well as intensive exercise.<sup>8</sup> For instance, the chokeberry fruit extract demonstrates anti-inflammatory effects in human aortic endothelial cells by suppressing the expression of endothelial cell adhesion molecules, inhibiting NF- $\kappa$ B activation, and reducing the production of reactive oxygen species (ROS).<sup>9</sup> Additionally, prolonged consumption of polyphenol-rich chokeberry juice by healthy adults has been shown to significantly increase levels of C22:6n-3 (DHA), total n-3 polyunsaturated fatty acids (PUFAs), total PUFAs, and the unsaturation index, while reducing monounsaturated fatty acids (MUFAs), the n-6 : n-3 ratio and levels of thiobarbituric acid-reactive substances (TBARS) – a biomarker of lipid peroxidation, suggesting a positive effect on lipid status.<sup>10</sup> Furthermore, it significantly increased the activity of antioxidant enzymes – superoxide dismutase (SOD) and glutathione peroxidase (GPx), which suggests that it could protect against cellular oxidative damage.<sup>10</sup> Chokeberry has also shown promising anti-diabetic, anti-atherosclerotic, antiviral, and antimutagenic properties.<sup>7,11</sup> Besides, it exhibits immunomodulatory, anti-proliferative, and anti-carcinogenic effects.<sup>11,12</sup>

Chokeberry fruits have a naturally sour and bitter taste, primarily due to their high polyphenol content, particularly strongly polymerized proanthocyanidins.<sup>13</sup> Procyanidin oligomers exhibit a high affinity for proteins, leading to protein denaturation, which contributes to sensations of sourness, astringency, and dry mouth.<sup>14</sup> Fresh, unprocessed chokeberries are not commonly consumed due to their intense sourness; however, they are extensively utilized in the food industry for producing beverages (juices, nectars, wines, and syrups), fruit-based products (jams, preserves, fruit desserts, and jellies), and dietary supplements (fruit teas and other formulations).<sup>1</sup> Additionally, due to its high anthocyanin content, chokeberry extracts can serve as a natural food coloring and a key ingredient in antioxidant-rich, health-promoting fruit juices, teas, and liqueurs.<sup>15</sup> Incorporating chokeberry powder into the development of functional foods, such as pomace-enriched white bread,<sup>16</sup> sweetened shortcrust pastries,<sup>17</sup> and wheat bread partially substituted with black chokeberry,<sup>18</sup> has demonstrated good consumer acceptability.

Chokeberries have a moisture content of  $\approx 80\%$ , making them highly perishable and prone to rotting, leading to significant waste.<sup>19</sup> Drying is widely used to extend the shelf life of fruits and incorporate them into various formulations as ingredients. Dried fruits with high sugar content are often very difficult to grind into powder.<sup>20</sup> Drying viscous foods with high sugar content is challenging due to the stickiness of the dried sample. Therefore, foam-mat drying technology has become popular among fruit processing units.<sup>21</sup> The principle of foam-mat drying involves creating foam by whisking the mixture together with foaming agents.<sup>22</sup> Whisking incorporates air, increasing the surface area for effective drying, while the foaming agent helps stabilize the foam during drying. Water vapor escapes from the foam through channels formed between the air cells, increasing the drying rate. This method provides the appropriate heat required for steam formation from foam. Foam-mat drying is a simple and alternative method for removing moisture from juices and purées.<sup>23</sup> The use of suitable

additives such as starch, maltodextrin, glycerol monostearate, propylene glycerol monostearate, carboxymethyl cellulose (CMC), and trichloro phosphate can minimize the stickiness problem and improve the hygroscopic properties of the powder. Foam-mat drying is an efficient, time-saving, and cost-effective method compared to other drying processes.<sup>23</sup> It is particularly suitable for drying heat-sensitive, sticky, and viscous food products that are difficult to dehydrate using other methods.

Incorporating chokeberry into food products presents challenges due to its high pectin content, which increases juice viscosity,<sup>24</sup> as well as its thick skin and high fiber content,<sup>25</sup> which hinder efficient juice extraction. Additionally, the instability of phenolic compounds and anthocyanins further complicates processing and product formulation. Thus, to turn this valuable and nutrient-rich fruit into a compatible food ingredient for functional foods, this study investigated the production of chokeberry pulp powder by foam-mat drying using two different drying methods: freeze-drying (effective in preserving heat-sensitive compounds such as anthocyanins but an expensive technique) and convective drying (a more economical method but operating at relatively higher temperatures). The effects of these methods on physicochemical properties, bioactive content (total phenolic, total flavonoid, and total anthocyanin content), and *in vitro* antioxidant activity were investigated.

## 2 Materials and methods

### 2.1. Samples

Fully mature black chokeberry (*Aronia melanocarpa*) fruits (1.5 kg) were sourced from local growers in the Bafra district of Samsun (41.5620° N, 35.9057° E) in September 2023. The fruits were washed, drained, and stored at  $-18\text{ }^{\circ}\text{C}$  in polyethylene bags. Before fruit pulp preparation, the black chokeberry berries were thawed overnight at  $4\text{ }^{\circ}\text{C}$ . The fruits were then crushed using a household blender and passed through a 3.5 mm metal sieve to obtain the pulp and prepare them for foaming. The prepared fruit pulp was divided into three replicates for each drying process before proceeding with the foaming step.

### 2.2. Technological processing

**2.2.1. Foam preparation.** Foamed chokeberry pulp was prepared by incorporating foaming agents and a foam stabilizer into the pulp, followed by a foaming process. The foaming agent and foam stabilizer were added at room temperature, and the mixture was blended using a household blender for 5 minutes to generate foam. Based on preliminary experiments, a foaming agent (fresh egg white (eggs sourced from local farmers), 10%), foam stabilizer (carboxymethyl cellulose, 1.0%), and maltodextrin (12%) were used to obtain stable foam. Preliminary experiments tested different proportions of each foaming agent and selected the foam formulations that achieved high stability and expansion while maintaining low foam density.

#### 2.2.2. Drying process

**2.2.2.1 Convective drying.** The foamed black chokeberry pulp was dried using hot air at a constant air speed of  $1\text{ m s}^{-1}$  at



varying temperatures of 50, 60, and 70 °C. The drying process was conducted in a convective cabinet dryer (E-GK1, EKSIS Endüstriyel Kurutma Sistemleri, Turkey) as described in our previous study.<sup>26</sup> The foamed pulp was spread in a 0.5 mm thick layer on a stainless-steel tray and placed in the drying cabinet. Drying continued until the foams reached a constant mass. During the drying process, mass measurements were taken every 15 min, and the drying process was considered complete when the difference between the last three weight measurements reached a stable state. Additionally, the final moisture content was determined to verify the completion of the drying process.

**2.2.2.2 Freeze drying.** The trays were first stored at −18 °C overnight before being transferred to a laboratory-scale freeze dryer (Labconco FreeZone 12 Plus, Labconco Corporation, USA) the following day. The freeze dryer operated at −80 °C and 13 Pa for 48 hours.

A summary of the main stages of powder preparation is provided in Fig. 1.

**2.2.3. Assessment of drying kinetics.** To identify the most suitable model for describing the drying behavior of chokeberry foam, drying curves were fitted using 12 thin-layer drying models, including the Aghabashlo model, Henderson and Pabis model, logarithmic model, logistic model, Midilli model, modified Midilli model, Newton model, Page model, two-term model, two-term exponential model, and Wang and Singh model (Table 1).<sup>27</sup> Data fitting was performed using MATLAB (R2016d, MathWorks, USA) with the curve fitting toolbox and the Levenberg–Marquardt algorithm. The quality of fit for each model was assessed based on the coefficient of determination ( $R^2$ , eqn (1)), reduced chi-square ( $\chi^2$ , eqn (2)), and root mean square error (RMSE, eqn (3)). The model that exhibited the highest  $R^2$  and the lowest  $\chi^2$  and RMSE values was considered the most appropriate for characterizing the thin-layer drying behavior of chokeberry foam.<sup>28</sup>

$$R^2 = \frac{\sum_j^n (MR_{\text{exp},i} - MR_{\text{pre}})^2 - \sum_j^n (MR_{\text{exp},i} - MR_{\text{pre},i})^2}{\sum_j^n (MR_{\text{exp},i} - MR_{\text{pre}})^2} \quad (1)$$

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{\text{exp},i} - MR_{\text{pre},i})^2}{N - n} \quad (2)$$

$$\text{RMSE} = \sqrt{\frac{\sum_j^n (MR_{\text{exp},i} - MR_{\text{pre},i})^2}{n}} \quad (3)$$

Drying curves were generated by plotting dimensionless moisture ratio (MR) as a function of time. The relative moisture ratio was calculated using eqn (4):

$$\text{MR} = \frac{(M_t - M_e)}{(M_i - M_e)} \quad (4)$$

where  $M_t$  is the moisture content at any given time during the drying process,  $M_i$  is the initial moisture content, and  $M_e$  is the equilibrium moisture content (db) (kg water/kg dry solid).  $M_e$  refers to the amount of moisture a sample retains when it reaches equilibrium with the surrounding air during the drying process. To determine the  $M_e$  value, samples were kept under specific temperature and relative humidity conditions, and weight changes were monitored.  $M_e$  was identified at the point where the weight change stabilized.

### 2.3. Characterization and quality assessment

**2.3.1. Foam properties.** The foam properties, including foam density, foam expansion, and foam stability, were determined.<sup>27</sup> Foam expansion (in %) was assessed by calculating the percentage change in volume before and after foam formation (eqn (5)). Foam density (in g cm<sup>−3</sup>) was measured by transferring the foam to a measuring cylinder, recording its volume and weight, and calculating their ratio (eqn (6)). Foam stability (in %) was evaluated by placing the foam in a measuring cylinder at room temperature for 3 h, with volume reduction recorded at 30 min intervals (eqn (7)).

$$\text{Foam expansion}(\%) = \frac{\text{volume after expansion} - \text{initial volume}}{\text{initial volume}} \times 100 \quad (5)$$

$$\text{Foam density} = \frac{\text{mass of foam}}{\text{volume of foam}} \quad (6)$$

$$\text{Foam stability}(\%) = \frac{\text{final volume}}{\text{initial volume}} \times 100 \quad (7)$$

**2.3.2. Powder properties.** The color of samples in terms of  $L^*$  (lightness/darkness),  $a^*$  (redness/greenness), and  $b^*$  (yellowness/blueness) was measured using a colorimeter (MiniScan EZ 4500, HunterLab, USA). Moisture content (using the gravimetric method in accordance with AOAC standard 934.06, which involves drying the samples under vacuum at 70 °C until a constant weight is achieved), water activity (using AquaLab 4 TE, METER Group, Inc., USA), pH (using Orion 3-Star, Thermo Fisher Scientific, USA), and solubility of all the

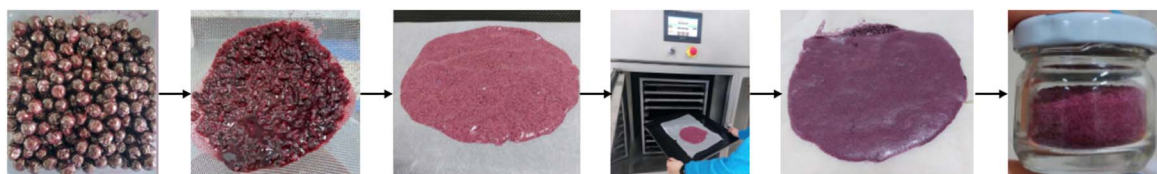


Fig. 1 Key preparation stages of foam-mat dried chokeberry pulp powder.



Table 1 Drying models and their respective equations

Model	Model equation
Aghabashlo	$MR = e^{-k \times t^n}$
Approximation of diffusion (diffusion approach)	$MR = a \times e^{-k \times x} + (1 - a) \times e^{-k \times b \times t}$
Henderson and Pabis	$MR = a \times e^{-k \times t}$
Logarithmic	$MR = a \times e^{-k \times t} + b$
Logistic	$MR = \frac{1}{(1 + a \times e^{-k \times t})}$
Midilli	$MR = a \times e^{-k \times t} + b \times t$
Modified Midilli	$MR = a \times e^{-k \times t} + b \times t^n$
Newton	$MR = e^{-k \times t}$
Page	$MR = e^{(-kt^n)}$
Two-term	$MR = a \times e^{-k_1 \times t} + b \times e^{-k_2 \times t}$
Two-term exponential	$MR = a \times e^{-k \times t} + (1 - a) \times e^{-k \times a \times t}$
Wang and Singh	$MR = 1 + a \times e^{-k \times t}$

produced powder samples were determined. For solubility analysis, 100 mL of distilled water was added to a blender jar, and 1 g of powder was gradually introduced while mixing at 1500 rpm. After 5 min of blending, the mixture was centrifuged at 4300 g for 10 min. A 25 mL aliquot of the resulting supernatant was transferred to Petri dishes and dried in an oven at 105 °C for 5 h. The solubility percentage was then calculated using eqn (8), where  $m_2$  represents the final mass of the Petri dish with the dried sample and  $m_1$  is the mass of the empty dish.

$$\text{Solubility}(\%) = \frac{m_2 - m_1}{0.25} \times 100 \quad (8)$$

In addition, browning index (BI) was also determined.<sup>29,30</sup> BI was calculated based on the absorbance readings obtained at 510 nm ( $A_{510}$ ), corresponding to anthocyanins, and at 420 nm ( $A_{420}$ ), corresponding to browning products, using a UV/vis spectrophotometer (LAMBDA™ 365, PerkinElmer, USA). To minimize variations in BI due to differences in anthocyanin content, measurements were conducted under acidic conditions (pH < 7.0). The BI was calculated using eqn (9):

$$\text{Browning index} = \frac{A_{510}}{A_{420}} \quad (9)$$

The physical properties of the powder including bulk and tapped density, as well as flowability (Hausner ratio (HR) and Carr index (CI)), were determined for all the produced powders.<sup>27,31</sup> Bulk density was measured by pouring 1 g of each powder into a 25 cm<sup>3</sup> graduated cylinder from top to bottom and recording the occupied volume. Bulk density was then calculated based on eqn (10) (in g cm<sup>-3</sup>). Tapped density was assessed by tapping the cylinder containing the sample 80 times to determine the effect of impact on the powder volume. Tapped density was then calculated based on eqn (11) (in g cm<sup>-3</sup>).

$$\text{Bulk density (g cm}^{-3}\text{)} = \frac{\text{mass of powder sample}}{\text{initial volume of powder sample}} \quad (10)$$

$$\text{Tapped density (g cm}^{-3}\text{)} = \frac{\text{mass of powder sample}}{\text{final volume of powder sample}} \quad (11)$$

HR and CI were also calculated using eqn (12) and (13), respectively.

$$\text{Hausner ratio} = \frac{\text{tapped density}}{\text{bulk density}} \quad (12)$$

$$\text{Carr index}(\%) = \frac{(\text{tapped density} - \text{bulk density})}{\text{tapped density}} \times 100 \quad (13)$$

Morphological analysis using scanning electron microscopy (JSM-7001F Schottky Emission Scanning Electron Microscope, JEOL Ltd, Japan) and Fourier-transform infrared (FTIR) (Spectrum Two, PerkinElmer, USA) analysis were also performed.<sup>27</sup>

## 2.4. Analysis of phenolic, flavonoid and anthocyanin content

The total phenolic content (TP), total flavonoid content (TF), and total monomeric anthocyanin content (TAC) were determined spectrophotometrically using a UV/vis spectrophotometer (LAMBDA™ 365, PerkinElmer, USA) following the methods reported in our previous study.<sup>27</sup> The TP, TF, and TAC were reported as gallic acid equivalents (GAE) mg GAE per g dm, quercetin equivalents (QE) mg QE per g dm, and cyanidin-3-glucoside equivalents (CGE) mg CGE per g dm, respectively.

## 2.5. Analysis of antioxidant activity

The antioxidant activity of the samples was tested by 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging and ferric-reducing antioxidant power (FRAP) assay, using a UV/vis spectrophotometer (LAMBDA™ 365, PerkinElmer, USA), and reported as mmol Trolox equivalent (TE) per g dm and mmol FeSO<sub>4</sub> equivalent (ISE) per g dm, respectively. These analyses were conducted according to the same methodology reported in our research investigating the production of cornelian cherry pulp powder by foam-mat drying.<sup>27</sup>

## 2.6. Statistical analysis

All experiments were conducted in triplicate. Statistical analysis was performed using IBM SPSS Statistics (V22.0, International Business Machines (IBM) Corporation, USA). Analysis of variance (ANOVA) followed by Duncan's multiple range test at 95% confidence level was performed to identify possible variations between groups. Differences were considered statistically significant when the *p*-value was less than 0.05.

# 3 Results and discussion

## 3.1. Properties of fresh chokeberry pulp

The physicochemical properties of chokeberry pulp were examined before any drying treatment. The pulp had a moisture content of 81.06 ± 1.09%, total soluble solids of 8.00 ± 0.09%, and a pH of 3.70 ± 0.10. The *L*\*, *a*\*, and *b*\* values of the pulp were 44.29 ± 0.41, 7.94 ± 0.20, and 19.99 ± 0.31, respectively. As





reported previously,<sup>1</sup> the composition and properties of chokeberry may vary depending on factors such as variety, maturity, and environmental and climatic conditions. They reported the dry matter and soluble solid content of chokeberry berries as 15.30–30.76% and 8.9–22.9%, respectively. It has been reported that the dry matter and pH values of chokeberry fruit can range from 15.6 to 28.8% and from 3.3 to 3.7, respectively.<sup>32</sup>

### 3.2. Foaming stability

Foam stability is crucial in the foam-drying technique to ensure product quality and rapid drying. During foaming, excess air is incorporated into the mixture, leading to foam expansion while reducing its density.<sup>30</sup> Lower foam density facilitates faster drying by increasing the available drying surface area. However, foams are thermodynamically unstable,<sup>23</sup> and their stability determines their ability to retain air. In this study, egg white, maltodextrin, and carboxymethyl cellulose were used to create

and stabilize foam. The additives, selected based on preliminary studies, were aimed at optimizing foam stability. The foam stability was measured at  $99.00 \pm 1.00\%$  (after 3 hours), foam expansion was  $75.00 \pm 1.00\%$ , and foam density was  $0.57 \pm 0.02 \text{ g cm}^{-3}$ . Previous studies have reported similar trends. The foam drying in apple juice using methylcellulose and egg white has been examined,<sup>33</sup> and it has been found that foam stability increased with the addition of these agents, with 0.2% methylcellulose and 2–3% egg white yielding the highest foam texture. Similarly, a study on black rice bran anthocyanin drying found foam density to be  $0.31 \text{ g mL}^{-1}$  and foam stability at 79.3%. A previous study<sup>34</sup> successfully produced foam from beet pulp using egg white and fish gelatine as foaming agents. Their results showed that foaming agent concentration (5–10%) influenced foam properties, with foam expansion ranging from 40.07 to 87.78% and foam density varying between 0.54 and  $0.72 \text{ g cm}^{-3}$ . These findings align with the foam properties observed in our study.

**Table 2** Statistical parameters of drying models used to produce foam-mat dried chokeberry powder by convective drying at various temperatures (50, 60, and 70 °C)<sup>a</sup>

Model name	Drying temperature (°C)	Statistical parameters		
		$R^2$	RMSE	$\chi^2$
Aghabashlo	50	0.999967	0.002341	0.000005
	60	0.984551	0.055147	0.003041
	70	0.985751	0.053836	0.002898
Approximation of diffusion	50	0.999726	0.011653	0.000136
	60	0.983018	0.100144	0.010029
	70	0.987108	0.088695	0.007867
Henderson and Pabis	50	0.993234	0.033412	0.001116
	60	0.982705	0.058349	0.003405
	70	0.959441	0.090827	0.008250
Logarithmic	50	0.999727	0.008221	0.000067
	60	0.983046	0.070756	0.005006
	70	0.987108	0.062715	0.003933
Logistic	50	0.993231	0.040931	0.001675
	60	0.987982	0.059571	0.003549
	70	0.978165	0.081620	0.006662
Midilli	50	0.999932	0.005785	0.000033
	60	0.999629	0.014800	0.000219
	70	0.987779	0.086353	0.007457
Modified Midilli	50	0.999944	0.005275	0.000027
	60	0.999944	0.005766	0.000033
	70	0.985895	0.092772	0.008607
Newton	50	0.992731	0.029992	0.000899
	60	0.982049	0.051481	0.002650
	70	0.958422	0.079640	0.006343
Page	50	0.999718	0.006824	0.000046
	60	0.988359	0.047872	0.002292
	70	0.975914	0.069993	0.004899
Two-term	50	0.999976	0.003443	0.000011
	60	0.983615	0.098368	0.009676
	70	0.974054	0.125825	0.015832
Two-term exponential	50	0.999600	0.008127	0.000066
	60	0.982050	0.059445	0.003534
	70	0.973143	0.073909	0.005463
Wang and Singh	50	0.987799	0.044867	0.002013
	60	0.992848	0.037522	0.001408
	70	0.986912	0.051594	0.002662

<sup>a</sup>  $R^2$ : regression analysis, RMSE: root mean square error,  $\chi^2$ : statistical chi-square.



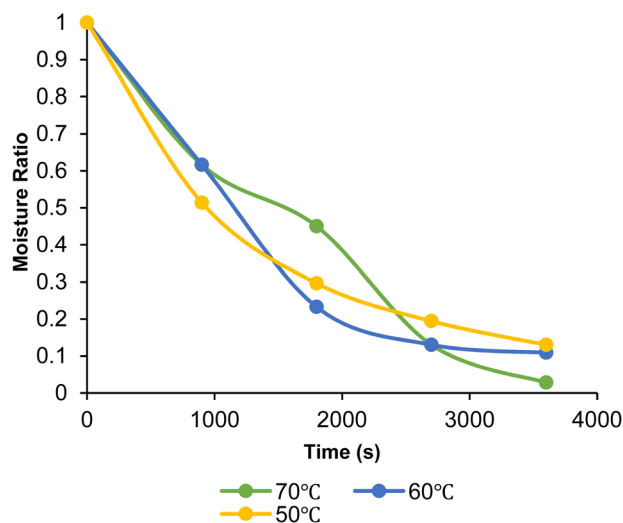


Fig. 2 Drying kinetics of convective-dried foam-mat chokeberry pulp at various temperatures (50, 60, and 70 °C).

### 3.3. Drying kinetics

Drying kinetics were calculated based on various models. The aim was to determine the most suitable drying temperature and model using statistical parameters ( $R^2$ , RMSE, and  $\chi^2$ ) for each model separately (Table 2). The two-term model at 50 °C, with the highest  $R^2$  and the lowest  $\chi^2$  and RMSE values, was selected as the most suitable model. The effect of drying temperature on the moisture ratio of chokeberry foam is illustrated in Fig. 2. As the drying temperature increases, the energy required (enthalpy) to remove the water bound within the food matrix during the drying process tends to decrease.<sup>35</sup> However, at lower temperatures, dehydration requires a higher energy input due to stronger interactions between water molecules and food components. It has also been reported that elevated drying temperatures lead to a more pronounced reduction in moisture content and shorter dehydration duration.<sup>36</sup> The literature indicates that the models identified in various foam drying studies differ. It has been reported that the foaming agents used in such studies influence drying time and kinetics.<sup>37</sup> It was observed that agents containing only albumin had no effect on foam drying time, whereas the drying time for xanthan gum and carboxymethyl cellulose varied depending on temperature. As expected, an increase in drying temperature led to a decrease in drying time and an increase in the diffusion coefficient.

In a previous study, Leite *et al.* (2023) dried *Mentha crispa* foam by adding foaming agents at different concentrations (3, 5, and 7%) and drying at temperatures of 50, 60, and 70 °C.<sup>38</sup> The study found that the Page model provided the most suitable drying kinetics. On the other hand, a study investigated the effect of different drying conditions (hot air and microwave drying methods) on the bioactive properties of black mulberry fruit.<sup>39</sup> Their results showed that the best bioactive properties were obtained at 300 W and 50 °C, with the logistic model providing the best drying kinetics. Additionally, for blackberry fruit dried using microwave and hot air methods, the logarithmic model provided the best fit.

### 3.4. Physicochemical properties of foam-mat dried chokeberry pulp powders

**3.4.1. Color and browning index.** When examining the color values of chokeberry powders, it was observed that the  $L^*$  values were similar across samples, while the  $a^*$  value was higher in convectively dried powders. As shown in Table 3, this increased redness cannot be solely explained by the anthocyanin content of the powders. Instead, the heat applied during drying may have induced browning reactions, contributing to the elevated redness values. The BI, presented in Table 2, further supports this finding.

A distinct difference was noted in the yellowness and blueness of the powders: lyophilized powders exhibited higher yellowness values, whereas convectively dried powders showed more pronounced blueness. The BI values of powders produced by foam-mat drying ranged from 0.96 to 1.15, with the highest BI observed in samples dried at 50 °C. Interestingly, a greater degree of browning was associated with lower drying temperatures, likely due to prolonged drying times promoting non-enzymatic browning reactions. However, no significant difference ( $p > 0.05$ ) was found between convectively dried and freeze-dried chokeberry powders. Similar trends have been reported in previous studies, where increasing the drying temperature from 50 to 70 °C resulted in lower BI values.<sup>40,41</sup>

Findings from other studies align with the observations of the current study. In an investigation of the drying and powder properties of chokeberries using various drying methods,  $L^*$  values of 13.87 for fresh berries, 20.80 for lyophilized samples, 15.18 for vacuum-dried samples, and 17.69 for convectively dried samples were reported.<sup>42</sup> Their results indicated that lyophilized aronia berries had the highest brightness values.

Table 3 Colour properties, pH, water activity ( $a_w$ ) and solubility of foam-mat dried chokeberry pulp powders produced by freeze drying and convective drying at various temperatures (50, 60, and 70 °C)<sup>a</sup>

Sample	Colour				pH	Water activity – $a_w$	Solubility (%)
	$L^*$	$a^*$	$b^*$	BI			
FD	35.99 ± 0.56	16.89 ± 2.47 <sup>b</sup>	1.71 ± 0.30 <sup>a</sup>	0.96 ± 0.01 <sup>a</sup>	5.65 ± 0.02 <sup>a</sup>	0.236 ± 0.000 <sup>a</sup>	76.33 ± 0.57 <sup>a</sup>
CD 50 °C	35.93 ± 0.31	24.67 ± 0.62 <sup>a</sup>	−0.25 ± 0.02 <sup>b</sup>	1.15 ± 0.01 <sup>a</sup>	5.50 ± 0.02 <sup>a</sup>	0.216 ± 0.009 <sup>bc</sup>	74.88 ± 1.07 <sup>a</sup>
CD 60 °C	37.09 ± 0.96	24.13 ± 0.15 <sup>a</sup>	−0.83 ± 0.21 <sup>c</sup>	1.08 ± 0.07 <sup>a</sup>	5.53 ± 0.01 <sup>a</sup>	0.205 ± 0.002 <sup>c</sup>	75.06 ± 0.37 <sup>a</sup>
CD 70 °C	37.03 ± 1.32	22.16 ± 0.46 <sup>a</sup>	−0.56 ± 0.04 <sup>bc</sup>	1.08 ± 0.09 <sup>a</sup>	5.49 ± 0.10 <sup>a</sup>	0.219 ± 0.001 <sup>b</sup>	75.21 ± 0.18 <sup>a</sup>

<sup>a</sup> BI: browning index. There is no statistical difference between the means indicated with the same letter in each column ( $p > 0.05$ ).



Further research on micelle formation from chokeberry pomace using egg yolk and egg white powders found that  $L^*$  values varied based on the composition: 80.65 for samples containing egg yolk powder, 91.70 for those with egg white powder, 35.16 for samples with both egg yolk and egg white powders, and 36.26 for the lyophilized control.<sup>43</sup> Similarly, another study<sup>44</sup> evaluated the color and nutritional properties of chokeberry pomace and found that hot-air-dried powders appeared darker than those dried *via* lyophilization. Their results confirmed that the  $L^*$  value was higher in lyophilized samples. In another study on chokeberry powder quality,<sup>42</sup> it was determined that the highest  $L^*$ ,  $a^*$ , and  $b^*$  values were found in lyophilized samples, further supporting the benefits of lyophilization in preserving color properties.

**3.4.2. pH, solubility and water activity.** The pH values of the produced powders ranged between 5.49 and 5.56 suggesting that the powders are slightly acidic, which is typical for fruit-based powders.<sup>30</sup> The slightly acidic nature of the powders may help preserve certain phenolic compounds, especially anthocyanins, which are generally more stable in acidic environments.<sup>45</sup> At the same time, the near-neutral pH of the powders suggests that their incorporation into food products would not result in an overly sour or sharp taste, and is unlikely to negatively affect consumer acceptance, which is a positive indication. Although the pH values of the powders were lower in convectively dried samples, the difference was not statistically significant ( $p > 0.05$ ).

Water activity is a key indicator of free water availability in food, which influences biochemical reactions. In this study, the water activity of the powders ranged between 0.205 and 0.236, well below 0.25, which suggests good microbiological stability and low risk of microbial growth in powdered products, contributing to extended shelf life and product safety.<sup>46</sup> There was a significant difference ( $p < 0.05$ ) between the water activity of powders produced by freeze drying and convective drying. The powder produced by freeze drying had a higher water activity, this is typically unexpected because freeze drying is capable of removing both free and some bound water more thoroughly in comparison to convective drying.<sup>47</sup> In addition, freeze drying typically creates a more porous structure, which allows for more efficient sublimation and lower final moisture content, in comparison to convective drying. Nevertheless, the porous structure of freeze-dried powders may also result in higher moisture reabsorption from the environment during handling or storage.<sup>47,48</sup> Overall, the water activity of the produced powders is acceptable (below 0.25). Studies on the

other fruits, such as black mulberry juice powder (0.15 to 0.32)<sup>49</sup> and spray-dried raisin powder (0.18 to 0.44),<sup>50</sup> have also reported similar ranges of water activity.

Solubility is one of the most important quality criteria for dried products. The solubility of the powders produced ranged between 74.88 and 76.33%. Literature reports indicate that freeze dried products typically exhibit the highest solubility values.<sup>51</sup> While lyophilized powders in this study also showed the highest solubility, the difference was not statistically significant compared to convectively dried samples ( $p > 0.05$ ). Overall, the solubility levels suggest that the powders have a good reconstitution ability. The powders would dissolve relatively well in water or aqueous solutions, which is desirable for applications in beverages, instant products, or functional ingredients. The solubility of the produced powder is close to previously produced foam-mat dried yacon (80.49–84.16%)<sup>52</sup> and mango (77.60–85.18%)<sup>53</sup> juice powders, which is affected by the type of foaming agent.

**3.4.3. Density.** The bulk density of chokeberry powder ranged between 0.30 and 0.41 g cm<sup>-3</sup> (Table 4). Freeze-dried chokeberry powder exhibited significantly ( $p < 0.05$ ) higher bulk density compared to convectively dried powders. Similarly, the tap density of freeze-dried chokeberry powder was also significantly higher ( $p < 0.05$ ) than that of convectively dried powders. The higher bulk and tap density of freeze-dried chokeberry powder compared to convectively dried powders can be attributed to several factors. Freeze-drying preserves the structural integrity of the material by removing water through sublimation, resulting in a more compact and denser powder. In contrast, convective drying involves direct water evaporation, which can cause particle shrinkage and lead to a looser, less dense structure.

The flowability and cohesiveness of chokeberry powder were evaluated using the Carr index and Hausner ratio, respectively (Table 4). Flowability values ranged from 18.93 to 26.84%, while cohesiveness values were between 0.73 and 0.81. Notably, the Carr index increased as drying temperature increased in convectively dried powders. A Carr index between 15 and 20% is classified as “Good”, while samples with a Hausner ratio below 1.2 are considered to have low cohesiveness.<sup>54</sup> A comparative analysis of drying methods revealed that freeze-dried and convectively dried chokeberry powders exhibited superior flowability (lower Carr index) compared to vacuum-dried powders.<sup>42</sup> Additionally, the lower Hausner ratio observed in these powders suggests reduced stickiness, which is desirable for improved handling and processing. These findings align with prior

**Table 4** Rheological properties (density and flowability) of foam-mat dried chokeberry pulp powders produced by freeze drying and convective drying at various temperatures (50, 60, and 70 °C)<sup>a</sup>

Sample	Bulk density (g cm <sup>-3</sup> )	Tapped density (g cm <sup>-3</sup> )	Carr index, CI (%)	Hausner ratio, HR
FD	0.30 ± 0.02 <sup>b</sup>	0.36 ± 0.03 <sup>b</sup>	18.93 ± 0.02 <sup>b</sup>	0.81 ± 0.00 <sup>a</sup>
CD 50 °C	0.41 ± 0.04 <sup>a</sup>	0.53 ± 0.05 <sup>a</sup>	22.58 ± 0.00 <sup>ab</sup>	0.77 ± 0.00 <sup>ab</sup>
CD 60 °C	0.40 ± 0.01 <sup>a</sup>	0.54 ± 0.02 <sup>a</sup>	26.61 ± 3.96 <sup>a</sup>	0.73 ± 0.03 <sup>b</sup>
CD 70 °C	0.41 ± 0.01 <sup>a</sup>	0.56 ± 0.00 <sup>a</sup>	26.84 ± 1.45 <sup>a</sup>	0.73 ± 0.01 <sup>b</sup>

<sup>a</sup> There is no statistical difference between the means indicated with the same letter ( $p > 0.05$ ).



studies indicating that the drying method significantly influences powder properties, affecting not only flowability but also structural integrity and rehydration potential. Furthermore, it has been reported that the inclusion of certain hydrocolloids, such as methylcellulose, increases powder stickiness, leading to material clogging and processing difficulties.<sup>55</sup> This observation highlights the importance of optimizing drying conditions and formulation strategies to enhance the functional properties of chokeberry powder for industrial applications.

### 3.5. Surface and structural characterization

**3.5.1. Scanning electron microscopy.** As shown in Fig. 3, the pores in the morphological structure are attributed to air bubbles formed during foaming, which create voids and cracks as they dissipate during the drying process. The freeze-dried samples exhibited the smoothest surface and a more porous, lightweight structure. In contrast, convectively dried samples showed reduced surface porosity at higher temperatures, resulting in a denser and more compact structure. These differences highlight the impact of drying methods on the final morphology of the samples.

Similarly, in a study on the foam mat drying of cantaloupe pulp powder using convective drying, researchers found that drying conditions significantly influenced the structural characteristics of the dried foam mats.<sup>56</sup> The porous structure varied depending on drying temperature and foam thickness, with higher drying speeds promoting a wider pore structure due to

enhanced heat and mass transfer. Additionally, at elevated drying temperatures, the reduced drying time minimized bubble coalescence and foam collapse, preserving the porous structure more effectively. In a study on foam mat drying of green banana powder using convective drying,<sup>57</sup> researchers observed that higher drying air temperatures accelerated moisture evaporation, leading to faster drying and case hardening of the surface. The increased vacuole vapor pressure helped prevent the deflation of hollow particles, ultimately resulting in smoother particle surfaces.

**3.5.2. Infrared spectroscopy.** The FTIR spectra of chokeberry powders are shown in Fig. 4. Due to the increased drying time in the samples, a decrease in peak heights was observed, which can be attributed to protein denaturation at 60–70 °C. The highest peak intensity was detected in freeze-dried foam and chokeberry, while the lowest peak intensity was recorded at 60 and 70 °C. Anthocyanins exhibited strong absorption in the 3750–2900  $\text{cm}^{-1}$  range, corresponding to O–H and C–H stretching vibrations in hydroxyl groups.<sup>58</sup> The peak around 1640  $\text{cm}^{-1}$  is predominantly associated with C=C bond vibrations of phenolic and aromatic components. A small peak at approximately 1600  $\text{cm}^{-1}$  corresponds to C=O double bonds present on the anthocyanin B ring.<sup>59</sup> The band at 1400  $\text{cm}^{-1}$  is attributed to in-plane O–H bending vibrations in phenols.<sup>60,61</sup> Variations in the spectral region between 1400 and 1199  $\text{cm}^{-1}$  are due to O–C–H, C–C–H, and C–O–H bending vibrations in carbohydrates.<sup>62</sup> Peaks observed between 1200 and 900  $\text{cm}^{-1}$  are associated with C–O and C–O–C bonds.<sup>63,64</sup> Weak bands at

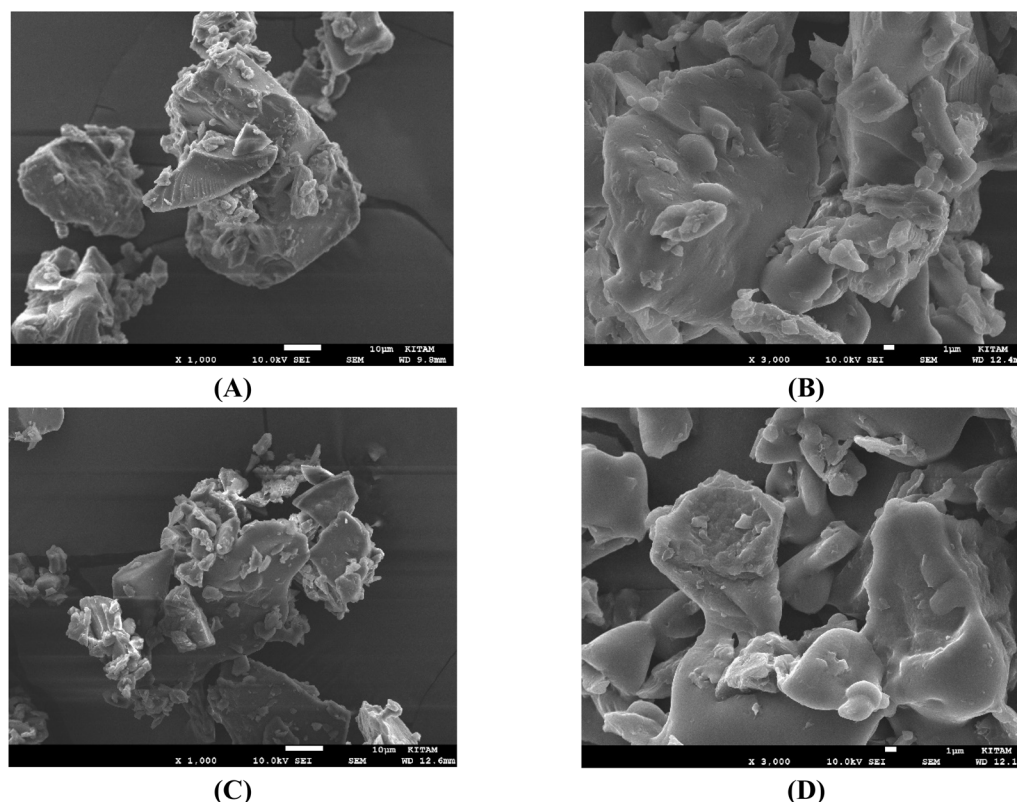


Fig. 3 Microstructural images of convective-dried foam-mat chokeberry pulp powder at (A) 50 °C, (B) 60 °C, and (C) 70 °C and freeze-dried foam-mat chokeberry pulp powder (D).





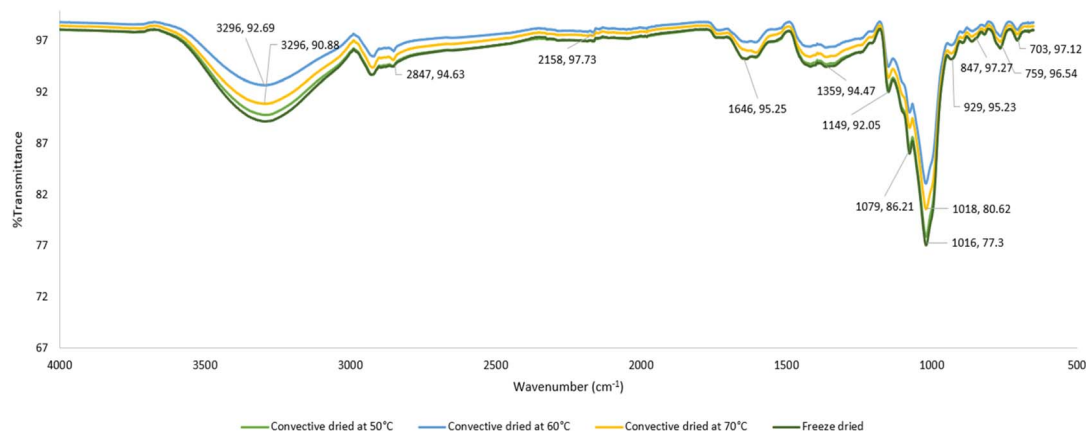


Fig. 4 FTIR spectra of dried chokeberry, and convective-dried foam-mat chokeberry pulp powder at 50 °C, 60 °C, and 70 °C and freeze-dried foam-mat chokeberry pulp powder (values on the figure indicate the wavenumber followed by the percentage transmittance).

930  $\text{cm}^{-1}$  and 850  $\text{cm}^{-1}$  result from  $\text{CH}_2$  group vibrations, while peaks below 800  $\text{cm}^{-1}$  are primarily attributed to benzene ring vibrations.<sup>59</sup>

### 3.6. Phenolic, flavonoid and anthocyanin content

As shown in Fig. 5 (numerical values used to construct Fig. 5 are provided in Table A – ESI<sup>†</sup>), the highest retention of total phenolics ( $41.14 \pm 0.22 \text{ mg g}^{-1}$ ), flavonoids ( $6.03 \pm 0.12 \text{ mg g}^{-1}$ ), and anthocyanins ( $4.42 \pm 0.27 \text{ mg g}^{-1}$ ) was observed in freeze-dried powders. Although the convective dried samples had a significantly lower TP (30.75–32.42  $\text{mg g}^{-1}$ ) and TF (5.20–5.43  $\text{mg g}^{-1}$ ) content compared to freeze-dried samples, their TP and TF content was not significantly affected by temperature ( $p > 0.05$ ) suggesting that the 50 to 70 °C can conserve chokeberry TP and TF. Nevertheless, a slightly different trend was observed with the TA content. The TA content decreased significantly when the drying temperature was increased above 50 °C ( $p < 0.05$ ); from  $4.11 \pm 0.01 \text{ mg g}^{-1}$  at 50 °C to  $2.94 \pm 0.11 \text{ mg g}^{-1}$  at 70 °C. Overall, as seen in Fig. 5, increasing temperature had a negative impact on the bioactive content, although not significant for TP and TF content.

A similar trend in anthocyanin content was observed during the foam mat drying of black rice bran anthocyanin.<sup>65</sup> When drying at temperatures of 60, 70, and 80 °C, the highest TA content was recorded at the lowest temperature (60 °C), with a significant decline at higher temperatures. Similarly, convective drying of hawthorn foam mat powders at 60, 65, and 70 °C demonstrated that increasing the drying temperature negatively affected TP, TF, and TA content, with 60 °C yielding results closest to those of freeze-dried powders.<sup>66</sup> A comparable trend was observed in the foam mat drying of jujube juice<sup>67</sup> and sour cherry concentrate,<sup>30</sup> where a negative correlation was found between increasing temperature and decreasing bioactive content. The convective-foam mat dried chokeberry powder in our study had a slightly lower TA content than the spray-foam mat dried blueberry powder (4.8–5.7  $\text{mg g}^{-1}$ ).<sup>68</sup> However, the TP content of their blueberry powder was lower (20.8–31.1  $\text{mg g}^{-1}$ ) than that of the chokeberry powder in our study. Similarly, our chokeberry powder exhibited a higher TP content compared to foam mat dried blackthorn powder, which ranged from 10.7 to 14.8  $\text{mg g}^{-1}$ .<sup>69</sup> This suggests that the current product is relatively rich in phenolic compounds. Nevertheless,

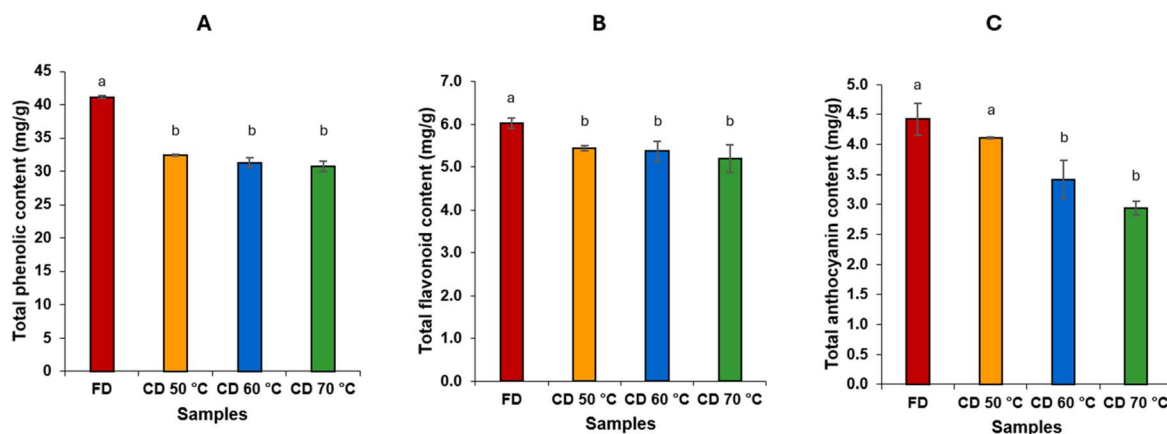


Fig. 5 Total phenolic (A), flavonoid (B), and anthocyanin (C) content of foam-mat dried chokeberry pulp powders produced by freeze drying and convective drying at various temperatures (50, 60, and 70 °C). There is no statistical difference between the means ( $\pm$  standard deviation) indicated with the same letter ( $p > 0.05$ ).



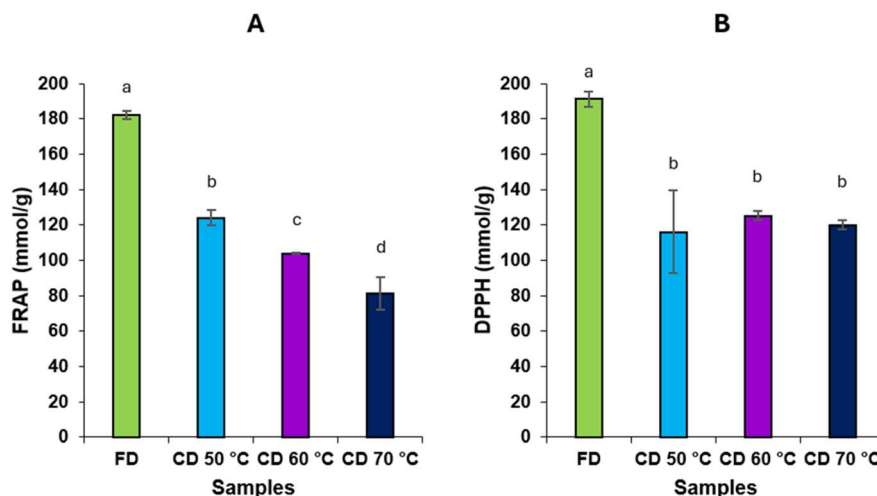


Fig. 6 Antioxidant activity of foam-mat dried chokeberry pulp powders produced by freeze drying and convective drying at various temperatures (50, 60, and 70 °C) based on Ferric Reducing Antioxidant Power (FRAP) assay (A), and 2,2-diphenyl-1-picryl-hydrazyl (DPPH) assay (B). There is no statistical difference between the means ( $\pm$  standard deviation) indicated with the same letter ( $p > 0.05$ ).

it is important to consider the differences in fruit type, growing conditions, and foam mat drying parameters, all of which can influence the retention of bioactive compounds.

### 3.7. Antioxidant activity

The antioxidant activity of the samples was assessed by the FRAP and DPPH *in vitro* assays. As seen in Fig. 6, the freeze-dried sample had the highest antioxidant activity in comparison to the convective-dried samples ( $p < 0.05$ ). Similarly, it has been reported<sup>70</sup> that low-temperature processes such as freezing and freeze drying of basil varieties and post-harvest preservation methods enabled obtaining plant material with higher antioxidant activity compared to convection drying, which is a thermal method. It is thought that chokeberry powders preserve their antioxidant compounds through the freeze-drying process. There was no significant difference between the DPPH values of the convective dried samples ( $p > 0.05$ ) which is aligned with the trend seen in TP and TF content. On the other hand, the FRAP values of convective dried samples were significantly affected by temperature ( $p < 0.05$ ). As the convective drying temperature increased, the FRAP value decreased significantly, suggesting a loss in antioxidant content at higher temperatures. The difference in the trend seen with the FRAP and DPPH values could be due to differences in the antioxidant assay's mechanism. DPPH is performed in methanol, making it less suitable for hydrophilic antioxidants. On the other hand, FRAP is performed in an aqueous acidic medium, favoring hydrophilic antioxidants. This can suggest that the chokeberry hydrophilic antioxidants, captured by the FRAP assay, are more heat sensitive. Chokeberries are a rich source of tannins<sup>71</sup> and vitamin C<sup>15</sup> which do not have a great solubility in methanol and thus may not be captured by DPPH assay. In comparison to other studies, a similar trend was observed in the antioxidant potential of foam-mat dried mango, where antioxidant activity, measured by FRAP, significantly decreased with increasing temperature, particularly above 60 °C.

C.<sup>72</sup> This decline could be associated with the degradation of heat-sensitive antioxidants.

Given the rich phenolic, flavonoid, and anthocyanin of chokeberries, they have a strong potential as a functional food ingredient to combat oxidative stress. The cellular antioxidant activity of chokeberry polyphenols is primarily attributed to the presence of anthocyanins and neochlorogenic acid.<sup>73</sup> Chokeberry proanthocyanidins contribute the most to its antioxidant activity, accounting for 40%, followed by anthocyanins at 24%, hydroxycinnamic acids at 18%, and epicatechins at 11%.<sup>74</sup> The procyanidin polymers in chokeberry can reach up to nearly 9.98 g/100 g dm<sup>3</sup>. Overall, chokeberry exerts its antioxidant effects through several mechanisms, including neutralizing free radicals, reducing the generation of reactive oxygen and nitrogen species, enhancing the activity of antioxidant enzymes, and inhibiting prooxidant enzymes.<sup>75,76</sup> Thus, the developed ingredient can support a wide range of nutritional benefits and it is important to mention that polyphenols have other benefits and are not limited to antioxidant activity and are being explored in new areas such as in promoting skeletal muscle health<sup>77</sup> and prebiotic effects to improve gut health.<sup>78</sup>

## 4 Conclusions

In this study, the effects of different drying methods (freeze-drying and convective drying at 50, 60, and 70 °C) on the physicochemical properties and bioactive composition of chokeberry pomace powder were evaluated. The drying method and temperature significantly influenced color parameters, with freeze-dried samples exhibiting lower  $L^*$  (lightness) values, while a slight increase in  $L^*$  was observed as the convective drying temperature increased. The  $a^*$  (redness) value remained low in freeze-dried samples but reached its highest level at CD 50 °C, suggesting that the Maillard reaction played a role in color changes during convective drying. In terms of solubility, freeze-dried samples had the lowest values, whereas CD 50 °C



exhibited the highest solubility, with slight fluctuations at higher drying temperatures. Flow properties and compressibility analysis showed that freeze dried samples had an acceptable flowability. Among convectively dried samples, the 50 °C-dried sample had higher compressibility but still maintaining flow properties comparable to FD. Overall, freeze drying was the most effective method for preserving both physical properties and bioactive content. However, convective drying at a lower temperature (50 °C) emerged as a viable alternative, balancing economic feasibility with the retention of TP, TF, and TA content and antioxidant capacity (FRAP). Additionally, based on the drying kinetics of convective drying, the two-term model provided the best fit for describing the drying process, further supporting CD 50 °C as an efficient and cost-effective option while maintaining favorable powder characteristics. The foam-mat drying approach in this study addressed challenges related to viscosity, pectin, and fiber content, which impacted the physicochemical properties of chokeberry and limited its applications in food product development previously. Additionally, this method helps reduce waste by utilizing mature berries to produce a functional ingredient appealing to a broad consumer base. One of the limitations of this study is that bioactive compounds and antioxidant activity were not measured before the drying process, which prevented the calculation of their retention levels. Future studies should take this aspect into account when designing experiments. Further studies could also evaluate the bioaccessibility and bioavailability of this ingredient when incorporated into different food matrices.

## Data availability

The data supporting this article have been included within the manuscript. Further information can be provided by the corresponding author (H. P.).

## Author contributions

YY and BK: methodology, writing – original draft, investigation, data curation and formal analysis. AAR: data curation, formal analysis, writing – original draft, and writing – review & editing. HP and IK: conceptualization, supervision, and project administration.

## Conflicts of interest

The authors declare that they have no competing interests.

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For the purpose of open access, the authors have applied a “Creative Commons Attribution (CC BY) license to any Author Accepted Manuscript version arising from this submission”. Artificial Intelligence (AI) was used to review the language of the manuscript.

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