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FIGURES

Figure 1. Percentage change in the tartaric acid (TA) and ascorbic acid (AA) content of the two varieties of grape with the different drying treatments. Different letters in the compounds indicate significant differences between treatments.

Figure 2. Percentage change in the sugar content of raisins dried by hot air with and without NaOH pretreatment (HA and HA+NaOH) and microwave with and without NaOH pretreatment (MW and MW+NaOH). Different letters in the compounds indicate significant differences between treatments.

Figure 3. Percentage change in the mineral content of both grape varieties for each drying treatment. Different letters in the compounds indicate significant differences between treatments.

Figure 4. Percentage change in total pectin (TP), oxalate soluble pectin (OSP) and water-soluble pectin (WSP) content of both grape varieties for each drying treatment. Different letters in the compounds indicate significant differences between treatments.

Figure 5. Percentage change in total phenolic content (TP,% of GAE) and antioxidant activity (AOA,% of Trolox) of raisins of both grape varieties for each drying treatment. Different letters in the compounds indicate significant differences between treatments.

Tables

Table 1. Mean values of water activity and main compounds in the Thompson seedless grape variety before and after each drying treatment. In parenthesis is the standard deviation.

Table 2. Mean values of water activity and main compounds in the Imperial Seedless variety before and after each drying treatment. In parenthesis is the standard deviation.

Table 3. Mean values of water activity and main compounds (referred to fresh fruit mass) of the two varieties of grapes dried by the two drying treatments. In parenthesis is the standard deviation.

Table 4. Maximum and minimum values of the fruit components (dry basis) in the two varieties of grapes dried through different drying treatments and in the commercial raisins. In parenthesis is the standard deviation.

Effects of drying and pretreatment on the nutritional and functional quality of raisins

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Abstract:

The close relationship between the consumption of fruits and health status stems from the nutritional and non-nutritional compounds found in fruits which play a key role in the prevention of different diseases. However, fruit processing and storage greatly affect fruit compounds. The aim of the present work was to study the influence of processing on the stability of macro and micronutrients present in grapes, with a view to recommending products that provide the highest nutritional quality and the best health conditions. The study focused on fruit dehydration treatments. Conventional and microwave-assisted air-drying processes were used to obtain raisins. Dehydration caused a decrease of all grape compounds studied excluding total phenols. Moreover, compared to conventional processing, microwave-assisted drying produced greater losses of ascorbic acid in the grape and increased pectin solubilization with a consequent change in texture. However the microwave-dehydrated samples showed higher antioxidant activity.

Keywords: microwave, air drying, pretratment, phenols, antioxidant activity, tartaric acid.

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1. Introduction

Traditionally raisins are obtained by sun drying of the fruit for eight to ten days, which substantially reduces water content. This drying method is cheap, but there is a risk of damage due to dust and insect infection (Pangavhane and Sawhney, 2002). An alternative to this is artificial drying. Convective drying is one of the oldest dehydration methods in which hot air passes through the fruit removing the water from the surface. This creates a diffusion gradient in the food that moves the water from the interior to the outer surface (Gowen et al., 2006). However, this process decreases the quality of the final product (Erentuk et al 2005). Moreover, dehydration causes damages in texture, color, taste and nutritional value of food due to the high temperatures and long drying times required in the process. According to Tarhan (2006), the dehydration of grapes affects their content of polyphenols, ascorbic acid and antioxidant activity. That is why efforts should be made to reduce drying times and decrease the temperatures used in the drying processes and, in this way, obtain better quality products. This has led to the development of less invasive technologies to reduce the moisture content of food. An example is the use of microwave energy alone or combined with hot air (Contreras et al., 2005 and 2007, Gowen et al., 2006).

Microwave drying is a technique that allows rapid dehydration and can be applied to certain foods, particularly fruits and vegetables (Zhang et al., 2006). The great interest in this technology is due to the high capacity of penetration of these waves, that heat not only on the surface but also inside the food. This speeds up the drying process and can improve the quality of the final product compared to other dehydration techniques like hot air drying (Contreras, et al., 2005 and 2007). Moreover, in microwave drying, heat is generated in the wet but not in the dry food areas, so that food areas with no water are not unnecessarily heated, which avoids the negative effects of heat on product quality.

(Bilbao, 2002, Martin, 2002). By contrast, microwave drying systems have the drawback that it is very difficult to know the distribution of the energy field, because it is modified by the introduction of a load in the system (Zhang et al., 2006). The combined use of microwaves and hot air drying improves final product quality (Ahrne et al., 2003, Contreras et al., 2005, Funebo et al., 2002, Piotrowski et al., 2004, Prothon et al., 2001, Raghavan and Silveira, 2001: Torringa et al., 2001).

On the other hand, the skin of some fruits such as grapes is covered by a waxy coating that reduces permeability and therefore hinders the loss of water (Tarhan, 2006). That is why prior to artificial drying other chemical and physical pre-treatments are used to enhance permeability by increasing the drying rate, while preserving the physical, chemical, nutritional and organoleptic qualities of the final product (Femenia et al., 1998).

The present work studies the changes in the nutritional and functional value of grapes as a result of convective and microwave-assisted hot air drying, with and without NaOH pretreatment to produce raisins

2. Materials and methods

2.1. Raw material

Grapes (Vitis vinifera) selected from the Imperial seedless and Thompson seedless varieties and purchased in supermarkets in the city of Valencia were used for the experiments. The grapes were stored in a refrigerator before handling (up to 12 h), rinsed with distilled water and dried with paper towels; the berries were then separated from the bunch and dry-treated. Additionally, commercial raisins were purchased at a local supermarket and compared with the raisins obtained experimentally in the laboratory.

2.2. Processing

The two grape varieties were treated using two drying methods: microwave-assisted hot air drying (MW) and hot air drying (HA); additionally the Imperial seedless grape variety was subjected to a pretreatment to shorten drying times, which consisted of dipping the berries in a NaOH solution (0.03%) at 95 º C for 45 s. In all cases the final moisture content was set at 30% for the dehydrated grapes. Next is the description of the procedure that was followed for each of these drying treatments.

For microwave drying a total of 100g of grapes was introduced in a laboratory dryer (Contreras et al, 2008). This device has a mechanism to control the microwave power (set at 0.2 W/g), air temperature (60 ºC), air velocity (1.6 m/s) and the evolution of the mass of the product over time with the help of an analytical balance. For hot air drying a laboratory dryer with larger sample capacity was used. A total of 450 g of grapes was introduced in the dryer, which could also control temperature and air velocity (60 °C, 10m/s) as well as the mass of the product by means of an analytical balance. The weight of the sample was recorded during the process and allowed for the calculation of the moisture content at each drying time providing the initial moisture content is known (Eq. 1).

$$X_{wt} = \frac{M_0 \cdot Xw_0 + \Delta P}{M_t} \qquad (1)$$

where:

Xwt = Moisture content at each drying time (g of water/g of product)

M_0 = initial grape mass (g)

Xw_0 = Initial moisture content (g of water/g of product)

ΔP = Mt-M0

99 Mt = grape mass at each drying time (g)

100

101 Using this equation, the drying process was stopped when the moisture content of
102 the dried product was approximately 30%. The drying times were: HA = 5 days, MW =
103 7.5 h, HA + NaOH = 34 h and MW + NaOH = 4.5 h.

104 **2.3. Sample Analysis**

105 All samples were analyzed in the moisture content (AOAC 20 013, 1997), the
106 soluble solids of the liquid phase of the samples (ºBrix) at 20 °C (refractometer Atago
107 NAR-3T, Japan) and water activity (a_w) (dew-point hygrometer GBX FA-st lab,
108 France). Total acidity was measured by titration with NaOH (0.1 N) and expressed in
109 mg of the main acid (tartaric acid, TA) (AOAC, 1997). Ascorbic acid (AA) was
110 determined by titration according to AOAC 985.33 (1997). The total pectin content was
111 analyzed by quantifying the galacturonic acid residues (AGU) following the procedure
112 used by Yu et al. (1996). To determine the AGU a Thermo Spectronic UV1
113 spectrophotometer was used for measuring absorbance at 520 nm. The determination of
114 phosphorus was analyzed by colorimetry, using the same spectrophotometer at 600nm.
115 Ca, K and Mg were calculated by high-performance anion-exchange chromatography
116 (HPAEC), using a Metrohm chromatograph (Herisau, Switzerland) and tartaric acid as
117 mobile phase (4 mmmol/l) and dipicolinic acid (0.75 mmmol/l) and a Metrosep C2-150
118 column (4x150 mm) with a particle size of 7□m. Like the minerals, the sugars were
119 determined by the same technique, using 0.1N NaOH as mobile phase and a Metrosep
120 Carb 1 column (4.6x250 mm) for carbohydrates with a particle size of 5□m. In both
121 cases the grapes were homogenized with an Ultra-Turrax T25 (Ika, Germany) and then
122 centrifuged at 4 °C and 10000 rpm for 10 min. The extraction for the quantification of
123 total phenols (TP) was carried out using the technique described by Peiró et al (2006).

The same extract obtained for TP quntification was used for the determination of antioxidant activity (AOA). The TP were quantified using the Folin-Ciocalteu test (Li et al, 2006) and expressed in mg of gallic acid/100g fresh grape. The antioxidant activity was determined by a modification of the spectrophotometric technique developed by Re et al. 1999, using the ABTS+ radical (Sigma) generated by 2.45mM potassium persulfate (K2S2O8). The results were expressed as antioxidant activity equivalent to mg of Trolox (TEAC) in 100g of fresh sample. All the experiments were replicated thrice.

3. Results and discussion

Tables 1 and 2 show the mean values of aw and the components analyzed in the samples of the fresh and treated grape varieties by different drying methods (MW, HA, and MW+NaOH and HA+NaOH). As expected, the decrease in moisture content caused a general increase in °Brix and, as a result, a decrease in water activity after dehydration with the drying methods under consideration.

Due to the variability in the initial composition of the fresh fruit, the gain or loss in the content of each compound was calculated in order to compare among the different dehydration treatments, taking into consideration the compound content in 100 g of fresh grapes and in the raisins referred also to 100 g of fresh grapes, according to Eq. 2 (Table 3). These data were used to calculate the percentage change in the content of each compound between the raisins and the untreated grapes, referred to that present in the untreated grapes. Analysis of variance was conducted to determine whether significant differences existed between the drying treatments under study.

$$\%\ Variation = \frac{P_{DG} - P_{FG}}{P_{FG}} \times 100 \qquad (2)$$

where:

P_{DG} = amount of compound in 100 g of dehydrated grape (wet basis)

P_{FG} = amount of compound in 100 g of fresh grape

Fig. 1 shows the variations in the content of tartaric and ascorbic acid. Both acids were affected by the drying treatments, causing losses in almost every case. No significant differences in TA content were observed between the dehydration treatments or with the application of pre-treatment. As regards ascorbic acid, only the HA dried samples presented no loss probably because the skin of the grapes did not change much in comparison with the other drying methods and protected the acid from the effects of oxygen. In general, as expected, the ascorbic acid exhibited significant losses due to hydrolysis and the high drying temperatures reached in the product, especially with the microwave technique and with the NaOH pretreatment. The large losses observed in the pretreated grapes may be caused by leaching during grape dipping and greater degradation of ascorbic acid in alkaline media (Fennema, 1993). On the other hand, it is well known that ascorbic acid is seriously affected by high temperatures, which would explain its significant reduction in the case of the microwave method (Vikram et al., 2005).

In general, drying treatments caused a decrease in sugar content (Fig. 2.), sucrose being the most strongly affected (not detected in dehydrated grapes) probably due to its susceptibility to hydrolysis by effect of the high drying temperatures. Glucose losses were lower when the grape was pretreated with NaOH, whereas fructose content decreased. This may be due to the keto-enolic equilibrium of fructose in a basic medium, i.e. the conversion of fructose into its enediol and of the latter into a glucose molecule.

Fig. 3 shows the effect of dehydration on the studied minerals. The mineral content decreased in the dehydrated fruit except for the fruit treated with NaOH and dried by convection. In this case, the content of calcium and potassium increased significantly. In microwave drying, the higher temperatures reached by the product together with a possible interaction of the microwaves with the minerals could be the cause of the substantial reduction in mineral content.

Similarly, the application of microwaves resulted in an increase of the water-soluble pectin fraction compared to convective drying (Fig. 4.). This indicates a greater cell disruption and rupture in the MW dried samples due to the high temperatures reached in the fruit (Contreras et al., 2005). Similarly, the oxalate soluble fraction, consisting of low-methoxylated pectins with the ability to bind calcium, was also affected by the drying treatments. When the fruit was not dipped in a NaOH solution, the bonds between calcium and the low-methoxylated pectins seem to be modified in the dehydrated fruits, as evidenced by a decrease in the oxalate soluble fraction. By contrast, pretreatment caused the opposite effect. The NaOH might have demethoxylated the high-methoxylated pectins (soluble in water), converting them into low-methoxylated pectins (oxalate soluble), which would explain the increase of the soluble oxalate fraction in pretreated grapes (Kim et al., 1978). In view of these results, we can conclude that the HA dried samples without pretreatment are the least affected while the MW dried samples undergo the greatest changes in pectic composition. As a result, the cell structure of the HA samples becomes less altered.

Fig. 5 presents the effects of grape processing on antioxidant activity and total phenols. The phenol content increased in dehydrated grapes, especially when the fruit was pretreated with NaOH. Greater ease in the extraction of these compounds as a result of the alteration of the structure during the drying and breakage of the skin during

pretreatment, could be responsible for the highest values obtained. The antioxidant activity, however, did not experience the same changes as the phenols. This may be due to the low correlation between the total phenolic content, ascorbic acid and antioxidant activity in the samples with no anthocyanins, as in the case of white grapes. In fact, some studies report no correlation between both elements (Kuskoski et al., 2005).

For comparison between the experimentally obtained raisins and the commercial raisins, and bearing in mind that the final moisture content of the product was higher in the treated grapes than in the commercial raisins (0.24 - 0.37 vs. 0.14 g water/g sample), the values of each compound studied was expressed in dry basis. In Table 4 the values of the analyzed compounds in the commercial raisins and in the experimental raisins are presented. In all cases, except for ascorbic acid, the commercial raisins exhibit intermediate values of the different compounds compared to those found in the different treated samples. The lower AA content of the commercial product is probably due to the higher drying intensities required to obtain a product with lower moisture content.

4. Conclusions

The use of microwaves yields a product with similar nutritional/functional properties but with lower drying times, particularly when using the NaOH pretreatment. Note that the drying times greatly differed depending on the treatment used: MW = 7.5 h, HA = 5 days, MW + NaOH = 4.5 h and HA + NaOH = 34 h. In all cases, the products obtained possess quality attributes equivalent to those of commercial raisins.

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301

Figure 1

Figure 1

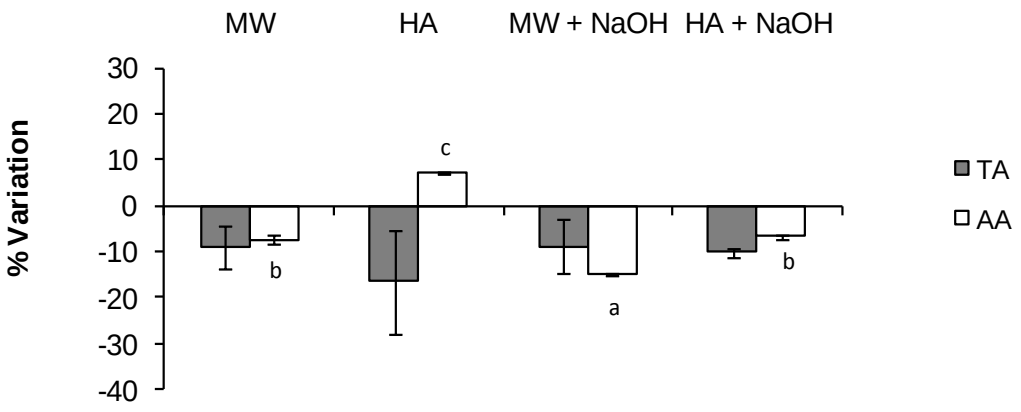

Figure 2

Figure 2

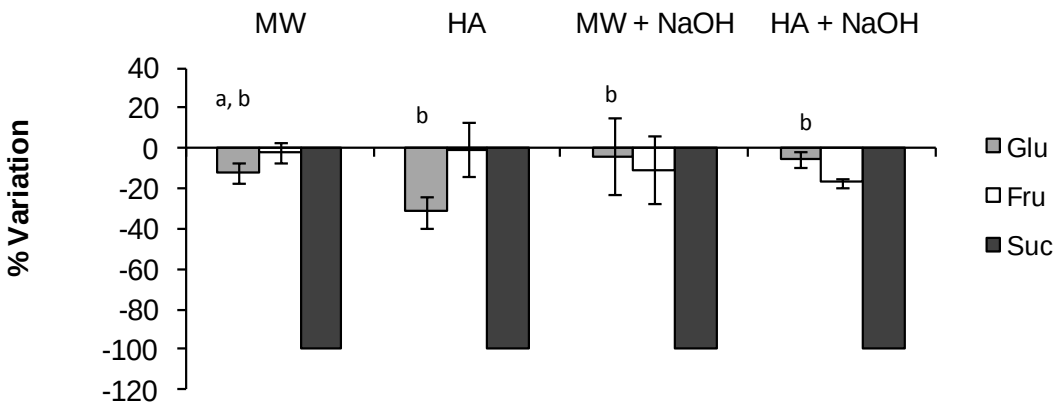

Figure 3

Figura 3

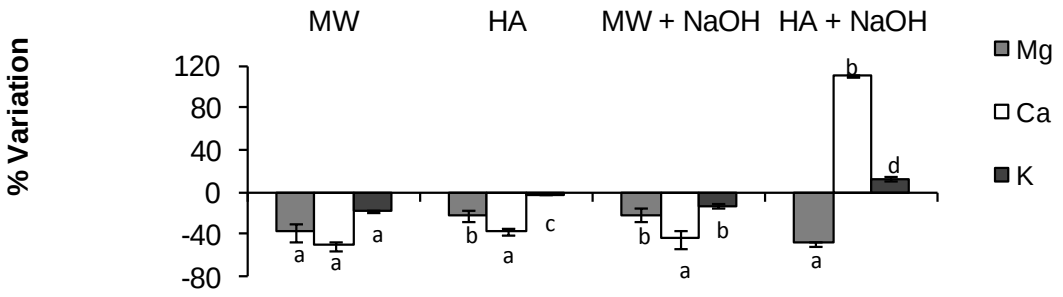

Figure 4

Figure 4.

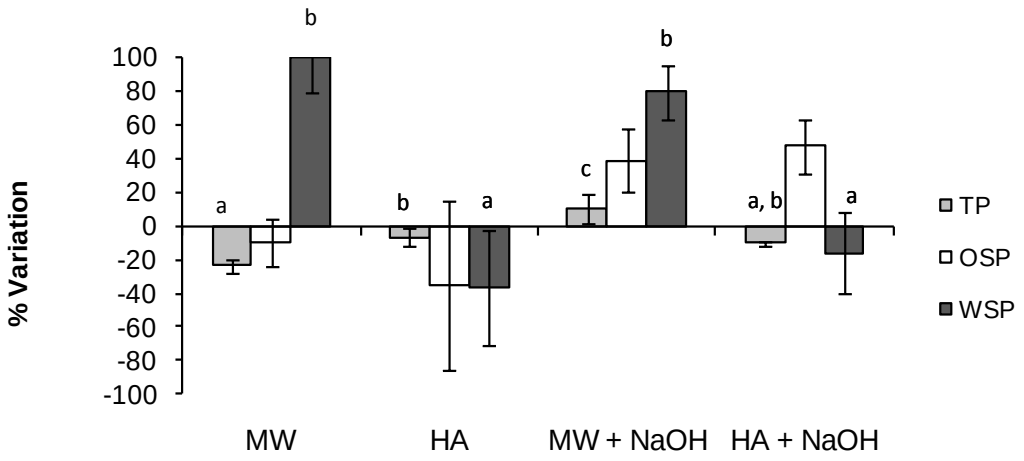

Figure 5

Figure 5

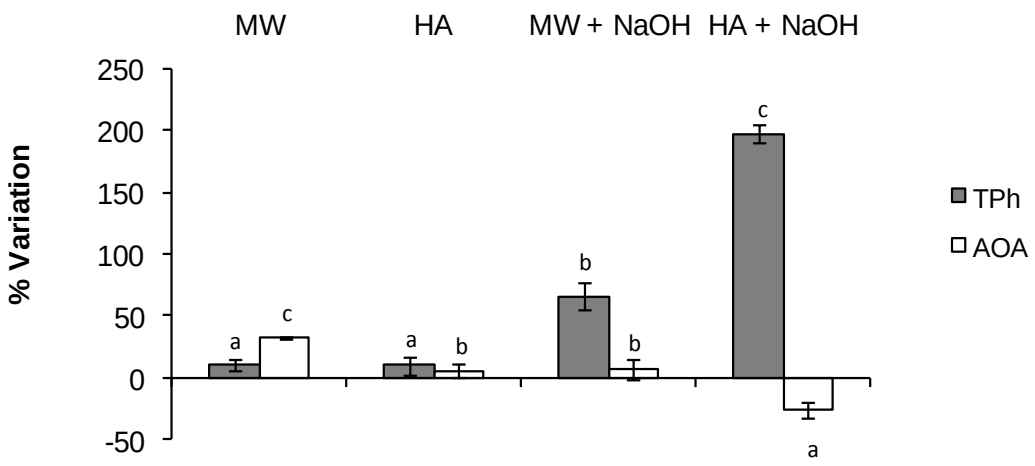

Table 1

Análisis	Thompson seedless			
	FG	MWD	FG	HAD
Xw (g/g)	0,80 (0,02)	0,24 (0,07)	0,798 (0,013)	0,31 (0,05)
a_w	0,977 (0,003)	0,915 (0,003)	0,971 (0,003)	0,854 (0,003)
ºBrix	19 (2)	75 (5)	20,9 (0,05)	69 (3)
Glucose (mg/100g)	8,2 (0,7)	34 (2)	10,4 (0,13)	22 (2)
Fructose (mg/100g)	9,1 (0,15)	41,3 (2,2)	11,4 (0,02)	34 (2)
Sucrose (mg/100g)	0,36 (0,06)	ND	0,93 (0,10)	ND
TA (mg tartaric acid/100g)	563 (10)	1847 (94)	388 (9)	974 (134)
AA (mg/100g)	2,313 (0,011)	8 (0,1)	3,596 (0,010)	11,54 (0,04)
Mg (mg/100g)	6,6 (0,7)	15 (2)	7,91 (0,13)	18,6 (1,5)
Ca (mg/100g)	7,6 (0,2)	13,9 (1,3)	9,8 (0,8)	18,4 (1)
K (mg/100g)	231,3 (1,1)	702 (11)	234 (6)	687 (3)
P (mg/100g)	27,7 (1,2)	50 (5)	25 (3)	130 (9)
TPectin(mg AGU/100g)	355 (3)	923 (48)	275 (20)	770 (43)
WSP (mg AGU/100g)	54 (8)	500 (163)	55 (36)	74 (28)
OSP (mg AGU/100g)	167 (32)	527 (108)	183 (53)	340 (252)
TP (mg GAE/100g)	72 (2)	335 (13)	52 (3)	172 (11)
AOA (mg TEAC/100g)	27,5 (1,3)	105 (1)	34 (2)	103 (5)

Table 2

Análisis	Imperial seedless			
	FG	MWD + NaOH	FG	HAD + NaOH
Xw (g/g)	0,84 (0,02)	0,37 (0,06)	0,846 (0,003)	0,26 (0,02)
a_w	0,973 (0,003)	0,793 (0,008)	0,972 (0,003)	0,820 (0,003)
ºBrix	18,7 (0,05)	60 (5)	18 (0,05)	60,03 (0,08)
Glucose (mg/100g)	6,7 (0,07)	27,7 (1,3)	6,7 (0,07)	26 (1)
Fructose (mg/100g)	8,3 (0,09)	28,7 (5,4)	8,3 (0,09)	28,2 (0,2)
Sucrose (mg/100g)	0,55 (0,05)	ND	0,55 (0,05)	ND
TA (mg tartaric acid/100g)	424 (7)	1506 (97)	494 (8)	1598 (20)
AA (mg/100g)	3,8 (1,1)	13,34 (0,05)	2,92 (0,012)	9,84 (0,03)
Mg (mg/100g)	9,2 (0,3)	28 (2)	9,2 (0,3)	17 (1)
Ca (mg/100g)	10,18 (0,12)	22 (3)	10,18 (0,12)	70 (12)
K (mg/100g)	149 (3)	500 (12)	149 (3)	602 (10)
P (mg/100g)	7,2 (0,6)	14 (4)	7,2 (0,6)	24(2)
TPectin (mg AGU/100g)	443 (73)	1941 (158)	387 (9)	1248 (21)
WSP (mg AGU/100g)	57 (3)	406 (36)	55,0 (1,6)	150 (56)
OSP (mg AGU/100g)	119 (50)	656 (87)	205 (17)	1085 (119)
TP (mg GAE/100g)	47,3 (1,6)	299 (20)	47,3 (1,6)	506 (12)
AOA (mg TEAC/100g)	30,1 (1,1)	129 (10)	28,6 (1,5)	76 (6)

Table 3

Análisis	Thompson seedless		Imperial Seedless	
	MWD	HAD	MWD + NaOH	HAD + NaOH
Xw (g/g)	0,19 (0,05)	0,31 (0,05)	0,39 (0,04)	0,25 (0,01)
a_w	0,915 (0,003)	0,85 (0,003)	0,79 (0,01)	0,812 (0,02)
ºBrix	79,3 (0,03)	54 (0,1)	60,4 (0,2)	72 (0,1)
Glucose (mg/100g)	7,2 (0,5)	7,2 (0,8)	6,4 (1,3)	6,4 (0,3)
Fructose (mg/100g)	8,9 (0,5)	11,4 (1,5)	7,1 (0,3)	6,9 (0,2)
Sucrose (mg/100g)	ND	ND	ND	ND
TA (mg tartaric acid/100g)	512 (26)	324 (43)	387 (25)	444 (6)
AA (mg/100g)	2,14 (0,02)	3,9 (0,01)	3,3 (0,01)	2,73 (0,01)
Mg (mg/100g)	4,1 (0,6)	6,2 (1,3)	7,2 (0,5)	4,8 (0,2)
Ca (mg/100g)	3,8 (0,4)	6,1 (2,2)	5,7 (0,9)	21,44 (0,01)
K (mg/100g)	193 (3)	228 (1)	129 (3)	167 (3)
P (mg/100g)	14,6 (1,6)	44 (3)	3,55 (1)	6,8 (0,6)
TPectin (mg AGU/100g)	272 (14)	256 (14)	433 (97)	347 (6)
WSP (mg AGU/100g)	108 (48)	25 (9)	102 (9)	42 (16)
OSP (mg AGU/100g)	156 (32)	113 (84)	165 (22)	302 (33)
TP (mg GAE/100g)	79 (3)	57 (4)	78 (5)	141 (3)
AOA (mg TEAC/100g)	36,2 (0,3)	34,3 (1,7)	32 (3)	21,1 (1,8)

Table 4

Analysis (dry basis)	Treated samples (experimental range)	Commercial product
Xw (g/g)	0,24(0,07)-0,37(0,06)	0,162 (0,005)
a_w	0,793(0,008)-0,915(0,002)	0,546 (0,009)
ºBrix	60(5)-75(5)	84,5 (0,05)
Glucose (mg/100g)	34(4)-43(3)	39,2 (5,9)
Fructose (mg/100g)	38,2(1,1)-53(3)	42,9 (6,7)
Sucrose (mg/100g)	ND	ND
TA (mg tartaric acid/100g)	1422(190)-2460(126)	1742 (46)
AA (mg/100g)	4,60(0,02)-23,10(0,08)	5,9 (0,6)
Mg (mg/100g)	22(3)-44(7)	35 (6)
Ca (mg/100g)	20,2(1,8)-95(16)	62,2 (10,8)
K (mg/100g)	786(105)-1038(60)	820,5 (90,3)
P (mg/100g)	33(3)-189(13)	67 (4)
TPectin (mg AGU/100g)	821(185)-1687(29)	944 (200)
WSP (mg AGU/100g)	108(41)-586(67)	52,9 (8,6)
OSP (mg AGU/100g)	496(368)-1467(161)	143,9 (28,3)
TP (mg GAE/100g)	251(16)-602(141)	341,4 (15,2)
AOA (mg TEAC/100g)	114(21)-223(18)	145 (9)