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RESEARCH

Effect of Ultrasound-Ethanol Pretreatment on Drying Kinetics, Rehydration, Quality Parameters, and Functional Groups of BRS Vitória Grape Using Convective Drying

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Abstract

The aim of this study was to investigate the effects of ultrasound pretreatment to improve BRS Vitória grape convective drying. For this purpose, samples without any pretreatment (control) and samples pretreated with ultrasound and water (USW) or ethanol (USEtOH) were analyzed. The effects on drying and rehydration kinetics, quality parameters, total phenolic content, antioxidant capacity, and functional groups were studied through FTIR spectrometry. The results showed that the pretreatment with ultrasound and ethanol reduced the grape's drying time by approximately 64%. The Weibull model was the best model to describe the kinetics of grape rehydration. USEtOH-pretreated samples exhibited the greatest rehydration ratio at both tested temperatures (25 and 50 °C), especially at the highest one. In terms of quality parameters, soluble solids content and color change were influenced by pretreatments, with USEtOH showing a substantial reduction in grape water activity and significant color change after drying. The content of total phenolics decreased after ultrasound pretreatments but increased during drying, with USEtOH showing better retention of these compounds. Antioxidant capacity was affected by drying, but ultrasound pretreatments mitigated losses. Functional group analysis revealed that pretreatments were able to preserve several groups identified by FTIR. These results highlighted the potential of emerging and non-thermal technologies as pretreatments for drying BRS Vitória grapes, which suggests their use in food industry applications.

Keywords Drying · Grapes · Pretreatment · Ethanol-ultrasound · Phenolics · Rehydration modeling

Introduction

Brazil is known as one of the world's leading fruit producers, with annual production of around 1.7 million tons (FAOSTAT, 2022). Grapes are the most widely planted fruit crop in the world and have various uses such as wine, juice, raisins, and table fruit productions. They are rich in minerals and phytochemicals such as flavonoids, anthocyanins, tannins, and phenolic acids (Pascoal et al., 2022).

The BRS Vitória grape has gained prominence in Brazil for its seedless table grape variety, recognized for its pleasant flavor, high yield, and resistance to mildew. In addition, its pulp and skin have a high content of phenolic compounds, resulting in an intense purple color and high antioxidant activity (Colombo et al., 2020). As grapes are highly perishable, drying can be used to extend their shelf life (Patidar et al., 2021).

Drying, which is the oldest method to preserve food including fruits and vegetables, allows to remove moisture, preventing spoilage due to the growth of microorganisms

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as well as maintaining the material's structural integrity during storage (Komble et al., 2023). However, the hot-air drying process consumes a lot of time and energy and can affect the quality of grapes, including their color, texture, and nutritional and bioactive components (Tao et al., 2016; Wang et al., 2019).

For this reason, pretreatment methods have been explored to improve the rate of drying and, consequently, reduce its negative effects (Shang et al., 2023; Sun et al., 2023; Tepe & Kadakal, 2022). Furthermore, grapes require pretreatment to create microcracks in the waxy cuticle of fruit skin, increasing its permeability and facilitating the drying process (Farias et al., 2021).

Pretreatments for grape drying can be divided into thermal, physical, and chemical methods. Chemical pretreatments involve the immersion of berries in solutions like sodium carbonate, potassium carbonate, sodium hydroxide, or oil emulsions to dissolve the waxy layer and increase the skin permeability to water (Khiari et al., 2019). However, this removal is heterogeneous and creates problems during the shelf life due to uneven removal of the wax layer, resulting in irregular texture and appearance (Deng et al., 2019). Therefore, microorganisms can more easily penetrate the grape skin during storage or transportation, increasing the risk of spoilage.

In contrast, physical pretreatments emerge as an alternative strategy to optimize grape drying, offering an array of methodologies to enhance the process. They include the utilization of both mechanical force (abrasion) and thermal energy (blanching, ohmic, and microwave heating) as well as innovative non-thermal techniques such as high-pressure processing, pulsed electric fields, and ultrasound (Patidar et al., 2021).

Emerging processing technologies, such as ultrasound, offer cost-effective and environmentally friendly options to replace or supplement traditional drying pretreatment methods, due to their ability to enhance mass transfer, reduce energy consumption, and minimize chemical usage (Amanor-Atiemoh et al., 2020; Zhou et al., 2021). Ultrasound involves sound waves with frequencies above the frequency range audible to humans, which lead to the phenomenon known as cavitation (implosion of bubbles) in a liquid, resulting in micromixing (Wang et al., 2019). Moreover, ultrasound pretreatment in solid–liquid systems can lead to some phenomena, such as the sponge effect (alternating expansions and compressions in the solid sample), high shear forces, heating effects, and cell membrane damage (Ricce et al., 2016). The use of ultrasound as a pretreatment is highly efficient in reducing drying time because it modifies the tissue structures, making them more inclined to moisture removal (Freitas et al., 2021; Miano et al., 2021). Even though in this technology water is commonly used as a medium to transmit mechanical waves to products, recent research has investigated alternatives such as osmotic solutions or ethanol (Miano et al., 2021; Rojas et al., 2020a).

Ethanol has been used as a pretreatment agent due to its ability to dissolve cell wall components, increase permeability, promote moisture transfer, and accelerate dehydration (Amanor-Atiemoh et al., 2020). Additionally, it is considered a green solvent, safe for humans, and has volatile properties, leaving no residues after drying (Granella et al., 2022). Some studies have already explored the potential combination of ultrasound and ethanol as a method to pretreat apples (Rojas et al., 2020a), carrot tissue (Dadan & Nowacka, 2021), celery slices (Miano et al., 2021), and scallion stalk (Zhou et al., 2021), showing significant reductions in drying time and improved quality characteristics.

Although the effect of ultrasound and ethanol pretreatment on the kinetics of food products' drying has already been reported (Freitas et al., 2021; Rojas et al., 2020a), its potential use in convective drying of grapes and its influence on the final product remain unclear. Therefore, the objective of this study was to test the ultrasound and ethanol pretreatment to dry BRS Vitória grapes and assess its effects on rehydration kinetics, quality parameters, and functional groups.

Materials and Methods

Raw Material and Pretreatments

Fresh, fully ripe grapes of the BRS Vitória variety were purchased from a local market in Recife, Brazil, and selected based on their integrity and homogeneity. After cleaning, fruits were stored in a refrigerator at 4 ± 1 °C for subsequent pretreatment and drying. Two pretreatments were performed, with ultrasound and water (USW) or 96% (v/v) ethanol (USEtOH), while no pretreatment was used for the control. For this purpose, grapes (100 g) were immersed in a beaker containing 200 mL of distilled water or ethanol and sonicated with an ultrasound probe (QR1000, Ultronique, Indaiatuba, Brazil; 20 kHz, 500 W, microtip of 25.4 mm) at 25 ± 1 °C for 30 min. At the end of pretreatment, grapes were removed, wiped out with a paper towel, and weighed. This process was conducted in three replicates.

Drying Process

The convective drying process was performed in a drying oven with air circulation and renewal (model MA035, Marconi, Piracicaba, Brazil). Drying was performed at 60 °C and 1.0 m/s, and samples were weighted at 1 h intervals until reaching equilibrium, i.e., a variation lower than 0.05 g in

the last consecutive three weight measurements. The initial and final moisture contents were obtained after the complete drying of the samples at 105 °C until constant weight in an oven (AOAC, 2016).

The drying kinetics was investigated using the dimensionless moisture ratio (*MR*) as a function of the time (*t*, min), according to Eq. 1:

$$MR = \frac{Mt - Me}{M_0 - Me} \tag{1}$$

where *Mt*, *Me*, and M_0 are the moisture contents expressed in g water/g dry matter (d.m.) at drying time *t* (min), at equilibrium after drying, and before drying, respectively.

The drying rate (*DR*) of grape was calculated through Eq. 2:

$$DR = \frac{Mt - Mt + \Delta t}{\Delta t} \tag{2}$$

where $Mt + \Delta t$ is the moisture content (g water/g d.m.) at time $t + \Delta t$, while Δt is the time interval (min).

Rehydration

To assess the rehydration characteristics of the sample under different conditions, rehydration was conducted according to the methodology proposed by Benseddik et al. (2019). Approximately 5 g of dried grapes, placed in a net bag, were soaked inside a beaker containing 250 mL of distilled water at 25 and 50 °C. The temperature was controlled using a thermostatic bath (NL21-03, New Lab, Piracicaba, Brazil). The samples were removed from the bath at pre-established intervals (2 h), gently dried with a paper towel, and weighed on a digital scale (JK EAB-2204N, JKI, Shanghai, China). The experiment was carried out until the samples reached constant weight, and each measurement was performed in triplicate. The rehydration ratio (*RR*) was calculated according to Buvaneswaran et al. (2022) using Eq. 3:

$$RR = \frac{Mt - M_0}{Me - M_0} \tag{3}$$

where *Mt*, *Me*, and M_0 are the moisture contents after rehydration for a time *t* (min), at equilibrium, and before rehydration, respectively.

In this work, to describe the rehydration curve of grapes, the experimental data at each of the two tested temperatures were fitted using the Peleg, Weibull, first order, and exponential models described by Eqs. 4–7, respectively:

$$RR = M_0 + \frac{t}{k_1 + k_2.t} \tag{4}$$

$$RR = \exp\left[-\left(\frac{t}{b}\right)^a\right] \tag{5}$$

$$RR = \exp(-at) \tag{6}$$

$$RR = \exp(-kt^a) \tag{7}$$

where M_0 is the initial moisture content (dry basis), and *k*, k_1, k_2, *a*, and *b* are constants.

To select the best model describing the rehydration kinetics, the coefficient of determination (R^2), root mean square error (*RMSE*), and chi-square (χ^2) were used, which were defined according to the following equations:

$$R^2 = 1 - \frac{\sum_{i=1}^{N} (RR_{pre,i} - RR_{exp,i})^2}{\sum_{i=1}^{N} (RR_{exp,i} - \overline{RR_{exp}})^2} \tag{8}$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} \left(RR_{pre,i} - RR_{exp,i}\right)^2} \tag{9}$$

$$\chi^2 = \frac{\sum_{i=1}^{N} (RR_{exp,i} - RR_{pre,i})}{N - z} \tag{10}$$

where *N* and *z* are the number of experimental data and the number of constants, respectively, $RR_{exp,i}$ and $RR_{pre,i}$ are the experimental and predicted rehydration ratios at time *t*, while RR_{exp} is the mean experimental rehydration ratio.

Quality Parameters

Water Activity and Soluble Solids

Soluble solids were determined using a digital refractometer (model r2 i300, Reichert, Buffalo, NY, USA), and the results were expressed in °Brix. Water activity was determined using a Decagon water activity analyzer (PawKit model, BrasEq, Jarinú, Brazil) at a temperature of 25 °C, with a standard deviation of ± 0.01 in the final value. Three replicates were performed for each treatment.

Texture

The texture of the fresh, pretreated, and dried grapes was evaluated using a Texture Analyzer (CT3-1000, Ametex Brookfield, Middleborough, MA, USA), with a 25.4-mm-cylindrical probe used to compress the samples up to 5 mm at 2 mm/s. The maximum force developed during the test was recorded every 5 s (Wang et al., 2017). Hardness, which is usually analyzed by measuring the resistance of a material to deformation or permanent changes when subjected to external forces or tensions, was determined

in the present study as the mean of five replicates and expressed in N.

Color Evaluation

A digital colorimeter (model CR-400, Konica Minolta Sensing, Inc., Osaka, Japan) was used to examine the surface color of fresh and treated grape before and after drying. After calibrating the equipment with a standardized white tile plate, grapes were placed on a round transparent glass plate (5 cm in diameter and 1.4 cm in height) superimposed on a white plate, and the analyses were carried out. Five repetitions were performed for each replicate. The total color difference (ΔE) of the samples was calculated by Eq. 11 using the fresh sample values ($L^* = 25.29 \pm 0.84$, $a^* = -0.66 \pm 0.17$, $b^* = 1.61 \pm 0.08$) as reference (Zhou et al., 2021):

$$\Delta E = \sqrt{\left(\Delta L^*\right)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (11)$$

where L^* is lightness, a^* is the color from red ($+a^*$) to green ($-a^*$), b^* is the color from yellow ($+b^*$) to blue ($-b^*$), while ΔL^*, Δa^*, and Δb^* are the related differences.

Bioactive Compounds and Antioxidant Activity

Sample Extraction

Sample extracts from fresh, pretreated, and dried grapes were prepared to assess their total phenolic content and antioxidant activity. Each extract was obtained by mixing 10 mL of acidified ethanol and sample (2 g of fresh or pretreated grapes or 1 g of dried grape). Extraction was performed in an ultrasonic bath (USC-2850A, Unique, Indaiatuba, Brazil) for 40 min. After centrifugation for 10 min at 4000 rpm, the extract was obtained by filtering through a filter paper and stored in an amber glass bottle. All determinations were conducted in triplicate. The extract was stored under refrigeration (4 °C) until the analyses.

Total Phenolic Content

The total phenolic content was determined using the Folin-Ciocalteu method (Wettasinghe & Shahidi, 1999). It consisted of the extraction of phenolic compounds from the matrix followed by the reaction with the Folin-Ciocalteu reagent in the presence of sodium carbonate to form a blue-colored complex, whose color intensity is proportional to the amount of reactive phenolic compounds in the sample. Thus, 0.5 mL of the diluted extract was mixed with 8 mL of water and 0.5 mL of Folin-Ciocalteu reagent. After 3 min, 1 mL of saturated sodium carbonate solution was added, and the mixture was then placed in the dark for 1 h. All tests were carried out in triplicate. The absorbance was read at 765 nm using a UV–Vis spectrophotometer (UV-1900i, Shimadzu, Kyoto, Japan) and expressed in mg gallic acid equivalent (GAE) per 100 g of dry matter.

Antioxidant Activity

The antioxidant activity of the extracts was determined by two different methods, namely the ABTS and the FRAP assays, as specified below. Each measurement was conducted in triplicate.

ABTS Assay

The ABTS assay involves the formation of the radical cation $ABTS^{\bullet+}$ by reaction of 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) with potassium persulfate (Re et al., 1999). For this assay, three different concentrations of the extracts were used, with each determination performed in triplicate. Aliquots of 100 µg of concentrated extract were mixed with the $ABTS^{\bullet+}$ solution. After 6 min, the absorbance was read at 734 nm wavelength in the above-mentioned UV–Vis spectrophotometer, and, with the aid of a standard curve prepared with solutions of 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) at different concentrations, the results were expressed in micromolar Trolox equivalent g^{-1} d.m.

FRAP Assay

The antioxidant activity was also determined by the ferric reducing antioxidant power (FRAP) assay according to the method described by Benzie and Strain (1996) with some modifications (Pulido et al., 2000). The working solution, prepared by mixing 2.5 mL of 10 mM tripyridyl triazine (TPTZ) solution as the iron-binding ligand, 25 mL of 3 M acetate buffer (pH 3.6), and 2.5 mL of 2 mM ferric chloride aqueous solution, was used immediately after the preparation. The extract (90 µL) and distilled water (270 µL) were added to 2.7 mL of the working solution, and the resulting mixture was incubated at 37 °C for 30 min. The absorbance was measured at 595 nm, and results were expressed as µM ferrous sulfate g^{-1} d.m.

Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The effect of pretreatment on functional groups of grapes was analyzed using a Fourier transform infrared (FTIR) spectrometer (IRTracer-100, Shimadzu, Kyoto, Japan) equipped with attenuated total reflectance (ATR) plate.

Samples were directly spread onto the surface of ATR crystal, and spectra were recorded in the wavenumber range from 4000 to 400 cm^{-1} with 16 scans.

Data Analysis

Data analysis for drying and rehydration kinetics, modeling, and one-way ANOVA with Tukey test was performed using the Statistica 13.1 software (TIBCO Software, Palo Alto, CA, USA) with a 5% level of significance. Principal component analysis (PCA) was performed using the OriginPro 2021 software (Origin Lab Corp., Northampton, MA, USA).

Results and Discussion

Drying Kinetics

The dimensionless moisture ratio (*MR*) evolution along the time under the different drying conditions tested is shown in Fig. 1a. Drying time under different conditions was calculated as the time required to reach a mass variation lower than 0.05 g in the three last recorded measures. At the end of drying, the moisture content was 0.21 ± 0.02 g water g^{-1} d.m. in the control, 0.23 ± 0.02 g water g^{-1} d.m. in the sample treated with ultrasound combined with water (USW), and 0.12 ± 0.01 g water g^{-1} d.m. in the one treated with ultrasound combined with ethanol (USEtOH).

Ultrasonication with ethanol significantly influenced the drying process, resulting in a substantial reduction (by 64%) in drying time, while USW showed an 18% reduction in drying time when compared to the control ($p < 0.05$). These results are comparable to those found by de Freitas et al. (2021) and Miano et al. (2021), who observed, for pineapple and celery slices pretreated with ultrasound and ethanol, reductions in drying time by more than 70% and 33.34–83.34% compared to the control, respectively. USEtOH also showed the greatest reduction (83.3%) in scallion slices' drying time (Zhou et al., 2021).

The drying rate (*DR*) was calculated using data from experiments carried out using different pretreatments, and its changes versus the moisture content are plotted in Fig. 1b. As can be seen, the drying process took place mainly in the falling rate period, with only short periods of nearly constant *DR*. A high initial peak was observed for the USEtOH-treated sample, showing the highest *DR* during processing. On the other hand, for the USW-treated and control samples, *DR* increased slowly up to the peak values. Martins et al. (2022) reported similar findings, suggesting that USEtOH as a pretreatment resulted in the highest yacon potato *DR*. It has been proposed that the ability of USEtOH to increase the drying rate of food products is due to the combined effects of ultrasound-induced cavitation and ethanol capability of modifying the physical and chemical properties of the material being dried (Amanor-Atiemoh et al., 2020). Furthermore, the combination of ultrasound and ethanol can increase the permeability of the cell membrane and improve the diffusion of moisture within the material (Pandiselvam et al., 2023; Zhou et al., 2021). A combination of these effects may have been responsible for the faster and more efficient moisture removal during the drying process observed in this study.

Effect of Pretreatment and Temperature on Rehydration

Rehydration is a complex process that aims to bring a dehydrated food product back to its natural state, restoring its

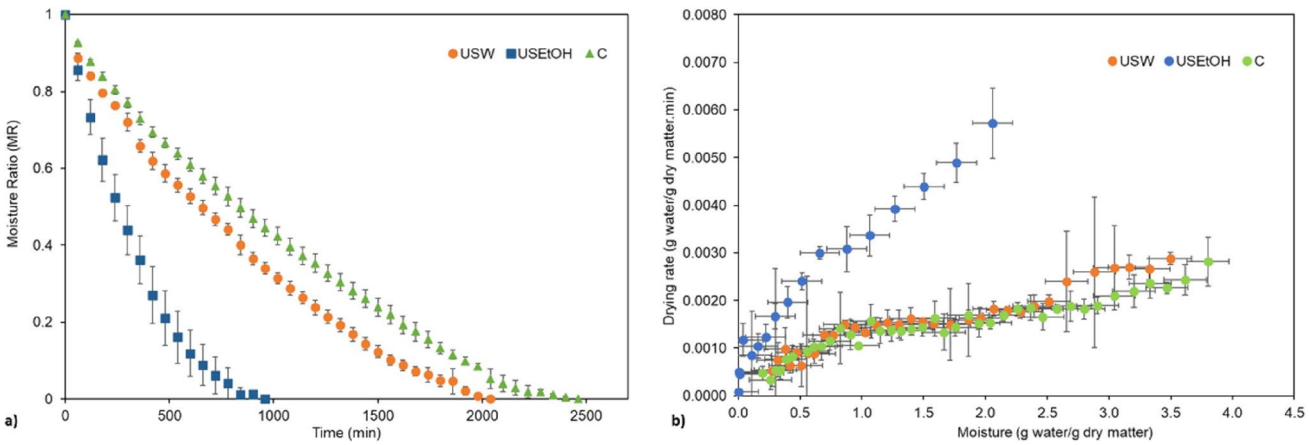

Fig. 1 Moisture ratio versus drying time (**a**) and drying rate versus moisture content (**b**) of grape samples submitted to different pretreatments: untreated sample (C), sample treated with ultrasound and water (USW), and sample treated with ultrasound and ethanol (USEtOH). Values are expressed as the mean of three determinations ± standard deviation

food properties by contacting it with water or steam (Berk, 2018). During drying, alterations in the structure of the matrix may occur, which affect the capacity of the dehydrated product to bind with water, preventing the restoration of the raw material initial volume (Sahoo et al., 2022; Wiktor et al., 2019). Thus, reconstitution properties are fundamental for most dehydrated foods and need thorough investigation. The relationship between rehydration ratio (*RR*) and rehydration time under different rehydration conditions at 25 and 50 °C can be seen in Fig. 2a, b, respectively.

Regardless of the rehydration temperature (25 or 50 °C), USEtOH-treated samples showed higher *RR* than the others. Some studies reported that ultrasound/ethanol pretreatment greatly improved rehydration of dried carrot slices (Santos et al., 2021), pumpkin (Rojas et al., 2020b), and scallion stalks (Zhou et al., 2021), likely due to structural improvement in tissues that allowed for better water absorption and faster rehydration. On the other hand, control and USW-treated samples exhibited reduced water absorption rates, which can be ascribed to the long drying time that resulted in excess structural changes (Santos et al., 2021). It has also been claimed that ultrasounds can damage cell structure, affecting cell's ability to retain water and resulting in a reduced rehydration capacity (Ricce et al., 2016).

Comparing the two tested temperatures, samples rehydrated at 50 °C showed a quicker water absorption than those rehydrated at 25 °C. High temperatures may have resulted in the increased expansion of microchannels and capillary tunnels, causing the fiber food structure to soften and leading to a higher *RR* (Tepe & Kadakal, 2022; Wang et al., 2019). The experimental data from the rehydration process were fitted to the Peleg, Weibull, first order and exponential models of *RR* versus time (Eqs. 4–7), whose results are summarized in Table 1. Compared to the other models, the Weibull one displayed the best fit with values of the determination coefficient (R^2) > 0.99, and values of root mean square error (*RMSE*) and chi-square (χ^2) close to zero for all pretreatments. Our results agree with those of previous studies in which the Weibull model proved to be the best option to describe the rehydration of dehydrated ginkgo seed (Boateng et al., 2021), tiger nuts (Osae et al., 2023), and plum (Hassan et al., 2022) slices.

Impact of Pretreatment on Quality Parameters

The results of quality parameters of samples are gathered in Table 2. After pretreatments, there was no significant change in sample water activity (a_w). Similarly, Dadan and Nowacka (2021), evaluating the effect of USEtOH pretreatment on convective drying in carrot tissue, did not observe any statistically significant effect on a_w, likely because ethanol dissolution in water already present in the sample prevented notable separation. As is known, a_w is an intrinsic factor of food that indicates its free water content, which is essential for assessing its stability after processing (Wiktor et al., 2021); in particular, a_w values < 0.6 are considered able to inhibit the growth of spoilage microorganisms (Dadan & Nowacka, 2021); therefore, the low a_w values (0.53 to 0.59) detected in dried samples in this study indicate their stability during storage.

Hardness, which is defined as the force required to cause deformation in a material (Macedo et al., 2021), is another important parameter to assess the texture of dried food. Table 2 shows the hardness of grapes following the pretreatment and drying process. The application of ultrasound resulted in a significant reduction ($p < 0.05$) in hardness

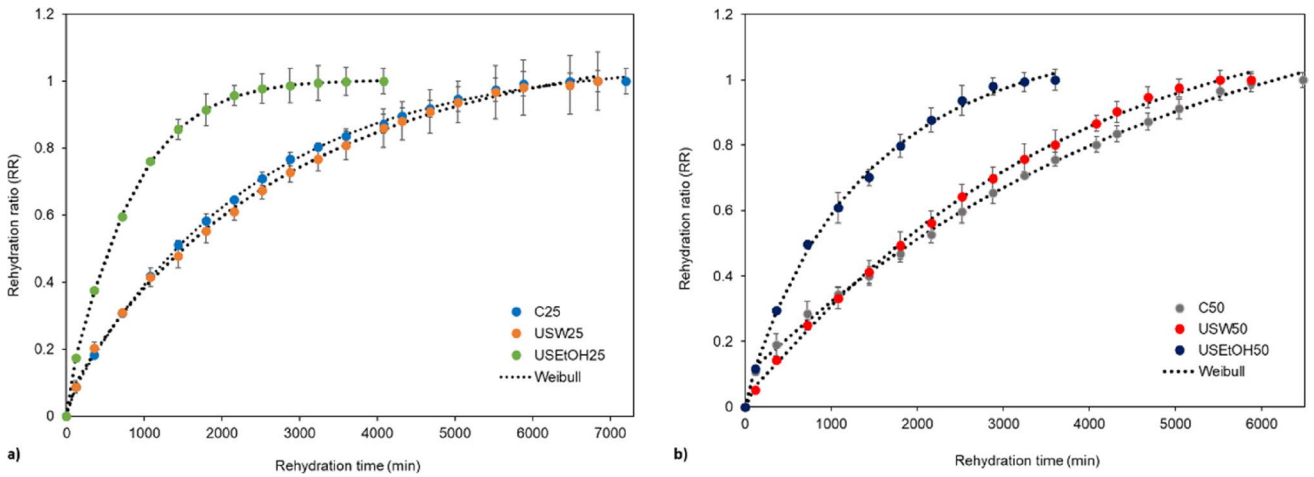

Fig. 2 Rehydration ratio assessed versus rehydration time at two different rehydration temperatures, namely 25 and 50 °C. **a** 25 °C: untreated sample (C25), sample pretreated with ultrasound and water (USW25), and sample pretreated with ultrasound and ethanol (USEtOH25). **b** 50 °C: untreated sample (C50), sample pretreated with ultrasound and water (USW50), and sample pretreated with ultrasound and ethanol (USEtOH50)

Table 1 Rehydration kinetic parameters for the control (C) and samples pretreated with ultrasound and water (USW) or ethanol (USEtOH) at 25 and 50 °C

Model	Parameter	C25	USW25	USEtOH25	C50	USW50	USEtOH50
Peleg	k_1	8.0915	7.5219	2.7398	4.6270	5.4107	1.4318
	k_2	0.0161	0.0303	0.5185	0.6296	0.4922	0.9210
	R^2	0.9128	0.8906	0.8998	0.9110	0.9138	0.9041
	RMSE	0.0896	0.1084	0.1067	0.0933	0.0902	0.8088
	χ^2	0.0090	0.0132	0.0137	0.0096	0.0090	0.0124
Weibull	*a*	1.0609	1.1408	1.0038	1.3603	1.2263	1.1377
	b	0.0378	0.0422	0.0843	0.0939	0.0330	0.0003
	R^2	**0.9995**	**0.9989**	**0.9999**	**0.9983**	**0.9988**	**0.9978**
	RMSE	**0.0064**	**0.0090**	**0.0031**	**0.0110**	**0.0104**	**0.0128**
	χ^2	**0.0001**	**0.0001**	**0.0000**	**0.0002**	**0.0001**	**0.0003**
First order	*a*	0.0004	0.0004	0.0009	0.0005	0.0005	0.0013
	R^2	0.9781	0.9781	0.9935	0.9958	0.9924	0.9987
	RMSE	0.0449	0.0484	0.0273	0.0202	0.0267	0.0118
	χ^2	0.0021	0.0025	0.0008	0.0004	0.0008	0.0002
Exponential	*a*	0.0002	0.0001	0.0007	0.0004	0.0005	0.0001
	k	1.0648	1.2269	1.0365	1.0413	0.9995	1.0020
	R^2	0.9799	0.9905	0.9939	0.9962	0.9924	0.9987
	RMSE	0.0430	0.0320	0.0263	0.0193	0.0267	0.0118
	χ^2	0.0021	0.0012	0.0008	0.0004	0.0008	0.0002

*The data in bold presented the best fit with the values of the coefficient of determination (R2), root mean square error (RMSE) and chi-square (χ2)

Table 2 Effects of pretreatments with ultrasound and water (USW) or ethanol (USEtOH), before and after drying, on water activity, hardness, total soluble solids, and color change (ΔE) of grapes

Pretreatment	Water activity	Hardness (N)	Total soluble solids (°Brix)	ΔE
After pretreatment/before drying				
Fresh sample	0.97 ± 0.01^{a}	13.61 ± 1.14^{c}	31.30 ± 6.56^{b}	REF*
USW	0.97 ± 0.01^{a}	9.51 ± 0.76^{c}	19.23 ± 1.45^{c}	1.93 ± 0.31^{d}
USEtOH	0.96 ± 0.01^{a}	8.59 ± 0.63^{c}	25.47 ± 2.63^{b}	5.70 ± 0.30^{bc}
After drying				
Control	0.53 ± 0.01^{d}	31.51 ± 3.07^{b}	77.78 ± 1.90^{a}	5.36 ± 0.78^{c}
USW	0.59 ± 0.02^{c}	41.14 ± 4.55^{a}	79.93 ± 1.97^{a}	7.41 ± 0.94^{ab}
USEtOH	0.55 ± 0.01^{b}	30.73 ± 4.86^{b}	80.21 ± 1.19^{a}	8.60 ± 0.71^{a}

REF* Fresh sample was taken as a reference

Data are given as means ± standard deviations. Different lowercase letters within the same column indicate that mean values are significantly different ($p < 0.05$)

compared to the fresh sample—although with no difference between the USW—and UEtOH-pretreated samples—likely due to the ultrasound-induced formation of pores and microscopic channels (Xu et al., 2022). These structural changes may have disrupted the integrity of the grape tissue, leading to a softer texture due to increased porosity and altered microstructure.

After drying, treatments resulted in increased hardness ($p < 0.05$), with the USW treatment having the highest hardness. However, there was no statistically significant difference between the control and USEtOH samples. This can be explained by the fact that drying removes moisture from the grapes, leading to a denser structure and consequently increased hardness. Rashid et al. (2022), who studied the effect of ultrasound pretreatments with infrared and hot-air drying on sweet potatoes, also observed a significant increase in hardness of ultrasound-pretreated samples, likely due to the low sample moisture content.

Additionally, the combination of ultrasound and water may have induced further structural changes, enhancing the hardness even more. On the other hand, the lack of significant difference between the control and USEtOH-treated

samples suggests that ethanol may not have had a significant effect on grape hardness during drying.

Table 2 also lists the content of total soluble solids (TSS) in grapes, which significantly increased after drying compared to both pretreated and fresh samples, with no significant effect of the ultrasonic pretreatment.

The L^*, a^*, and b^* parameters were used to calculate the total color change (ΔE) (Table 2), which describes the general variations compared to a reference (fresh sample). Significant color change was observed in samples submitted to ultrasonic pretreatments ($p < 0.05$). The combination of ethanol and ultrasound resulted in higher ΔE values compared to the sample pretreated with ultrasound and water before drying ($p < 0.05$). Furthermore, USEtOH-pretreated sample before drying showed a similar value to the control after drying. The possible effect of ultrasound and ethanol on fruits and vegetables may be due to the presence of pigments in them, which can be reduced by ethanol, leading to a change in color (Feng et al., 2019).

After drying, all pretreated samples showed a notable ΔE increase ($p < 0.05$) compared to their initial values before drying. This trend is in line with the findings of Martins et al. (2022), who observed significant color alterations in yacon potato slices due to a combination of chemical bleaching, ethanol, and ultrasound pretreatment both pre- and post-drying.

Regarding the effect of ultrasound, it was found that the USW- and USEtOH-pretreated samples after drying had similar ΔE values. Ren et al. (2022) observed that the ultrasonic treatment of dried ginger slices resulted in an increase in color change and that the type of solvent (water or ethanol) had no statistically significant effect. Some enzymatic and non-enzymatic oxidative reactions during ultrasound pretreatment and drying can influence the overall color of the final dried products (Xu et al., 2022).

Total Phenolic Content

The total phenolic content (TPC) in fresh samples, in USW- and USEtOH-pretreated samples and in dried samples is depicted in Fig. 3.

Fresh grapes had the highest mean TPC (1597.42 ± 28.04 mg GAE/100 g d.m.), which was within the range reported for fresh grapes (1037.0 and 5759.10 mg/100 g d.m.) from different varieties and locations (Samoticha et al., 2017), while the lowest values were detected in samples pretreated with USW (563.91 ± 10.61 mg GAE/100 g d.m.) and USEtOH (780.25 ± 31.94 mg GAE/100 g d.m.) ($p < 0.05$) before drying. However, a statistically significant difference was observed in TPC of USEtOH- and USW-pretreated grapes ($p < 0.05$), with the former showing higher levels. The mechanical disruption caused by ultrasound may have led

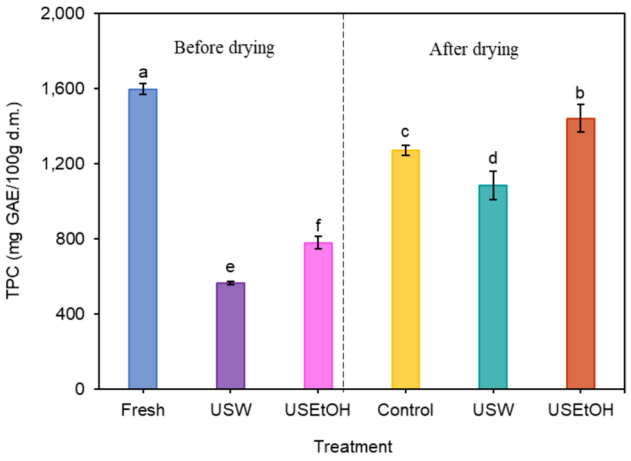

Fig. 3 Total phenolic content in fresh grapes, pretreated grapes before drying and dried grapes expressed as mean values ± standard deviation. Pretreatments were performed with ultrasound and water (USW) or ethanol (USEtOH). Different letters indicate statistically significant differences determined by Tukey's test ($p < 0.05$)

to the release of phenolic compounds from plant cells, making them more susceptible to degradation or loss prior to the drying process (Xu et al., 2022). After drying, TPC increased in all pretreated samples ($p < 0.05$), which suggests that the drying process led to a concentration effect in pretreated grapes, resulting in higher levels of phenolic compounds per unit dry weight in grapes. During drying, phenolic compounds may have been involved in further chemical reactions due to increased temperature and exposure to air, which may have led to the formation of new phenolic compounds or modification of existing ones, resulting in an increase in TPC after drying (Bai et al., 2023).

Despite the reduction of TPC in samples after the pretreatment, the values obtained after drying were high for a typical dried product. The USEtOH treatment had a positive effect on the preservation of phenolic compounds compared to the USW one ($p < 0.05$). The preservation of bioactive compounds may have been enhanced by ethanol due to its ability to reduce drying time, thereby minimizing the exposure of the product to hot air (Araújo et al., 2022). According to Ren et al. (2022), the combination of ethanol and ultrasound may have a synergistic effect on the retention of phenolic compounds. The osmotic dehydration effect of ethanol and the enhanced mass transfer due to ultrasound could work together to preserve the bioactive compounds.

Antioxidant Activity

Figure 4 shows the antioxidant activity (AA) of grapes determined by the ABTS and FRAP assays, considering the different pretreatments. As for the ABTS assay, the AA of fresh sample (753.66 ± 20.51 (μM Trolox/g d.m.) was higher

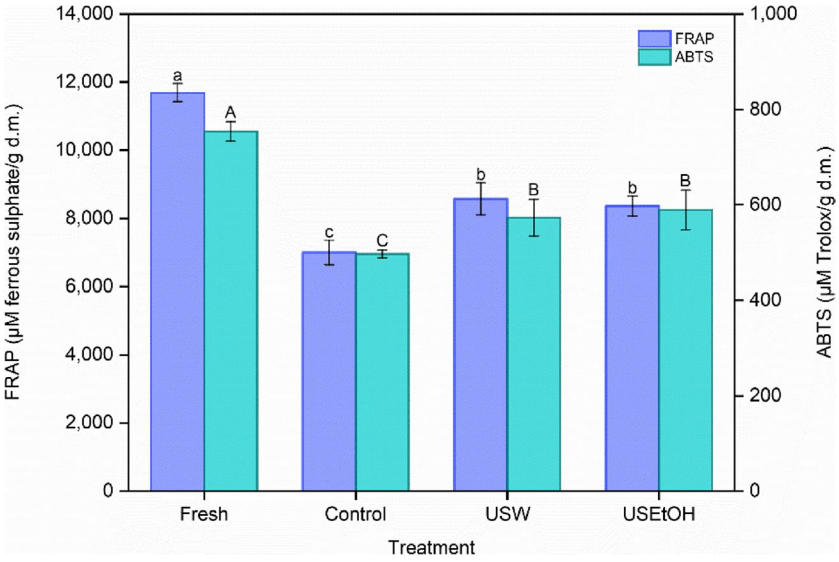

Fig. 4 Antioxidant activity of fresh and dried grapes determined by the FRAP and ABTS assays expressed as mean values ± standard deviation. Different letters indicate statistically significant differences determined by Tukey's test ($p < 0.05$)

than that of pretreated dried grapes (496.71–589.16 µM Trolox/g d.m.). Similarly, the FRAP AA, which achieved 11,689.26 ± 26.82 µM ferrous sulfate/g d.m. in fresh grape, decreased to 7003.39–8573.08 µM ferrous sulfate/g d.m. in the dried ones. These results suggest that thermal degradation of bioactive compounds, due to long exposure to high temperature and hot air during drying, was responsible for the significant reduction ($p < 0.05$) in AA assessed by both assays. In addition, the AA values of control samples by both assays were lower than those of grapes pretreated with USW and USEtOH, with no significant differences between pretreatments.

According to previous studies, ultrasound pretreatment can enhance AA of fruits and vegetables. For instance, Xu et al. (2021) found that ultrasound pretreatment improved AA in vacuum freeze-dried okra samples. Such an effect of ultrasound pretreatment can be attributed to several mechanisms. Fonteles et al. (2016) pointed out that it is able to disrupt the structure of plant tissues, such as the parenchyma of cashew bagasse, thereby leading to a decrease in resistance to water diffusion, an increase in the rehydration rate and, above all, an easier release of bioactive compounds and a higher AA. Tao et al. (2016) also reported that ultrasound pretreatment enhanced the convective drying kinetics and reduced total energy consumption while maintaining the quality properties such as color and AA in mulberry leaves. Similarly, multifrequency ultrasound pretreatment on sweet potatoes demonstrated that moderate ultrasound frequency improved phytochemical properties and AA (Rashid et al., 2019). Based on these considerations, we can conclude that, regardless of the ultrasonic propagation medium used (water or ethanol), ultrasonic pretreatment had a positive impact on AA of dried grapes, despite the reduction caused by the drying process.

Fourier Transform Infrared Spectroscopy

Fourier transform infrared (FTIR) spectroscopy was used to detect the main functional groups of compounds contained in samples and their possible changes caused by the different pretreatments and convective drying. One can observe in Fig. 5 broad bands between 3500 and 3000 cm^{-1} corresponding to the OH groups of water and carbohydrates (Kalušević et al., 2017), a deformation in the range 3000–2800 cm^{-1} that can be ascribed to the doublet of the aldehyde C–H bond as well as O–H and NH_3 groups of amino acids, phenolics, carboxylic acids, and carbohydrates (Tahir et al., 2017). The drying process resulted in intense peaks at 2924 and 2846 cm^{-1}, which suggests the occurrence of changes in chemical composition through oxidation, thermal degradation, and enzymatic and non-enzymatic reactions such as the Maillard reaction (Zhao et al., 2018).

Control and USW-pretreated samples showed more intense peaks when compared to the USEtOH-pretreated ones, as the likely result of the longer heating time. Amanor-Atiemoh et al. (2020), who studied the influence of USEtOH pretreatment duration on apple slices dried at different temperatures, observed that higher temperatures and longer pretreatment led to intense peak deformation as the result of degradation of some functional groups relating to nutrient composition.

The small band at 1732 cm^{-1} could be assigned to C = O stretching (do Carmo et al., 2021), whereas those observed at 1640 cm^{-1} may have been due to the deformation of H–O–H, indicating the presence of residual water. The peak intensity decreased with the reduction of water content due to the drying process. Stretching vibration at 1100 to 900 cm^{-1} can be attributed to carbohydrates (Chen et al., 2021), while the 820 cm^{-1} band to C–H deformation, which is characteristic

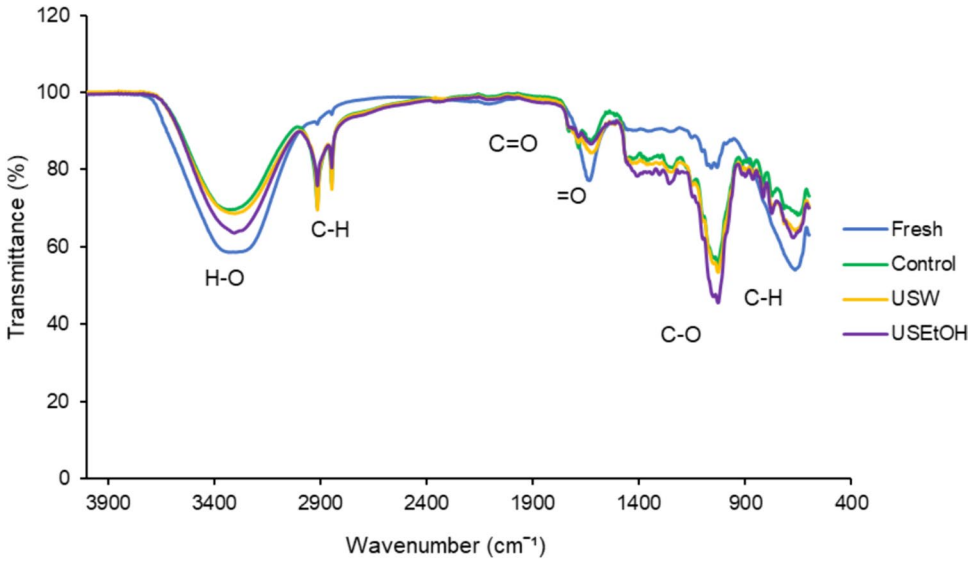

Fig. 5 Fourier transform infrared (FTIR) spectra of fresh and dried pretreated grapes. Pretreatments were performed with ultrasound and water (USW) or ethanol (USEtOH)

of the phenolic compounds (Tahir et al., 2017). All dried samples exhibited similar FTIR absorption patterns.

Principal Component Analysis

Principal component analysis (PCA) was employed to assess the effects of pretreatments on bioactive compounds, antioxidant activity, chemical group alterations, and quality parameters of dried grapes. Figure 6 shows that the two principal components (PC1 and PC2) accounted for 95.5% of the total data variance, highlighting the method's efficacy. Principal component 1 (PC1), responsible for the majority of data variance (88.3%), emphasized the predominant influence of variables related to chemical composition, such as TPC and characteristic functional groups identified by C–H and C = O bonds. This component also underscored the importance of physical characteristics, such as moisture content and water activity, indicating a strong correlation between chemical composition and sample physical properties. On the other hand, principal component 2 (PC2), although capturing a significantly smaller amount of variance (7.2%), emphasized the relevance of properties related to grape processing, such as drying time.

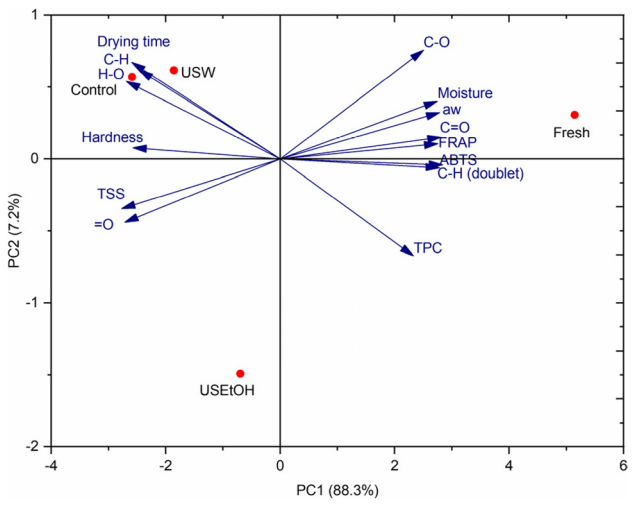

Fig. 6 Principal component analysis (PCA) of the first two components (PC1 and PC2) applied to fresh and differently pretreated grapes

Interpretation of the biplot graph revealed that control and USW-pretreated samples displayed a positive correlation and were closely linked to drying time, C–H functional groups, O–H bonds, and firmness. Additionally, soluble solids and the = O group showed a positive correlation, suggesting that the presence of = O groups in water-soluble compounds may be responsible for an increase in soluble solids. Variables such as AA by FRAP and ABTS assays, moisture content, water activity, and various types of chemical bonds, including C–H, C = O, and C–O, also exhibited a positive correlation and were clustered in the same quadrant as the fresh sample. This correlation was indeed expected from the fact that all these variables are directly influenced by the high sample moisture content. The presence of these functional groups and bonds can directly influence the antioxidant properties and overall chemical composition of samples. Additionally, it was observed that the sample pretreated with USEtOH had a higher negative score on PC2, indicating its distinct chemical characteristic in terms of pretreatment. In summary, PCA demonstrated that pretreatment with ultrasound and ethanol influenced phenolic content and AA, providing valuable insights into the chemical diversity and processing influence on analyzed samples, and highlighting potential areas for further investigation in subsequent studies.

Conclusions

This study dealt with the effects of using ultrasounds in two different media (water and ethanol) as pretreatment before convective drying of BRS Vitória grapes. Our results showed that the pretreatment with ultrasound and ethanol (USEtOH) improved the drying rate and reduced the drying time by up to 64%, as the likely result of its ability to modify the physicochemical properties and increase the diffusion of moisture within the material. Samples subjected to rehydration at temperature of 50 °C and pretreated with USEtOH showed a higher rehydration rate compared to the others. Furthermore, the Weibull model was found to best fit the hydration results. Regarding quality parameters, pretreatments had a significant impact on the soluble solid content and color change of the grapes before drying. The USEtOH pretreatment led to a significant reduction in grape water activity and contributed to color change after drying. On the other hand, the use of ultrasound in water had an effect on hardness, the higher values of which may have caused further structural changes. The total phenolic content (TPC) decreased after ultrasound pretreatments. In contrast, the drying process after both pretreatments increased TPC in samples. The TPC of USEtOH-pretreated sample was better preserved than that of the USW-pretreated one. The drying process greatly affected the antioxidant activity by both the ABTS and FRAP assays, but the ultrasound pretreatment minimized these losses. In addition, pretreatments preserved the various functional groups identified by FTIR spectroscopy. On the whole, the results of this study showed that USEtOH pretreatment had a positive impact on drying, which could be used as a highly efficient grape drying technology. Further research could focus on the costs of dried products and the recovery of waste ethanol after pretreatment.

Author Contribution Nathalia Barbosa da Silva: conceptualization, investigation, methodology, validation, data curation, resources, writing—original draft, visualization.

Attilio Converti: validation, writing—review and editing.

Maria Inês Sucupira Maciel: conceptualization, validation, formal analysis, resources, writing—original draft, writing—review and editing, supervision, project administration, funding acquisition.

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Data Availability Data will be made available on request.

Declarations

Ethics Approval Not applicable.

Consent to Participate Not applicable.

Consent for Publication Not applicable.

Competing Interests The authors declare no competing interests.

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