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Effects of dehydration temperature on physico-chemical, antioxidant and antimicrobial properties of grape pomace powder

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ABSTRACT

This study investigated the impact of dehydration temperature on physicochemical, antioxidant and antimicrobial properties of grape pomace. To this end, grape pomace from *Vitis vinifera*, a by-product of the wine industry, was dehydrated at 35, 50, and 65 °C in a conventional hot air dryer and subsequently ground into fine powders. The three powders were characterized in terms of microbial contamination, antimicrobial activity, antioxidant properties, condensed tannin content and total phenolic content. Furthermore, particle size distribution, nano-scale structure, thermal properties, and water content of each powder were also determined.

The results demonstrated that increasing the dehydration temperature from 35 to 65 °C led to a reduction in microbial contamination and the powders exhibited more pronounced effects against selected microbial and fungal species. Notably, the antioxidant properties of the powders remained largely unchanged up to 65 °C, although a kinetic effect was observed, associated with a slower release of the active components in the more efficiently dehydrated samples. Physicochemical analysis indicated that dehydration temperatures ≥50 °C induced minor alterations in the powder size, nano-scale organization, and composition, while substantially reducing hygroscopicity.

1. Introduction

Grape pomace, a mixture of grape skin, stem, pulp and seeds, is the most widely available by-product of the wine industry, with more than 10 million tons produced worldwide every year after the pressing and fermentation process of grapes. Numerous studies conducted in the last ten years indicate that grape pomace is an important source of value-added bioactive molecules, including phenolic acids, flavonoids, tannins, and stilbenes (Hassam et al., 2019; Peixoto et al., 2018; Chemat et al., 2012; Xu et al., 2016; Teixeira et al., 2014), which are endowed with antioxidant, anti-inflammatory, antimicrobial, antifungal and anticarcinogenic properties (Machado et al., 2024). On this basis, grape pomace extracts are extensively exploited in several fields, spanning from health supplements and food packaging to the pharmaceutical and cosmetic sectors (Leal et al., 2020; Samani et al., 2025; Wang et al., 2024). Grape pomace recycling offers a dual benefit, promoting the recovery of valuable phenolic compounds, dietary fibers, and other phytochemicals (Tseng & Zhao, 2013), while simultaneously reducing the costs and environmental impact associated with winery waste disposal (García-Lomillo et al., 2014; Ilyas et al., 2021; Wang et al., 2024). However, grape pomace is highly perishable due to its high moisture content (50–60 %), which can easily facilitate microbial and

Abbreviations: ABTS, 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt; CFC, cetrimide fucidin cephaloridine; DPPH, 2,2-diphenyl-1-picrylhydrazyl; DTG, Differential thermogravimetry; DSC, Differential Scanning Calorimetry; FRAP, Ferric reducing/antioxidant power; FWI, Free Water Index; LT-DSC, Low-Temperature DSC; PAB, *Pseudomonas* Agar Base; PCA, Plate Count Agar; PCB, Plate Count Broth; TGA, Thermogravimetry; TPC, Total phenolic content; TPTZ, 2,4,6-tris(2-pirydyl)-*s*-triazine; SAB, Sabouraud Dextrose Agar; SAXS, Small Angle X-ray Scattering; VRBA, Violet Red Bile Agar.

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fungal proliferation. To overcome this limitation, dehydration of grape pomace is now considered a viable strategy to extend its shelf life (Ramirez-Pulido et al., 2021).

Various dehydration techniques have been developed over the years, highlighting for each of them advantages and disadvantages. In particular, the potential impact of these dehydration processes on the pomace phenolic content has been extensively investigated (Carmona-Jiménez et al., 2018; Chamorro et al., 2011; Goula et al., 2016; Kim et al., 2006). In fact, in the literature it is widely acknowledged that drying methods, including emerging drying intensification technologies (e.g., vacuum-drying, ultrasound-assisted osmotic dehydration, microwave-freeze-drying), can influence both the retention and the quality of bioactive compounds in fruit and vegetable by-products, as well as their bioaccessibility (Cilla et al., 2018; Radojcin et al., 2021; Ramírez-Pulido et al., 2021). Therefore, the selection of an appropriate dehydration process must consider several variables, such as the initial moisture content, the temperature, and the time of dehydration (Carmona-Jiménez et al., 2018; Tseng & Zhao, 2012), to preserve the bioactive components, which are generally sensitive to heat and oxygen (Poblete et al., 2024). Among all the dehydration technologies, hot air dehydration is commonly applied in the food industry to obtain powdered ingredients, given the reduced costs, the low handling requirements and the integral recovery of the material. Despite the considerable interest, few efforts have been made to predict the dehydration kinetics of grape pomace (Conte et al., 2024), and to the best of our knowledge, a systematic investigation on the effects of hot air dehydration at different temperatures on the physical and chemical properties of grape pomace for its integration within polymeric materials is apparently lacking in the literature. By-products have been successfully incorporated into biodegradable plastics for food packaging, both to protect the polymeric matrix against thermal and photo-induced degradation, and to extend food shelf life (Coppola et al., 2021; Ortega et al., 2022). However, further research is still necessary on the functional properties of powders from agri-food by-products for a rational implementation of active food packaging materials.

On this basis, the present study investigated the effect of hot air dehydration at three different temperatures (35, 50 and 65 °C) on the functional properties and physicochemical characteristics of grape pomace from *Vitis vinifera*. Firstly, the contamination level and the antimicrobial activity of dehydrated grape pomace powders were compared with those of the fresh grape pomace sample. In addition, hot air-dehydrated by-products were evaluated in terms of their phenolic composition, antioxidant properties, size, nanoscale structure, and thermal properties. Therefore, the paper aimed to verify the activity of grape pomace powders, their hygroscopicity, and aggregate morphology to facilitate powder dispersion for potential incorporation within water-based polymeric matrices.

2. Material and methods

2.1. Raw materials and dehydration process

Vitis vinifera (cv. Montepulciano) grape pomace was kindly provided by a local winery (Cantine della Bardulia, Barletta, Italy). The sample was immediately stored at −18 °C to avoid enzymatic degradation and contamination. The initial by-product moisture content was around 85 g/100 g. The product was then thawed at room temperature and a hot air dehydration was carried out at three different temperatures (35 °C, 50 °C and 65 °C) in a conventional dryer, according to the procedure already reported by Conte et al. (2024). The temperatures were selected to assure an effective dehydration without applying excessive heat treatments. The temperature started from less more than room temperature (35 °C) to reach 65 °C, increasing by 15 °C. Dehydration was completed when the grape pomace weight was constant for three to four consecutive measurements. Moisture contents of final dehydrated powders were 4.73 g/100 g at 35 °C, 2.82 g/100 g at 50 °C and 2.55 g/100 g at 65 °C. A total time of 45 h and 25 min, 20 h and 30 min and 17 h and 30 min was necessary for the pomace dehydration carried out at 35, 50 and 65 °C, respectively. The dehydrated by-product was reduced to a fine powder (size <500 μm) using a hammer mill (16/BV-Beccaria s.r.l., Cuneo, Italy) and then stored under refrigerated conditions before further utilization.

2.2. Contamination level of grape pomace powders and pH determination

To evaluate the effects of the dehydration process on microbial and fungal level, microbiological analyses were carried out on grape pomace, before and after the dehydration at different temperatures. A sample of grape pomace (10 g) was diluted in physiological water under sterile conditions and homogenized in a laboratory blender (Stomacher LAB Blender 400 (Pbi International, Milan, Italy). The homogenized samples were subjected to decimal dilutions, then plated on specific media in Petri dishes according to the following groups: Plate Count Agar (PCA, Oxoid) incubated at 30 °C for 48 h for mesophilic bacteria; *Pseudomonas* Agar Base (PAB, Oxoid) with a selective supplement of cetrimide fucidin cephaloridine (CFC), incubated at 25 °C for 48 h for *Pseudomonas* spp.; Violet Red Bile Agar (VRBA, Oxoid) incubated at 37 °C for 24 h for Coliforms; Sabouraud Dextrose Agar (SAB, Oxoid) with a selective supplement of chloramphenicol incubated at 25 °C for 48 h and 120 h, for total yeasts and molds respectively. Microbiological analyses were performed in duplicate.

The pH was measured two times on different samples, using the first sample dilution. To the aim, a pH meter (Crison, Barcelona, Spain) was used after proper calibration.

2.3. In vitro antimicrobial activity of grape pomace powders

The antimicrobial activity of the three grape pomace powders was evaluated by *in vitro* test against a mixture of *Pseudomonas* spp. (*P. fluorescens* and *P. putida*) and yeasts, both isolated from spoiled food. For the two *Pseudomonas* spp. strains, exponentially growing cultures were obtained in Plate Count Broth (PCB, 5 g/L tryptone, 1 g/L glucose and 2.5 g/L yeast extract, Oxoid) at 25 °C for 24 h. Subsequently, a cocktail of them was diluted in saline water (NaCl in water, 9 g/L) to obtain approximately 10^3 CFU/mL. The samples were: the inoculated PCB with 5 g/100g (w/w) grape pomace powder dehydrated at 35 °C, 50 °C and 65 °C, and the inoculated PCB without any powder, used as the control. All the samples were incubated at 25 °C for 72 h. The same test was carried out against yeasts. The analyses were performed after 0, 4, 24, 48, and 72 h. After appropriate dilutions with saline water the samples were plated on PAB, with a selective supplement of CFC incubated at 25 °C for 48 h to count *Pseudomonas* spp., and on SAB, with a selective supplement of chloramphenicol incubated at 25 °C for 48 h, for counting yeasts. Experiments were performed in duplicate. A fitting model based on a re-parameterized version of the Gompertz equation (Lacivita, Lordi, Kalaydzhiev, et al., 2023) was used for the *in vitro* test data to calculate the time within the viable cell concentration of *Pseudomonas* spp. and yeasts remained under the threshold for acceptability, set at 10^6 CFU/g and 10^5 CFU/g for *Pseudomonas* spp. and yeasts, respectively. The model parameters are referred to as microbial acceptability limits (MAL) (Lacivita, Lordi, Kalaydzhiev, et al., 2023).

2.4. Antioxidant properties of grape pomace powders

2.4.1. a2,2-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assay

The following chemicals were adopted: methanol, hydrochloric acid, 2,2-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS), potassium persulfate, Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid). All solvents used for chemical analyses were of analytical grade. The antioxidant properties were measured on each dehydrated grape pomace powder according to the

method reported by Cedola et al. (2019). Briefly, the ABTS radical cation was obtained by reacting ABTS stock solution with 2.45 mmol/L potassium persulfate. The mixture remained in the dark at room temperature for 12–16 h. Using 5 mmol/L phosphate buffered saline (pH 7.4), the solution was diluted to an absorbance of 0.700 ± 0.020 at 728 nm. Then, 300 μL of sample extract were added to 2.2 mL of diluted solution and mixed for 6 min at room temperature before the measure. A calibration curve was built using Trolox as standard, at concentrations between 1.56 mg/L and 50 mg/L ($R^2 = 0.9904$). The antioxidant activity was expressed as mg of Trolox equivalents (eqs) per gram of dry weight (dw) of sample. The analyses were run in triplicate.

2.4.2. a2,2-Diphenyl-1-picrylhydrazyl (DPPH) assay

To a freshly prepared 200 μmol/L solution of DPPH (Sigma Aldrich, Milan, Italy) in ethanol, a proper amount of each dehydrated powder (final dose 0.2–0.5 mg/mL) was added. The mixtures were kept under vigorous stirring at room temperature and the absorbance at 515 nm was periodically measured on a Jasco V-730 (Cremella, Lecco, Italy) spectrophotometer (Goupy et al., 2003). EC_{50} values - the dose at which 50 % DPPH reduction is achieved -were determined at 10 min. Experiments were run in triplicate.

2.4.3. Ferric reducing/antioxidant power (FRAP) assay

The dehydrated powders were added (final dose 0.2–0.3 mg/mL) to a FRAP solution prepared by mixing 0.3 mol/L acetate buffer (pH 3.6), 20 mmol/L ferric chloride (Sigma Aldrich, Milan, Italy) in water and 10 mmol/L 2,4,6-tris(2-pirydyl)-s-triazine (TPTZ, Sigma Aldrich, Milan, Italy) in 40 mM HCl, in the ratio 10:1:1 v/v/v (Benzie & Strain, 1996). The mixtures were kept under vigorous stirring at room temperature and the absorbance of the solution at 593 nm was measured after 10 min. Results were expressed as Trolox eqs. The experiments were run in triplicate. When required, the absorbance at 593 was monitored over a longer time (up to 24 h).

2.4.4. Condensed tannin content (CTC) assay

The dehydrated powders were added (final dose 0.3–1 mg/mL) to 1 mL of 0.01 g/mL w/v vanillin solution in methanol (Herald et al., 2014). 1 mL of 9 mol/L HCl was then added, and the mixture was incubated at 30 °C under stirring. The absorbance at 500 nm was measured after 10 min. Results were expressed as catechin eqs. The experiments were run in triplicate. When required, the absorbance at 500 nm was monitored over a longer time (up to 40 h).

2.4.5. Total phenolic content (TPC) assay

Samples were added (final dose 0.5–2.5 mg/mL) to 2 mL of a solution containing the Folin-Ciocalteu reagent (Sigma Aldrich, Milan, Italy), Na_2CO_3 (75 g/L) and water in a 1:3:14 v/v/v ratio (Seifzadeh et al., 2019). After incubation for 2 h at room temperature, the absorbance at 765 nm was measured. Gallic acid was used as a reference compound. Experiments were run in triplicate.

2.5. Physico-chemical characterization of grape pomace powders

2.5.1. Laser granulometry

The size distribution of dehydrated grape pomace powders was determined using a Mastersizer 3000 Laser granulometer (Malvern Instruments Ltd., Malvern, UK). Sample powders fractionated through 500 μm and 355 μm sieves were analyzed to assess the potential aggregation upon dispersion in water. The measurements were conducted with a Hydro SM dispersing unit with a stirring speed of 3000 rpm. The powder was added until a laser obscuration of ~5 % was achieved. For each sample, 20 runs of 10 s each were averaged. Water (refractive index at 20 °C: 1.333) was used as dispersant, while the optical properties of grape pomace were: density 0.40 kg/m^3 (Zhao et al., 2015), absorption coefficient 0.01, refractive index 1.46. The Fraunhofer approximation was used to extract the size distributions. The results (average ± standard deviation) were expressed as volume fractions, reported as D_V (10), D_V (50) and D_V (90), and width of the distribution (SPAN = [D_V (90)-D_V (10)]/D_V (50)).

2.5.2. Small angle X-ray scattering (SAXS)

Scattering experiments on grape pomace powders (sieved <355 μm) were performed with a Xeuss 3.0 (Xenocs, Grenoble, France) working with a Cu K_α radiation ($\lambda = 1.542$ Å) provided by Genix3D X-ray source (operating at 50 kV and 0.6 mA) and equipped with a Dectris 1 M Eiger detector. Two instrument configurations (sample to-detector distance of 300 mm and acquisition time of 600 s and sample to-detector distance of 1800 mm and acquisition time of 1800 s)were selected to access the 0.0075–0.5 1/Å scattering vector (q) range. All the measurements were carried out at room temperature. Samples were sealed in a powder sample holder (sample thickness 1 mm) between two Kapton windows (thickness 50 μm each). Scattering curves were corrected for Kapton contributions using the XSACT software (Xenocs, Grenoble, France). Data were analyzed with SasView software (http://www.sasview.org/). The *Unified fit* model was used for modelling the experimental curves; the *Unified fit* equation is:

$$I(q) \approx \sum_{i=1}^{n} G_i \, exp\left(\frac{-q^2 R_{gi}^2}{3}\right) + B_i \left(\frac{-q^2 R_{g(i+1)}^2}{3}\right) \left\{ \frac{\left[erf\left(qR_g / \sqrt{6}\right)\right]^3}{q} \right\}^p$$

where n is the level number used in the fitting, G_i and B_i are, respectively, the Guinier and the Power-Law scaling factors, R_{gi} is the radius of gyration and, lastly, p is the Power-Law exponent.

2.5.3. Thermogravimetric analysis (TGA)

Thermogravimetry was performed with a SDT Q600 thermogravimetric analyzer from TA Instruments (New Castle, DE, USA). An open-type alumina pan was chosen as sample holder. The grape pomace powder samples (sieved <355 μm) were subjected to a linear temperature ramp from room temperature to 1000 °C at a rate of 10 °C/min in N_2 atmosphere (flow rate 100 mL/min).

2.5.4. Differential scanning calorimetry (DSC)

DSC measurements were carried out with a DSC 2500 **differential scanning calorimeter** from TA Instruments (New Castle, DE, USA). Grape pomace samples (sieved <355 μm) were equilibrated at 20 °C and heated to 80 °C at 10 °C/min in N_2 atmosphere (flow rate 50 mL/min). Low-Temperature DSC (LT-DSC) measurements were performed with the following temperature program: equilibration at 5 °C, cooling ramp from 5 to −80 °C, at 0.5 °C/min, heating ramp from −80 to 20 °C, at 0.5 °C/min. The Free Water Index (FWI) was calculated as:

$$FWI = \frac{\Delta H_{exp}}{\Delta H_{free\ water}} * 100$$

where ΔH_{exp} is the enthalpy change (in J/g) of the water melting experimentally determined by measuring the area under the peak in the DSC curve, while $\Delta H_{free\ water}$ (333.6 J/g) is the standard melting enthalpy of water (333 J/g) (Gelli et al., 2017).

2.6. Statistical analysis

Experimental data and fitting parameters were compared by the one-way analysis of variance (ANOVA), using STATISTICA 7.1 for Windows (Stat Soft, Inc., Tulsa, OK, USA). Duncan's multiple range test, with the homogeneous group option ($p < 0.05$), was carried out to determine significant differences among samples.

3. Results and discussion

3.1. Effects of dehydration temperature on contamination level and antimicrobial activity

A progressive reduction in contamination level was found in dehydrated grape pomace at increasing temperature (Table 1). Mesophilic bacteria and yeasts decreased from 5.79 to 6.41 to 2.42 and 2.95 log CFU/g, respectively, in the fresh sample and in the pomace dehydrated at 65 °C. A very low cell number (1.5 log CFU/g) was recorded for coliforms, regardless of the dehydration temperature. Viable cell concentrations of approximately 3 log CFU/g were measured for *Pseudomonas* spp. on fresh by-products and the value decreased to 2 log CFU/g after the dehydration process. Therefore, the thermal treatment in the range of 50–65 °C is effective to reduce the microbial and fungal viable cell concentration, which is generally high in food matrices with high humidity content and elevated water activity (Tseng & Zhao, 2012). No major variations among samples were recorded in terms of pH, which ranged from 3.51 to 3.95 (Table 1).

After grinding, the pomace powders were tested against *Pseudomonas* spp. and yeasts as typical food spoilage (Lacivita, Lordi, Posati, et al., 2023). From data reported in Table 2 it is possible to infer that increasing values of the fitting parameter (MAL) were recorded when the dehydration temperature was increased from 35 to 65 °C. Specifically, for *Pseudomonas* spp. less than 1 day was necessary to reach the threshold in the control sample, whereas 3 days were required when the powder was dehydrated at 65 °C. Regarding yeasts, 5 h were necessary in the control to reach the set threshold and almost 30 h when the powder was dehydrated at 65 °C. Findings in the literature demonstrate that dehydrated pomace generally retains over time the polyphenolic composition (Chikwanha et al., 2018; Guaita et al., 2021). Therefore, our experimental findings are in agreement with the literature, suggesting that the effects against bacterial and fungal species not compromised by thermal treatment. It is worth noting that literature data indicate that two different heating effects may occur simultaneously: on one hand, increased extractability of polyphenols due to disruption of the pomace matrix; on the other hand, the depolymerization of high molecular weight tannins. In other words, the thermal degradation of some phenolics compounds may be balanced by the release of other bound phenolic compounds from the matrix (Khanal et al., 2009; Kim et al., 2006).

3.2. Effect of dehydration temperature on grape pomace antioxidant properties

To evaluate the effects of dehydration temperature on the antioxidant potential of grape pomace and its phenolic content, validated chemical assays were performed. The results of ABTS and DPPH assays run over 10 min reported in Table 3 indicated a comparable efficacy for grape pomace samples dehydrated at the different temperatures. In contrast, the pomace sample dehydrated at 35 °C exhibited significantly higher reducing properties in the FRAP assay compared to the samples dehydrated at 50 and 65 °C.

This result could be attributed to the presence of a higher content of thermolabile anthocyanins in the sample dehydrated at 35 °C, as evident by the UV–Vis spectra of water-ethanol extracts of grape-pomace samples in the range 500–600 nm (Fig. S1). Anthocyanins could be in fact rapidly released in the acidic medium in which the FRAP assay is performed, thus affecting the antioxidant response in the first 10 min. To understand if the amount of residual water, and hence the dehydration temperature, could affect powder wettability, which is necessary for a prompt release of the active components in solution, in other experiments the FRAP assay was performed over a longer time (up to 24 h). As reported in Fig. 1, no substantial differences were observed between the samples dehydrated at 35 °C and 65 °C.

Notably, a similar trend was observed in the vanillin assay for the determination of the CTC. Indeed, the highest value was observed for the sample dehydrated at 35 °C (Table 3), whereas when the assay was performed for longer time comparable values were determined for the samples dehydrated at 35 and 65 °C (Fig. 2), in agreement with the FRAP assay result.

Conversely, no significant differences in TPC were observed among the three powders. Overall, the different trends observed in the various assays could be interpreted based on the different ability of the grape pomace samples dehydrated at the different temperatures to release the active components in the assay media. Indeed, in separate experiments (data not shown) it was possible to demonstrate that the response to the different assays is due to the material passed in solution from the dehydrated powders. Given the lower solubility of phenolic compounds in acidic media compared to ethanol or neutral/alkaline solutions, the FRAP assay and the vanillin assay, which work at acidic pH, are expected to be affected to a higher extent with respect to DPPH and Folin-Ciocalteu assays, by the ability of the dehydrated powders to promptly release the active compounds in solution. In line with the observed results, this ability could be in turn related to the amount of residual water affecting the wettability of the powder samples and hence could be significantly dependent from the dehydration temperature.

3.3. Physico-chemical characterization of grape pomace powders

3.3.1. Grape pomace powder size distribution

The volume-weighted size distribution curves as obtained by laser granulometry are reported in Fig. 3 and the main mathematical descriptors are listed in Table 4. The average powder sizes are described using the D_V (10), D_V (50) and D_V (90) descriptors, referring to the particle diameter at which 10 %, 50 % and 90 % of the sample total volume, respectively, consists of particles with a smaller size. The SPAN value is directly proportional to the width of the distribution. The reported results clearly demonstrate the heterogeneity of the powders in terms of size distribution, regardless of the dehydration temperature.

To assess the tendency of powders to dissolve or aggregate when dispersed in water, the powders were sieved at 355 and 500 μm prior to

Table 2
MAL values calculated as fitting parameters for *Pseudomonas* spp. and yeasts in contact with grape pomace. Means in a column without a common letter differ, $P < 0.05$.

Sample	MAL-Pseudomonas [h]	MAL-Yeast [h]
Fresh sample	11.4627 ± 1.143^{a}	5.3584 ± 1.035^{d}
35 °C	68.5486 ± 103.08^{a}	13.3516 ± 2.170^{c}
50 °C	60.2886 ± 4.903^{a}	22.8192 ± 4.236^{b}
65 °C	>72	29.57 ± 2.528^{a}

Table 1
Viable cell concentration of bacterial and fungal counts and pH values of fresh and dehydrated grape pomace samples. Reported are the mean ± SD values. Means in a column without a common letter differ, $P < 0.05$.

Sample	Mesophiles [log CFU/g]	Coliforms [log CFU/g]	Pseudomonas spp. [log CFU/g]	Yeasts [log CFU/g]	Molds [log CFU/g]	pH
Fresh sample	5.79 ± 0.43a	1.50 ± 0.71a	3.21 ± 0.29a	6.41 ± 0.57a	3.17 ± 0.18a	3.51 ± 0.02c
35 °C	5.56 ± 0.07a	1.50 ± 0.71a	2.00 ± 0.01b	5.61 ± 0.40a	2.74 ± 0.37a	3.63 ± 0.01b
50 °C	2.48 ± 0.67b	1.50 ± 0.71a	2.00 ± 0.01b	3.00 ± 0.01b	2.50 ± 0.70a	3.55 ± 0.01c
65 °C	2.42 ± 0.59b	1.00 ± 0.01b	2.00 ± 0.01b	2.95 ± 0.92b	2.98 ± 0.70a	3.95 ± 0.05a

Table 3
Antioxidant properties of grape pomace samples dehydrated at different temperatures. Reported are the mean ± SD values from at least three experiments. Means in a column without a common letter differ, $P < 0.05$.

	ABTS assay [mg Trolox./g dw]	DPPH assay EC_{50} [mg/mL]	FRAP assay [Trolox eqs]	CTC assay [Catechin eqs]	TPC assay [Gallic acid eqs]
35 °C	71 ± 9[a]	0.30 ± 0.02[a]	0.0297 ± 0.0003[a]	0.21 ± 0.05[a]	0.0019 ± 0.0009[a]
50 °C	73 ± 8[a]	0.31 ± 0.01[a]	0.0146 ± 0.0002[b]	0.15 ± 0.01[b]	0.0016 ± 0.0001[a]
65 °C	70 ± 7 [a]	0.34 ± 0.01[a]	0.0149 ± 0.0002[b]	0.16 ± 0.04[a,b]	0.0017 ± 0.0003[a]

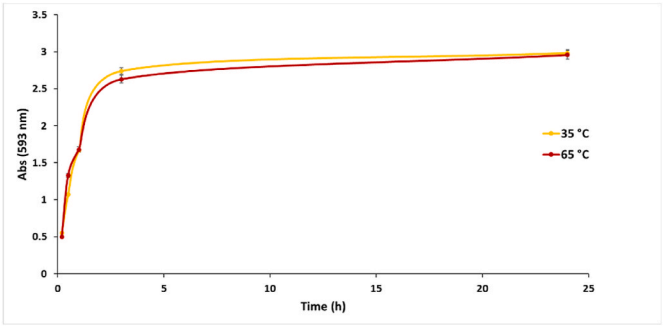

Fig. 1. Fe^{3+} reducing activity of grape pomace powder dehydrated at 35 or 65 °C over time. Reported are the mean ± SD values of three experiments.

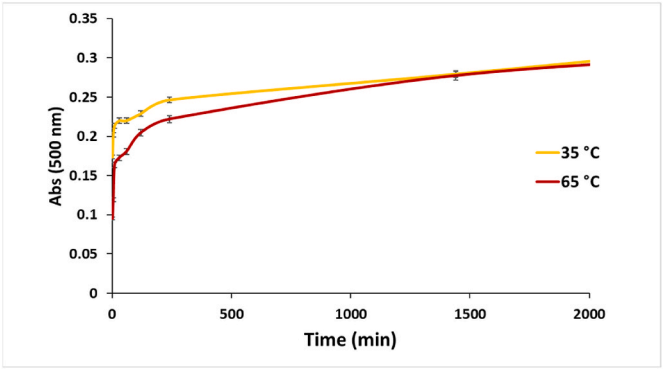

Fig. 2. CTC evaluated by measuring the increase in the absorbance at 500 nm in the vanillin-HCl assay of grape pomace powder dehydrated at 35 or 65 °C over time. Reported are the mean ± SD values of three experiments.

the measurement. The results clearly show that the particles spontaneously tend to form aggregates that are larger than the cut-off of the corresponding sieve. This phenomenon is particularly relevant for the design and engineering of food packaging systems incorporating grape pomace powders and should be considered when such systems require dispersion in water.

Other relevant aspects are the larger presence of particles with size between 10 and 100 μm and the significantly lower SPAN value in the sample dehydrated at 35 °C. On the other hand, no major differences were observed between 50 and 65 °C, suggesting that clustering occurs predominantly in the temperature range between 35 and 50 °C.

3.3.2. Nanostructure of grape pomace powders

Grape pomace powders were also investigated by means of SAXS, to probe the internal nanostructure and potentially highlight the effects of dehydration temperature. The experimental scattering curves, depicted in Fig. 4A, showed an exponential decay which was successfully modelled according to the empirical *Unified Fit* model, originally proposed by Beaucage (1995, 1996). This model, which represents the scattering intensity through the combination of multiple Guinier and structurally limited Power-Law levels, effectively describes the hierarchical nanostructure of a material in terms of levels and, consequently, is

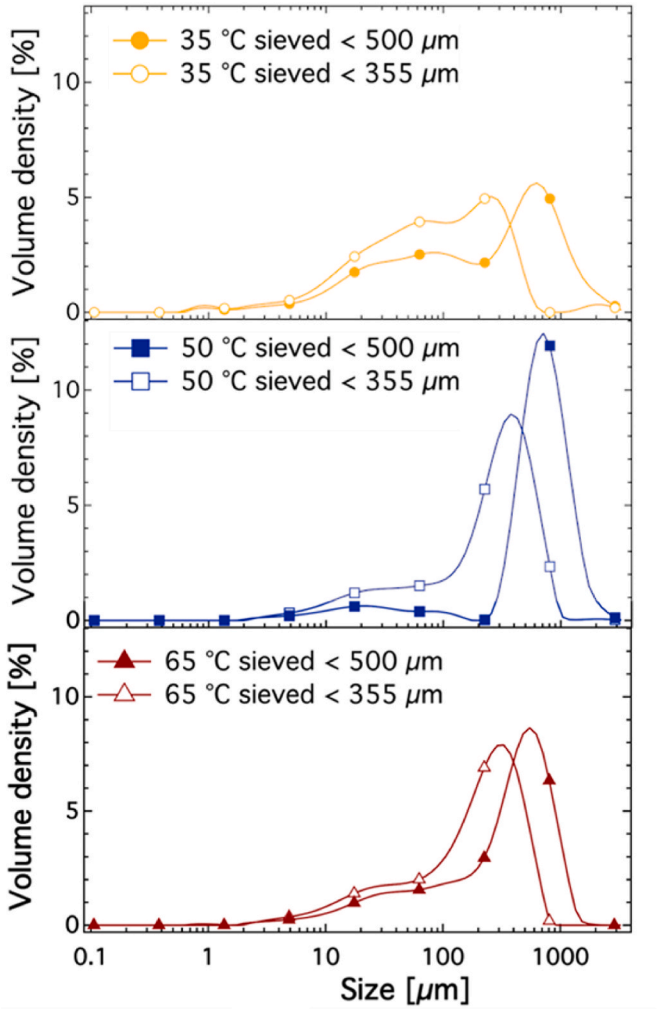

Fig. 3. Volume distribution curves of grape pomace powders dehydrated at different temperatures obtained from laser granulometry experiments.

Table 4
Size distribution parameters for grape pomace powders as obtained by laser granulometry.

Sample		D_V (10) [μm]	D_V (50) [μm]	D_V (90) [μm]	SPAN
35 °C	**< 500 μm**	17 ± 1	252 ± 30	1028 ± 102	4.01 ± 0.63
	< 355 μm	14.7 ± 0.3	88 ± 2	332 ± 7	3.60 ± 0.15
50 °C	**< 500 μm**	168 ± 95	675 ± 22	1242 ± 111	1.59 ± 0.17
	< 355 μm	23.5 ± 0.9	275 ± 6	578 ± 28	2.01 ± 0.05
65 °C	**< 500 μm**	31 ± 1	387 ± 14	844 ± 39	2.10 ± 0.08
	< 355 μm	22.0 ± 0.7	205 ± 5	474 ± 11	2.20 ± 0.06

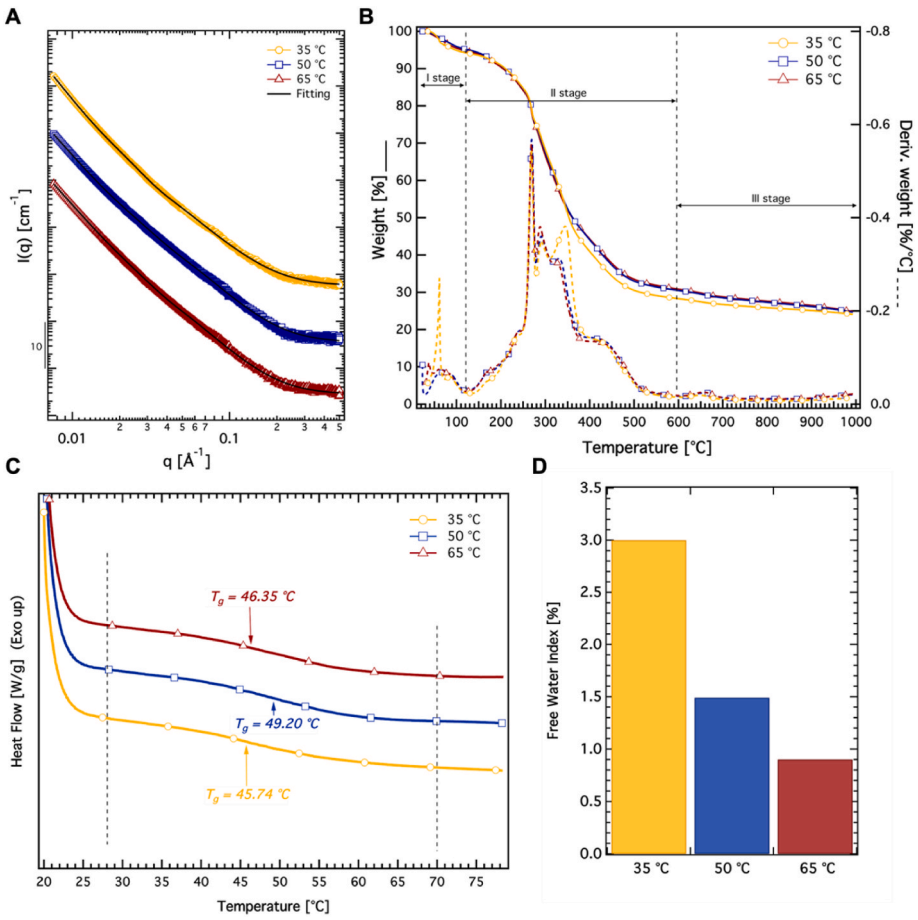

Fig. 4. A) SAXS curves of grape pomace powders and corresponding *Two Lorentzian* model fittings (black line). Curves are vertically offset for clarity of presentation; B) TGA (curves corresponding to the left Y-axis) and differential thermogravimetry (DTG) thermograms (curves corresponding to the right Y-axis) for grape pomace powders. The main decomposition stages are represented by vertical lines; C) DCS curves of grape pomace powders; D) FWI of grape pomace powders samples.

frequently employed to model the scattering from systems exhibiting features across multiple length-scales.

Specifically, the first term accounts for large-scale scattering objects of average size R_{gi}, composed of small-scale structures of average size $R_{g\,(i+1)}$. The exponent of the Power-Law term is interpreted as the fractal dimension (D_f), which provides insights into the spatial organization of the scattering centers.

Table 5 reports the results obtained from the fittings of the experimental data. Grape pomace powders, irrespective of dehydration temperature, exhibit two distinct length-scale regimes. The contributions from level 2 are attributed to the scattering from macromolecule-size objects, such as tannins, characterized by a R_g value of less than approximately 100 Å and assembled into a mass fractal with D_f close to ~2.8 (for $p < 3$, $p=D_f$) (Anitas, 2018, chap. 10). No major differences were observed between samples dehydrated at 50 and 65 °C, while a slightly smaller fractal dimension was found for the powder dehydrated at 35 °C, consistent with lower compactness and suggesting that dehydration at higher temperatures slightly enhances macromolecular organization.

Conversely, level 1 accounts for the scattering from larger structures. The results returned by the fitting show that the macromolecular aggregates in all the samples are assembled into aggregates whose size exceeds the maximum dimension accessible by the instrumental setup. Regarding the exponential factor p, the values obtained are consistent with surface fractal scattering, with a fractal dimension close to ~2.3 (for $3<p<4$, $p=6-D_f$) (Anitas, 2018, chap. 10), as observed in protein gel-like structures (Chen et al., 2024; Shi et al., 2017). Also in this case, samples dehydrated at high temperatures (*i.e.,* 50 and 65 °C) do not show significant differences, whereas grape pomace dehydrated at 35 °C is characterized by a higher exponent (p, corresponding to a lower D_f), suggesting that dehydration at 35 °C, compared to 50 and 65 °C, results in aggregates with distinct internal and surface structural features.

Table 5
Results from the fitting according to the *Unified Fit* model of the SAXS data obtained from grape pomace powders.

Sample	Level 1		Level 2	
	R_g (Å)	p	R_g (Å)	p
35 °C	> d_{max} [a]	3.93 ± 0.01	102 ± 1	2.76 ± 0.01
50 °C	> d_{max} [a]	3.61 ± 0.01	76 ± 1	2.90 ± 0.01
65 °C	> d_{max} [a]	3.58 ± 0.01	84 ± 1	2.92 ± 0.01

[a] The maximum dimension accessible with the current instrumental set-up can be calculated as $d_{max}=2\pi/q_{min}$ and is equal to 838 Å.

3.3.3. Thermal stability and properties of the grape pomace powders

The thermal stability of grape pomace powders dehydrated at the different temperatures was investigated by monitoring weight changes as a function of temperature (TGA) and its time derivative (DTG). The corresponding thermographs are depicted in Fig. 4B (TGA and DTG data are shown on the left and right axes, respectively) and in Fig. S2. As indicated by the changes in the slope of the TGA plots and by the peaks in the DTG profiles, the grape pomace thermal decomposition consisted in multiple degradation steps occurring in a broad range of temperature, which could be related to the heterogeneous chemical composition of grape pomace.

The first weight loss (occurring below 130 °C) is characterized by a minimal weight change (approximately 5 %), corresponding to the forced dehydration of the sample and to the loss of organic volatiles (Torres-Garcia & Brachi, 2020; Coelho et al., 2018; Khiari & Jeguirim, 2018; Berger et al., 2022; da Silva et al., 2024). Although the total weight loss is minor, the sample dehydrated at 35 °C exhibits a slightly higher loss, and the derivative signal reveals a notable peak around 55 °C, suggesting the release of volatile organic molecules. As previously reported (Khanal et al., 2010) heating at 60 °C or above caused a significant decrease in procyanidins and anthocyanins content in grape pomace, due to their partial degradation.

The second stage, accounting for the largest fraction of total weight loss (approximately 64 %), takes place approximately between 130 °C and 590 °C. The DTG thermographs of this stage correspond to pyrolytic events and are characterized by multiple peaks in the derivative signal located at around 270, 290 and 330–350 °C, consistent with the thermal degradation of polymeric materials such as cellulose-derived components (Monari et al., 2020; Torres-Garcia & Brachi, 2020; Coelho et al., 2018; Khiari & Jeguirim, 2018; Berger et al., 2022; da Silva et al., 2024). Notably, the sample dehydrated at 35 °C shows a peculiar behavior when compared to the other; in fact, the sample shows a higher weight loss and a distinct peak around 350 °C in the derivative signal, thus suggesting that dehydration at temperatures ≥50 °C causes a change in the powder composition.

On the other hand, no notable differences were observed among the samples in the broad shoulder around 450 °C, which can be ascribed to the degradation of lignin (Monari et al., 2020; Berger et al., 2022; Yang et al., 2007; Orfão et al., 1999). In the third stage, a flat tailing section above 600 °C was observed, together with a weak signal at ~650 °C for all the DTG curves. The weight loss is mainly ascribed to the formation of char and to the final degradation of lignin (Khiari & Jeguirim, 2018; Torres-Garcia & Brachi, 2020). Importantly, all thermograms are characterized by a significant carbonaceous residue at 1000 °C, associated with the presence of ash and highly condensed aromatic structures (Gowman et al., 2018; Monari et al., 2020).

Based on the experimental results obtained by DTG, the dehydration process carried out at 50 and 65 °C does not induce dramatic modifications on the grape pomace composition and on its thermal stability. However, minor differences observed in the 35 °C sample suggest reduced loss of water and volatile organics during drying.

Aiming at further understanding the influence of the dehydration temperature on the thermal properties of grape pomace powders, differential scanning calorimetry measurements were performed in a selected temperature range (20–80 °C), where the powders are not subjected to thermal degradation. All the obtained DSC thermograms, displayed in Fig. 4C, show a step-like change in the baseline, referred to as the glass transition temperature (T_g), between 45 °C and 49 °C (see Fig. S3 for more details). The glass transition is a reversible phenomenon typical of amorphous or partially amorphous materials, where the material transitions from a solid, brittle “glassy” state to a more rubbery or molten state with increasing temperature. In food products, this transition, which can occur in a wide range of temperatures, is affected by several factors, such as moisture content, chemical composition, and processing conditions (Joardder et al., 2024).

The presence of a T_g in the DSC indicates that the grape pomace powders are at least partially amorphous and that their amorphous structure is not significantly affected by the dehydration temperatures tested. This suggests that an alternative explanation is needed for the peculiar behavior of the 35 °C sample. Moreover, similar T_g values have been found for alginate/pullulan films enriched with water grape pomace extract (Mugnaini et al., 2024), implying that the glass transition is largely related to the water-soluble components of grape pomace, which are present in the solid powders, as well as in the water extract.

Although food products are often dried, they generally retain water molecules that are bound or adsorbed to the solid matrix (e.g., polysaccharides or proteins), and quantifying this residual moisture is essential. LT-DSC measurements, which involves cooling samples to −80 °C and then warming them to ~20 °C, is especially well-suited to quantify the amount of water strongly bound to the matrix. During the cooling and warming scans, the evolution of the heat flow was recorded. The heating scans corresponding to the melting of the freezable water are reported in Fig. S4, showing a single endothermic peak centered at around −16 °C. A common way to quantify the amount of freezable water (*i.e.,* the water able to solidify and melt) present in a sample is the Free Water Index (FWI)., As reported in Fig. 4D, the FWI decreases markedly with increasing drying temperature: in fact, the value is halved when moving from 35 °C to 50 °C, and is halved again from 50 °C to 65 °C.

These results highlight the effect of the dehydration process and indicate that the different samples display a different hygroscopicity, a minor difference in absolute terms, but relevant to the properties of grape pomace powders. These findings are consistent with the differences in the antioxidant properties observed between the grape pomace sample dehydrated at 35 °C and those dehydrated at 50 °C and 65 °C (see above).

These results suggest the potential use of grape pomace powders in functional formulations. However, processability remains a critical aspect that could influence their successful integration into various applications. One element that deserves further attention is the clustering or agglomeration that may occur during the drying process, which was not directly assessed in this work. Such microstructural changes could impact dispersibility, rheological behavior, mechanical properties of composite materials, and compatibility with formulation processes. Future research should explore the influence of drying-induced structural modifications on the functional performance of the powders, particularly under conditions relevant to food and packaging applications. This would support the optimization of processing conditions and post-treatment steps (e.g., milling or granulation), ultimately enhancing the technological value of grape pomace as a sustainable ingredient.

4. Conclusions

Grape pomace powders, dehydrated at three different temperatures (*i.e.,* 35, 50 and 65 °C), were investigated for the impact of dehydration temperature on contamination level, antimicrobial effects against *Pseudomonas* spp. and yeasts, phenolic compounds and condensed tannin contents, antioxidant properties, size, nanoscale structure and thermal properties, for their potential application as active agents in polymeric films. The experimental results demonstrated that dehydration temperature has a significant influence on the final properties of this by-product. Indeed, increasing the temperature from 35 to 65 °C, the microbial and fungal spoilage of pomace decreased to a very low contamination level without affecting the antimicrobial and antifungal activity. More specifically, when yeasts were considered, the efficacy increased as the dehydration temperature increased, probably due to combined effects of heating on extractability of polyphenols and depolymerization of high molecular weight tannins. On the other hand, dehydration temperature significantly affected the release of antioxidant phenolic compounds, mainly anthocyanins and condensed tannins, and hence impacting on the shot-term antioxidant properties of grape pomace powders, particularly in acidic media. These results are in line with the physico-chemical characterization, which highlighted the powder dehydrated at 35 °C differed from those treated at higher temperatures, where a clustering process occurred as suggested by the particle size distributions obtained from laser diffraction analysis and fractal dimensions calculated from X-ray scattering. The TGA/DTG results stated that higher dehydration temperatures (≥50 °C) minimally altered the powder composition and reduced volatiles content, while significantly affected the hygroscopicity, a feature which is also relevant to their antimicrobial and antioxidant properties. Notably, the presence of residual moisture and a different morphology of the aggregates may also facilitate pomace powder dispersion and incorporation within

water-based polymeric matrices towards the development of composite materials.

In conclusions, these results provide detailed information on the impact of the dehydration temperature on the functional properties of grape pomace, of technological relevance for the exploitation of this agri-food by-product in the food packaging sector.

CRediT authorship contribution statement

O. Panza: Writing – original draft, Formal analysis. **M.L. Alfieri:** Writing – original draft, Formal analysis. **M.A. Del Nobile:** Writing – original draft, Data curation, Conceptualization. **G. Mugnaini:** Writing – original draft, Formal analysis, Data curation. **A. Conte:** Writing – review & editing, Supervision, Data curation, Conceptualization. **Lucia Panzella:** Writing – review & editing, Validation, Data curation, Conceptualization. **M. Bonini:** Writing – review & editing, Visualization, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2025.118029.

Data availability

Data will be made available on request.

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