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## Chapter

# Effects of Pretreatments with Ethanol and Ultrasound on Convective Drying of BRS Vitória Grapes

*Nathalia Barbosa da Silva, Patrícia Moreira Azoubel  
and Maria Inês Sucupira Maciel*

## Abstract

The objective of this study was to evaluate the effect of ethanol and ultrasound as pretreatment to improve the convective drying of the BRS Vitória grape. The drying kinetics, rehydration, quality parameters, and phenolic compounds were evaluated. Before drying, grapes cv. BRS Vitória was ultrasound treated using two separate means, with ethanol (99.5% v/v) and distilled water. After pretreatment, the grapes were dried at 60°C and 0.1 m/s. The Logarithmic model provided a better prediction to describe the drying of grapes. Peleg's model showed satisfactory adjustments to predict rehydration. Compared to the Control, pretreatment using the combination of ultrasound and ethanol decreased the drying time of the grapes by 61%. The pretreatments did not influence in quality parameters. In contrast, phenolic retention was observed in samples with ethanol. These results open new perspectives on the drying process and product quality by combining ethanol and ultrasound.

**Keywords:** ultrasound, dehydration, ethanol pretreatment, raisin, logarithmic model

## 1. Introduction

Grape is a berry belonging to the Vitaceae family and is widely cultivated and frequently consumed in the world. According to the Food and Agriculture Organization (FAO), its world production in 2020 was approximately 100 million tons. The principal producers are China, Italy, Spain, and France. Currently, Brazil occupies the 15th position of grape producers, with a production of 1,435,596.00 tons in 2020 [1].

In Brazil, grapes are consumed in fresh or processed form as juices, vines, jams, and raisins. Part of the production of grapes in Brazil comes from the São Francisco valley, a region in northeastern Brazil with productive potential for different grape cultivars [2]. Therefore, Research Institutions have been developing grape cultivars adapted to Brazilian conditions to meet the high demand of the foreign market [3].

Grape cv. BRS Vitória was developed by a Brazilian agricultural research company (EMBRAPA) in 2012 to increase the production and improve climate adaptation of grapes in

the country. Seedless grape, the productivity of this cultivar can exceed 30 t/ha and shows good tolerance to berry splitting and downy mildew. The berry is spherical, black in color, with thick and resistant skin and colorless pulp. This fruit could provide health-related benefits (rich in phenolics, anthocyanins, and flavonoids with antioxidant properties) [4].

However, the grapes have a high moisture and sugar content, reducing the shelf life of the fruit [5]. Drying is one of the most used conservation methods to increase the shelf life of perishable foods such as grapes. Drying reduces the food moisture content to a level that allows safe storage for an extended period, reducing weight and volume, and minimizing packaging, storage, and transport costs [6].

For food drying to occur effectively, it is necessary to evaluate the following issues: the drying kinetics and factors that affect the drying rate; product quality, since water removal is not the only consequence of the process. Other important quality-related changes in taste, flavor, appearance, texture, structure, and nutritive value may occur in the course of drying [7].

The intrinsic characteristics of the berries also influenced the drying process. Grapes have waxy skin, which makes it difficult to mass transfer [8, 9]. To remove the waxy layer and accelerate the dehydration process of the grapes, several pretreatments have already been applied and investigated, such as blanching, the alkaline emulsion of ethyl oleate solution (AEEO), abrasion, and carbonic maceration [10–12].

Some novel non-thermal technology like ultrasound has been employed to enhance the drying process. This technology could be used with pretreatment for their benefit in enhancing heat and mass transfer in the course of dehydration [13–15]. Ultrasonic waves cause structural changes in the products, enabling increased permeability of the material. This effect can be obtained due to the “sponge effect,” cavitation phenomenon, and the effects accompanying cavitation, such as the formation of microchannels, facilitating mass, and/or heat transfer [16]. Ultrasound applications allow reducing drying time and energy consumption, obtaining high-quality dried materials [17]. This technology has been applied as a pretreatment in the drying of sweet potatoes [18], bitter melon [19], and kiwifruits [20]. The studies revealed that ultrasound pretreatment was effective to improve the process.

There are different types of immersion mediums used in ultrasound. Ethanol is an organic solvent with lower surface tension than water and facilitates the solvent into the food. Ren et al. [21] investigated the effects of different pretreatment methods on the drying process and the quality of catalytic infrared dried ginger slices. They observed sample pretreatment by ethanol + US had the highest drying efficiency and highest bioactive content retention. However, no studies have examined the effect of ultrasound combination as pretreatment on drying kinetics, quality parameters, and phenolic compounds from grapes.

Thus, the objective of this study was to evaluate the application of ultrasound as pretreatment to improve the convective drying of BRS Vitória grapes. For this purpose, the effect of an aqueous medium (ethanol and water) on drying kinetics, quality parameters, and phenolic compounds of raisins have been studied.

## **2. Materials and methods**

### **2.1 Materials**

For this study, grape cv. BRS Vitória was produced in the São Francisco Valley region (Latitude 09° 09' South; Longitude 40° 22' West). The grapes were washed

to remove surface impurities and sanitized with sodium hypochlorite (200 ppm) for 10 minutes. Then they were dried with absorbent paper, packed in polyethylene bags, and stored at  $-18 \pm 1^\circ\text{C}$ , until use.

## 2.2 Pretreatments

The pretreatments were conducted to evaluate the effect of ultrasound with different solvents (ethanol and distilled water). The sample (100 g) was placed in a beaker containing 200 mL of ethanol (99.5% v/v) encoded as US+ETOH. This beaker was then positioned in a thermostatic bath to maintain  $-5^\circ\text{C}$  during sonication. The same process was conducted with 200 mL of distilled water and encoded as US+WATER. An ultrasonic probe (QR1000 Ultronique, Ecosonics, Brazil) with a constant frequency of 20 kHz, maximum power of 550 W, and microdot with a diameter of 25.4 mm was used. The operating time on the ultrasound was 30 minutes.

## 2.3 Drying process

Drying was performed for pretreated and untreated (control) grapes at  $60^\circ\text{C} \sim 1 \text{ m/s}$ . For each batch, 100 g of grapes were placed on a metal net in a drying oven (MA035, Marconi, Brazil), with air circulation and renewal. All drying processes were performed by periodic weight (every 1 h). The initial moisture content was determined according to AOAC [22]. All experiments were repeated three times at the respective temperature, and the average measurements are contained within this study.

## 2.4 Mathematical modeling

To calculate the moisture content (MR) of the grape, the following equation was used (1).

$$MR = \frac{Mt - Me}{M_0 - Me} \quad (1)$$

Where Mt., M0, and Me are the moisture content at a given drying time (g water/100 g dry matter), the initial moisture content (g water/100 g dry matter), and the balance moisture content (g water/100 g dry matter), respectively. The drying curves were fitted with eight distinct thin layer dehydration equations to test which system most accurately described the drying process. These equations are listed in **Table 1**.

Various statistical parameters including the Coefficient of Determination (R<sup>2</sup>) and root mean square error (RMSE) were used to describe the best fit. The higher value of R<sup>2</sup> and the lower value of RMSE indicate the goodness of fit. R<sup>2</sup> and RMSE equations can be described by eqs. (2) and (3).

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

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (MR_{pre,i} - MR_{exp,i})^2} \quad (3)$$

Models	Equations	References
Newton	$MR = \exp.(-k t)$	[23]
Page	$MR = \exp.(-k t n)$	[24]
Henderson and Pabis	$MR = a \exp.(-k t)$	[25]
Logarithmic	$MR = a \exp.(-k t) + c$	[26]

**Table 1.**  
*Mathematical models provided by several authors for drying curves.*

Where  $N$  and  $z$  are the number of experimental data values and the number of constants, respectively.  $MR_{exp,i}$ ,  $MR_{pre,i}$ , and  $MR_{exp}$  are the experimental moisture ratio, predicted moisture ratio at time  $t$ , and the mean of experimental moisture ratio, respectively.

## 2.5 Quality parameters

The quality parameters were evaluated in fresh and processed samples. For quality analyses, grapes were dried until a final moisture content of 20% (wet basis), which is a value within the range allowed by Brazilian legislation [27]. Water activity ( $a_w$ ) was determined in three repetitions for every sample (fresh and dried grapes) at a temperature 25°C, using equipment Aqualab 4TE (Meter group, USA) according to the manufacturer's instructions. One sample of the tested material was placed into the chamber of the apparatus and closed. After about 5 min, the results were determined [28]. The soluble solids data were obtained using a digital refractometer (r2 i300, Reichert, USA). Juice from the sample was extracted and inserted into the equipment for reading, and the results were expressed in °Brix [29]. All measurements were carried out in triplicate.

### 2.5.1 Texture

The texture was evaluated using a texture meter (CT3–1000, Brookfield, USA), with the aid of data acquisition software of the same equipment brand. The hardness of fresh grapes was evaluated according to the methodology described by Rolle et al. [30]. For raisins, the method described by Wang et al. [31] with some modifications was used. Compression tests were carried out by compressing the raisin to 5 mm on the mid-axis with a cylindrical probe of 25.4 mm in diameter, with a waiting time of 5 seconds between the two bites, and at a speed rate of 1 mm.s<sup>-1</sup> to determine hardness.

### 2.5.2 Color

The color parameters of grapes were determined by using a colorimeter (CR-400, Konica Minolta Sensing, Japan). The samples were analyzed and expressed as color coordinates in the CIELAB space where  $L^*$  (brightness–darkness),  $a^*$  ( $+a^*$ : red,  $-a^*$ : green), and  $b^*$  ( $+b^*$ : yellow,  $-b^*$ : blue). White tile was used as a standard ( $Y = 93,40$ ;  $x = 0,3136$ ;  $y = 0,3196$ ). The parameters  $L$  (Luminosity),  $a^*$ , and  $b^*$  allowed the calculation of the Hue angle, that is, the color tone using the following eq. (4) [32]:

$$h = \tan^{-1} \left( \frac{b^*}{a^*} \right) \quad (4)$$

## 2.6 Total phenolic content

The phenolic compounds were extracted using an ultrasonic bath (USC-2850A, Unique, Brazil) and as a solvent, ethanol (60% acidified with 0.1% HCL). The total phenolics content present in this extract was quantified according to the methodology proposed by Wettasinghe & Shahidi [33] using Folin-Ciocalteu reagent and gallic acid as a reference standard. 0.5 mL of the extract was homogenized with 8 mL of distilled water, 0.5 mL of Folin Ciocalteu reagent, and 1 mL of saturated sodium carbonate solution. The flasks were shaken and then kept at rest, in the dark, for 1 h. The absorbance at 765 nm was measured using a UV-vis spectrophotometer (UV-1900i, Shimadzu, Japan), and the results were expressed in mg of total phenolics in gallic acid equivalent (EAG) per 100 g of fresh grape and 100 g of raisin of dry matter.

## 2.7 Statistical analysis

Nonlinear regression was used to find model parameters to fit drying kinetics data. For this, Origin Pro 2019b software (Origin lab Inc., USA) was used. All determinations were performed in triplicate, and the data were submitted to the two-way Analysis of Variance (ANOVA) and Tukey post hoc test at a 5% significance level using Statistica 10.0 software (StatSoft Inc., USA).

## 3. Results and discussion

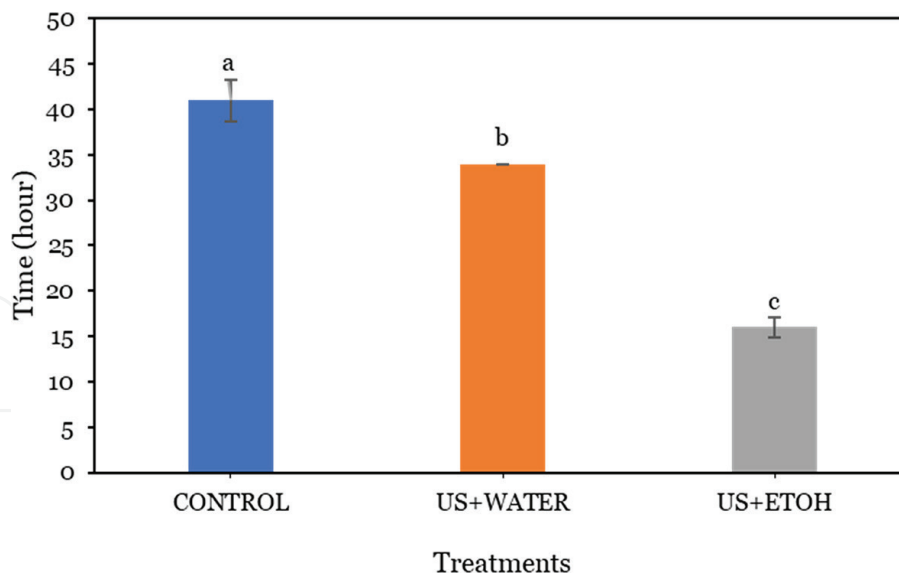
### 3.1 Drying kinetics

Fresh grape samples used in this work presented a moisture content of  $84.33 \pm 0.9\%$  (w.b), which was in the range ( $80.04 \pm 1.10$ – $84.01 \pm 1.6\%$ ) reported by Okzan et al. [34] and Adietta et al. [5] for black “Isabel” and Red Globe grapes, respectively. Before starting drying, the ultrasound with different mediums (water and ethanol) was applied. The effect of each pretreatment on the processing time was compared with the control treatment, as shown in **Figure 1**.

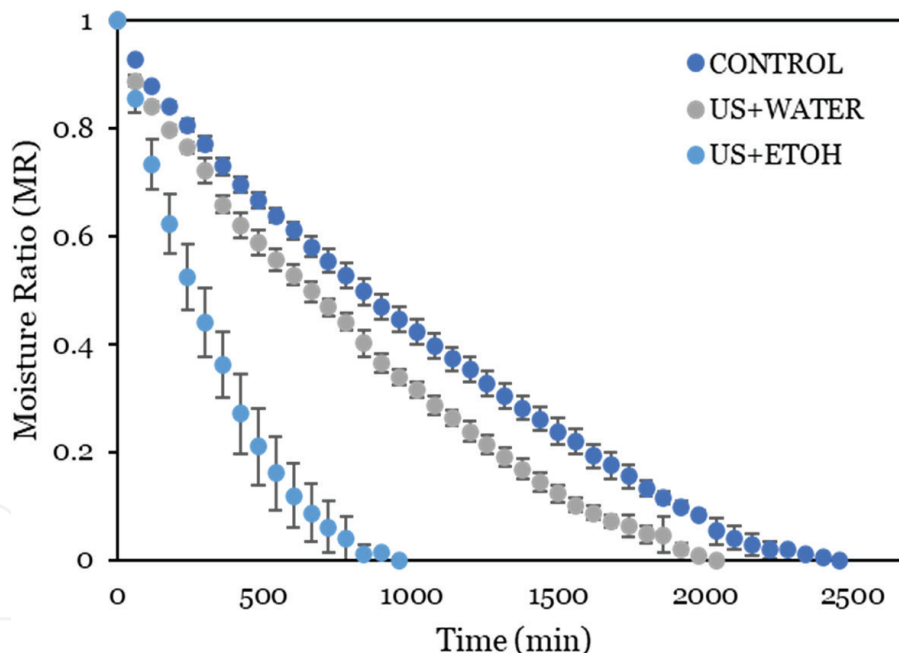
Grapes have high moisture and require a long drying time. To reach equilibrium moisture, the time required for the control sample under drying conditions ( $60^{\circ}\text{C}$  and  $1\text{ m/s}$ ) was 41 hours. **Figure 1** shows there was an effect on drying time reduction, indicating that ethanol was the medium that reduced the time by 61%, with a processing time of 16 hours, while the medium with water reduced the drying time by 17% (34 hours).

Rojas, Silveira and Augusto [35] studied the application of ethanol and ultrasound combined as pretreatment in the drying kinetics of pumpkin using air at  $50^{\circ}\text{C}$ . The authors observed that the combination of ethanol and ultrasound for 30 minutes reduced the drying time of pumpkin by 59% compared to the control. Da Cunha et al. [36] evaluated the effectiveness of the use of ethanol, ultrasound, and/or vacuum as a pretreatment to melon drying. They observed a reduction of 44.62% in drying time with the use of ultrasound associated with ethanol. The authors reported a positive effect on the drying rate with the combination of medium and ultrasound, similar to the results found in this study.

The moisture kinetics of grape cv. BRS Vitória under different treatments is illustrated in **Figure 2**. The moisture ratio with time showed an exponentially decreasing trend in all treatments. **Figure 2**, it was observed similar behavior on the drying curve



**Figure 1.** Drying time reduction for all treatments. The values presented refer to the arithmetic mean of three determinations  $\pm$  standard deviation. Equal letters do not differ statistically from each other at a 5% probability level by the Tukey test (ANOVA  $p < 0.05$ ).



**Figure 2.** Convective drying kinetics for all treatments. The values presented refer to the arithmetic mean of three determinations  $\pm$  standard deviation.

for control and US+WATER samples. However, the longest drying time was obtained for the control sample. Drying kinetics is an important task to observe the behavior of the product during drying. The use of mathematical models is useful to design drying systems and analyze the complex phenomena of heat and mass transfer [37]. **Table 2** shows the statistical parameters estimated for the comparison between the four mathematical models of drying.

The best mathematical model was selected based on a comparison of the statistical values of the coefficient of determination ( $R^2$ ) and root mean square error (RMSE).

Treatment	Models*	Constants				R <sup>2</sup>	RMSE
		<i>k</i>	<i>n</i>	<i>a</i>	<i>c</i>		
Control	Newton	0.0014	—	—	—	0.966	0.049
	Page	0.0015	0.9862	—	—	0.966	0.049
	Henderson e Pabis	0.0013	—	0.9447	—	0.971	0.045
	Logarithmic	0.0007	—	1.1526	-0.2671	0.991	0.025
US+ETOH	Newton	0.0033	—	—	—	0.986	0.035
	Page	0.0009	1.2093	—	—	0.996	0.020
	Henderson e Pabis	0.0034	—	1.0400	—	0.988	0.033
	Logarithmic	0.0028	—	1.0828	-0.0685	0.995	0.021
US+WATER	Newton	0.0012	—	—	—	0.962	0.057
	Page	0.0003	1.1965	—	—	0.981	0.039
	Henderson e Pabis	0.0012	—	1.0533	—	0.971	0.053
	Logarithmic	0.0004	—	1.7728	-0.7994	0.999	0.008

\* All models were significant  $p < 0.05$ .

**Table 2.**

Estimated parameters, coefficient of determination (R<sup>2</sup>) and root mean square error (RMSE), for mathematical models with and without ultrasound pretreatment.

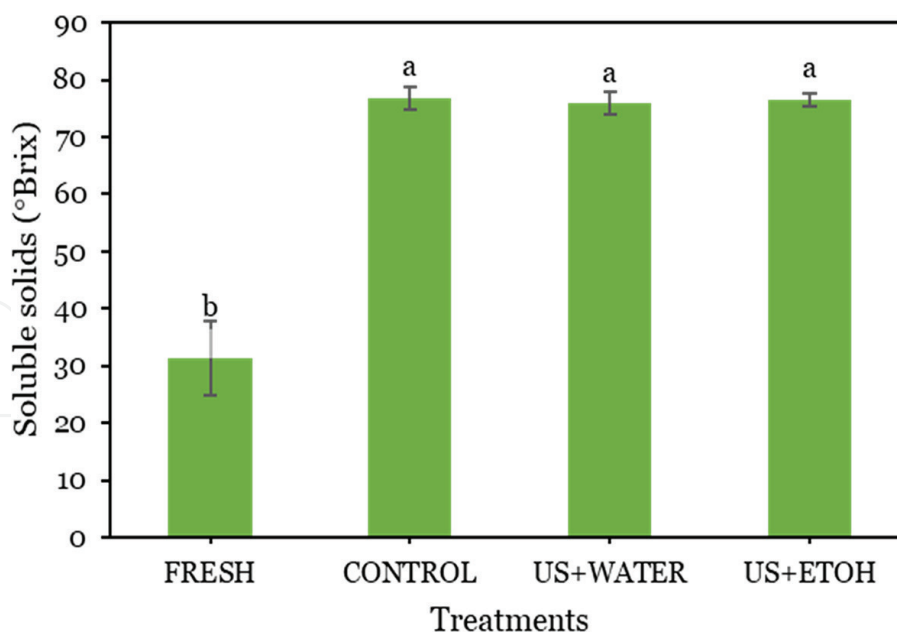
The models fitted to the experimental data presented R<sup>2</sup> values between 0.962 and 0.999 and the RMSE values were between 0.008 and 0.057, indicating that a good fit was obtained for all the proposed models. The logarithmic model presented the best fit for the drying processes performed in different treatments, indicating that in this model, changes in the moisture content of the grapes could be predicted with the drying time. The values of the constant *k* of the Logarithmic model indicated that with the decrease in the drying time, the constant increases. This behavior was observed with the pretreatment with ultrasound-assisted and ethanol medium.

### 3.2 Quality parameters

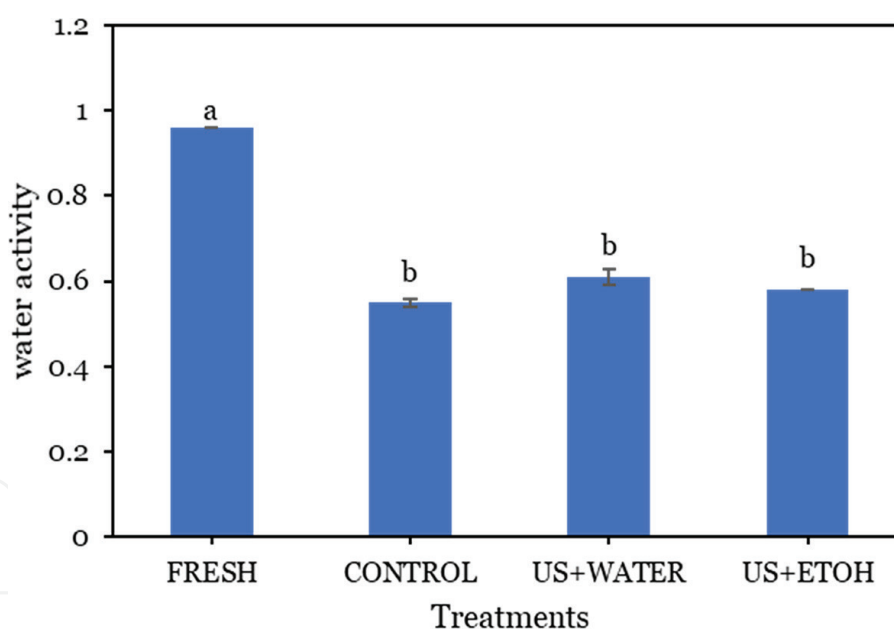
The results of the soluble solids content of fresh grapes and raisins in different treatments shown in **Figure 3**. In fruit drying, with the removal of moisture, the food content is concentrated and increases in the soluble solids content [38]. The soluble solids of BRS Vitória grapes dried with different treatments increased significantly than fresh grapes ( $p < 0.05$ ). However, there was no difference between the treatments used and the control sample ( $p > 0.05$ ). This result indicates that the media used do not affect the soluble solids content.

Water activity is an intrinsic factor in the food and indicates the free water contained in the food. This parameter is relevant to assess the stability of the product after processing [39]. Water activity below 0.6 can prevent the growth of microorganisms, increasing the shelf-life of dehydrated products during storage [40]. **Figure 4** compares the water activity of different treatments and fresh grapes. The water activity content for fresh grapes was 0.96. The treated samples ranged from 0.55 to 0.59 after drying. All dehydrated samples obtained water activity results below 0.6, guaranteeing the stability of the raisin. No significant differences were found between samples treated with different mediums and control samples. Similar behavior occurred in the soluble solids content.





**Figure 3.** Soluble solids content of BRS Vitória grapes in different treatments. The values presented refer to the arithmetic mean of three determinations  $\pm$  standard deviation. Equal letters do not differ statistically from each other at a 5% probability level by the Tukey test (ANOVA  $p < 0.05$ ).



**Figure 4.** Water activity of BRS Vitória grapes in different treatments. The values presented refer to the arithmetic mean of three determinations  $\pm$  standard deviation. Equal letters do not differ statistically from each other at a 5% probability level by the Tukey test (ANOVA  $p < 0.05$ ).

The instrumental color is one of the most important parameters to analyze the drying process. Color is measured using the  $L^*a^*b^*$  system, in which  $L^*$  indicates lightness,  $a^*$  indicates color from green ( $-a^*$ ) to red ( $a^*$ ), and  $b^*$  indicates color from blue ( $-b^*$ ) to yellow ( $b^*$ ). The changes in the values of the color parameters, mainly in the  $a^*$  and  $b^*$  coordinates, it is possible to predict pigmentation changes or the occurrence of enzymatic or non-enzymatic browning reactions [37].

The results of the color parameters are shown in **Table 3**. The luminosity value ( $L^*$ ) of all samples decreased with drying. This result indicates that the raisins became

	Fresh	Control	US+WATER	US+ETOH
L*	2.29 ± 0.84	19.57 ± 2.39	21.42 ± 18.54	18.50 ± 1.59
a*	- 0.66 ± 0.17 <sup>b</sup>	1.58 ± 1.28 <sup>a</sup>	1.05 ± 0.20 <sup>a</sup>	1.37 ± 0.08 <sup>a</sup>
b*	1.61 ± 0.08	1.54 ± 0.15	1.44 ± 0.40	1.42 ± 0.32
Hue	112.00 ± 5.11 <sup>a</sup>	42.23 ± 26.23 <sup>b</sup>	53.09 ± 1.62 <sup>b</sup>	45.52 ± 7.33 <sup>b</sup>

*\*\*ANOVA p value < 0.05. Means on lines followed by the same letters do not differ statistically from each other at the 5% probability level by the Tukey test.  
 \*\*\*values without letters were not significant p > 0.05.\*The values presented refer to the arithmetic mean of three determinations ± standard deviation.*

**Table 3.**  
 Color parameters of BRS Vitória grape in different treatments.

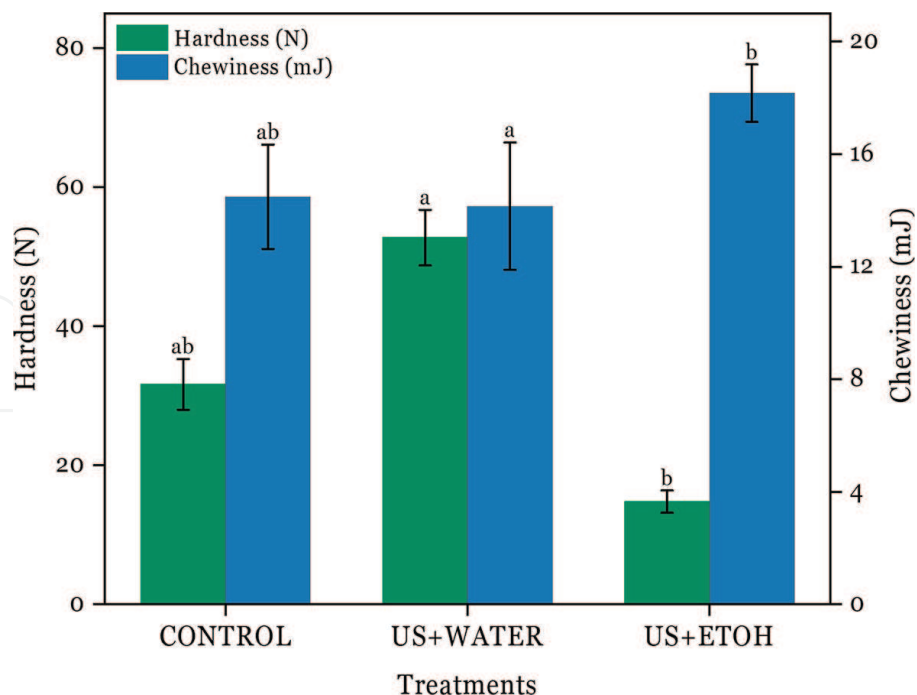
opaquer. However, this coordinate showed no statistical difference. The values of a\* coordinate for the control, ethanol, and water samples increased compared to fresh grapes, but no significant difference between the treatments (p > 0.05). There were no significant changes in the b\* coordinate. The values obtained of hue angle for fresh grapes differed statistically from raisins (p < 0.05), showing a change in hue as an effect of drying. The results indicate that the drying of the grape causes changes in the luminosity, making it darker, with reddish and bluish nuances and with changes in tonality, regardless of the treatment used.

The hardness and chewiness of dried samples were evaluated by texture profile analysis TPA (**Figure 5**). The dried grapes presented values between 14.77 N and 31.65 N and the US+WATER treatment showed the highest value of hardness. There was a significant difference between the two treatments using ultrasound (p < 0.05). In the drying process, structural changes occur with the shrinkage of the product. The removal of moisture causes the surface of the sample to harden. Thus, the adhesive force between the cells forms a compact tissue when the water is removed [41]. It was observed that the raisin treated with ultrasound and ethanol is the one that necessitates less force for deformation.

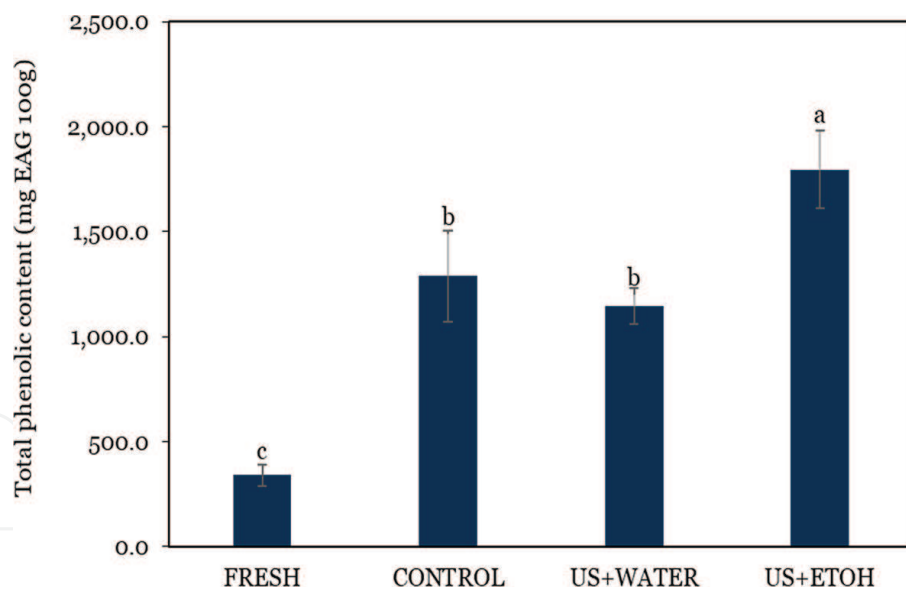
In chewiness, raisins using ultrasound with ethanol had the highest average (p < 0.05), showing that the sample treated with ethanol needs more energy for the mastication forces. According to [42], the application of ultrasound pretreatment can cause significant changes in physical characteristics such as the hardness and chewiness of fresh food when subjected to drying. This behavior occurs due to the simultaneous transfer of heat and water during drying leading to tension and shrinkage, increasing the texture of the dehydrated products. However, in sonicated fruits, most of the cell walls are broken during ultrasonic vibration, and there is a network of micro-channels in the plant tissue, which favors the formation of a softer dried product.

### 3.3 Total phenolic content

The results of the total phenolic contents of BRS Vitória grape are presented in **Figure 6**. Total phenolic content was in the range of 340.98–1794.80 mg EAG/100 g. The TPC concentration of grape BRS Vitória increased with the drying process. Our results were in agreement with Serni et al. [43] determined TPC in dried grape pinot blanc skin during ripening in the range from 582.33 to 705.50 mg GAE/100 g and Ozakan et al. [34], who reported TPC for black Isabel grape of 351.89 ± 35.12 to



**Figure 5.** Texture profile analysis (hardness and chewiness) of BRS Vitória grape in different treatments. The values presented refer to the arithmetic mean of three determinations  $\pm$  standard deviation. Equal letters do not differ statistically from each other at a 5% probability level by the Tukey test (ANOVA  $p < 0.05$ ).



**Figure 6.** Total phenolic content in BRS Vitória grape in different treatments. The values presented refer to the arithmetic mean of three determinations  $\pm$  standard deviation. Equal letters do not differ statistically from each other at a 5% probability level by the Tukey test (ANOVA  $p < 0.05$ ).

1101.61  $\pm$  35.12 mg GAE/100 g. However, in this study, all drying methods reduced TPC concentration significantly. It should be noted that the US+ETOH treatment increased the TPC compared to the control and US+WATER samples ( $p < 0.05$ ). It is due to the shortest drying time observed for US+ETOH treatment, as fewer phenolics were exposed to the heat, which increased the retention. Ren et al. [21] and Granella et al. [17] observed similar behavior for Chinese ginger and banana slices, respectively.

## 4. Conclusion

This work evaluated the effect of ultrasound with different media (water and ethanol) as a pretreatment in the convective drying of the BRS Vitória grape. The pretreatment with ultrasound in the different media increased the efficiency of convective drying of the BRS Vitória grape, reducing its drying time by up to 61% using ethanol. In addition, it was observed that, of all the mathematical models evaluated, the Logarithm was the best adjusted to the grape drying process when compared to the other models. In quality parameters of the raisin, no significant differences were observed between the media used and the control sample regarding texture, color, soluble solids, and water activity. Compared to fresh, no loss of phenolic content in grapes after drying. Ultrasound with ethanol combined showed the highest phenolic content between the treatments. Therefore, pretreatment with ethanol proved to be effective in obtaining raisins, reducing the drying time, not altering the quality characteristics of the product, and promoted more retention of nutrients.

## Conflict of interest

The authors declare no conflict of interest.

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