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Additional Information

Jam processing and storage effects on β -carotene and flavonoids content in grapefruit.

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Abstract

Grapefruit phytochemicals (β-carotene and flavonoids) stability after different jam processing was evaluated. Osmotic dehydration, microwave energy and conventional heating techiques have been used to obtain jam. β-carotene and individual flavonoids were analyzed by HPLC technique. The results showed that jam obtained from osmodehydrated fruit (ODJ) is the only that preserved completely the β-carotene content. All processes of production of jam significantly decreased the content of narirutin (NAT), poncirin (PON), naringenin (NAG) and quercetin (QUER), while naringin (NAR) remained stable. Jams obtained by applying a heat treatment showed significant lower values of NAG and QUER in comparison with ODJ. The jam obtained from osmodehydrated fruit, without being submitted to any heat treatment, showed at the end of storage the highest contents of naringin, hesperidin, neohesperidin, didymin, quercetin, poncirin and the total sum of analysed flavonoids. In general, the phytochemical loss in jams as a consequence of processing was lower than those provoked by storage effect.

Keywords: grapefruit jam, osmotic dehydration, microwaves, flavonoids, β-carotene,

23 phytochemicals

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1. Introduction

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Numerous epidemiological studies suggest that diets rich in phytochemicals and antioxidants perform a protective role on health and diseases. Frequent consumption of fruits and vegetables is associated with a lowered risk of cancer, heart disease, hypertension and stroke (Marco et al., 1997; Vinson et al., 2001; Wolfe & Liu, 2003). Phytochemicals are some of the bioactive non-nutrient compounds present in fruits, vegetables, grains and other plant foods that have been associated with the protection of human health against chronic degenerative diseases (Kalt, 2001; Martínez-Navarrete et al., 2008; Shahidi & Naczk , 1995). Cells in humans are constantly exposed to a variety of oxidizing agents. These agents may be present in air, food and water, or they may be produced by metabolic activities within cells. The key factor is to maintain a balance between oxidants and antioxidants to sustain optimal physiologic conditions in the body. Overproduction of oxidants can cause an imbalance, leading to oxidative stress, especially in chronic bacterial, viral and parasitic infections (Liu & Hotchkiss, 1995). Moreover, oxidative stress can cause oxidative damage to large biomolecules such as proteins, DNA and lipids, resulting in an increased risk of cancer and cardiovascular disease (Ames, & Gold, 1991; Ames et al., 1993). To prevent or slow down the oxidative stress induced by free radicals, sufficient amounts of antioxidants need to be consumed. Fruits and vegetables contain a wide variety of phytochemicals compounds, such as phenolics and carotenoids, that may help to protect the cellular systems from oxidative damage and to decrease the risk of chronic diseases (Liu, 2003). Citrus fruits are especially valued for their antioxidant capacity, which has been linked to the presence in them of vitamin C, phenols (mainly flavonoids and some phenolic acids) and terpenes (carotenoids). Flavones are phenolic compounds that exist almost exclusively in citrus plants and they have been of particular interest due to their documented broad spectrum of biological activity, including anti-inflammatory, anti-

carcinogenic, and anti-atherogenic properties, among others (Shiming et al., 2009). Citrus fruits are also particularly rich in pectin, implicated in colon cancer prevention and regulation of glucose and cholesterol level (Wang et al., 2007), and in minerals. The amount of each of these compounds found is specific to the citrus fruit and the variety considered. Among them, the orange is the most consumed and therefore has been the most studied. However, the grapefruit is a citrus fruit also of interest, with important health benefits. According to some authors (Peterson et al., 2006a, b; Xu et al., 2008), grapefruit is an excellent source of phytochemicals, more than orange, tangerine, lemon or lime, emphasis in the presence of naringin (which owes its bitter taste) and neohesperidin and, in pink varieties, also β-carotene (pro-vitamin A) and lycopene, responsible for its colour. Despite the high functional value of the grapefruit, it is not widespread consumed, probably because of its strong bitter taste. In this sense processed products that mask this flavour in some extent, such as jams, could be even more acceptable than the fresh fruit. In traditional jam manufacture, all the ingredients are mixed in adequate rates and the mix is concentrated by applying an intense thermal treatment to reach the required final soluble solid content. This process also implies an undesirable impact in colour, flavour and nutritional and functional value of the fruit due to the long time and high temperature reached in the cooking process. An alternative for jam formulation is to use dehydrated fruit obtained by osmotic dehydration (OD) at mild temperature. This technique has been proposed to obtain fruit products without being so aggressive to the antioxidant compounds in the fruit (García-Martínez et al., 2002; Igual et al., 2010; Shi et al., 1996). OD consists of immersing the fruit in a highly concentrated solution in order to promote the water loss of the fruit cells (Lazarides, 2001). The high concentration of solutes reached on the surface of the product contributes to obtain a product with good taste, flavour and colour, to improve the cellular structure and to prevent the pigments and aromatic compounds loss also as the browning of the products (Moraga et al., 2000; Moreno et al.,

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2000; Shi et al., 1996). Another proposed alternative for jam cooking has been the employ of a faster heating method as it is the application of microwave energy (Igual et al., 2010). Microwave absorption provokes internal water heating and evaporation, greatly increasing the internal pressure and concentration gradients and thus the effective water diffusion. As a consequence, shorter processing time is required and higher product quality may be achieved. The different processing technique may affect in a different way to their bioactive compounds. For this reason, the most suitable method to process each product should be selected depending on the type of compounds considered to be the most important (Siriamornpuna et al., 2012). In this sense, the aim of this work was to evaluate the influence of processing (osmotic dehydration, microwave energy and conventional heating) and storage on flavonoids and β -carotene content of grapefruit jam.

2. Materials and methods

2.1. Raw materials

Grapefruits (*Citrus paradise* var. Star Ruby) from the city of Murcia (Spain) were purchased from a local supermarket. Fruit pieces were peeled and cut perpendicularly to the fruit axis into 10 mm thick half-slices. Food grade commercial sucrose was used to prepare conventional and microwave (MW) jams. In the case of the jam obtained by osmotic dehydration, an osmotic solution (OS) was prepared by mixing sucrose with distilled water until it was completely dissolved, forming a 65 °Brix syrup. In this case, citrus peel pectin (60% degree of esterification, Fluka Biochemika, Switzerland) was used as a gelling agent.

2.2. Jams preparation procedures

The following procedures were applied to obtain a 40-60 °Brix product, as described by the Spanish quality norm for fruit jam (BOE, 1990). In all the cases, the obtained jams were placed in sterile glass jars and stored at room temperature for 24 h till analysis. Water activity (a_w) and pH of the jams were analysed by means of a dew point hygrometer FA-st Lab, GBX (Bourg de Peage, France) and a CRISON pH-meter (Barcelona, Spain), respectively. Each analysis was carried out in triplicate.

2.2.1. Conventional process

Fresh fruit (FG) (67 g grapefruit/100 g mixture) was pre-cooked at 85 °C for 10 min, added to the sugar and potassium sorbate (32.99 and 0.01 g/100 g mixture, respectively) and cooked at 95-100 °C for 20 min longer. An electrical food processor (Thermomix TM 21, Vorwerk, Spain) was used for the process. The conventional jam obtained with this procedure was named CJ.

2.2.2. Microwave process

FG (67 g grapefruit/100 g mixture) was pre-cooked (900 W, 5 min), added to the sugar and potassium sorbate (32.99 and 0.01 g/100 g mixture, respectively) and cooked at 900 W for 10 min longer. A household microwave-air oven (Moulinex 5141 AFW2, Barcelona, Spain) was used to obtain this jam, named MWJ.

122 2.2.3. Osmotic process

Half slices of peeled grapefruit were placed in a 65 °Brix OS (ratio OS:fruit 5:1) for 10 min at room temperature and 50 mbar pressure and then maintained for 10 min longer at atmospheric pressure. After that, the fruit pieces and the OS were heated to 40 °C (water bath P-Selecta Precisterm, Barcelona, Spain) with continuous stirring of the OS(200 rpm, Heidolph Instruments, RZR 2020, Schwabach, Germany) for 3 h, to reach grapefruit with

about 30 °Brix according (Igual et al., 2010). Osmo-dehydrated grapefruit pieces (ODG), potassium sorbate (0.01 g/100 g jam) and pectin (1 g/100 g jam) were ground with the required part of the OS to obtain jam with 60 g fresh fruit/100 g jam, taking into account °Brix of ODG and °Brix of the OS. The jam thus obtained was referred as ODJ.

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- 2.2.4. Combined osmotic-microwave process
- 134 Jams obtained from osmo-dehydrated grapefruit, as described in Section 2.2.3, were
- cooked in the microwave-air oven at 900 W for 5 min to obtain OD+MWJ samples.

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- 137 2.3 Storage conditions
- 138 Jams were stored for 3 months at room temperature, except ODJ which was stored at 4 °C
- (García-Martínez et al., 2002; Igual et al., 2011a). Analyses were carried out after 1, 7, 15,
- 140 30, 45, 60, 75 and 90 days of storage.

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- 142 *2.4. Analysis*
- 143 2.4.1. β-carotene
- 144 Samples were homogenized. Ethanol (4 mL) was added to 2 g homogenate paste and the 145 mixture was centrifuged (Selecta Medifriger-BL, Barcelona, Spain) at 2000 rpm for 3 min 146 at 4 °C. The supernatant was filtered through a Whatman No.1 paper and 0.5 mL of n-147 hexane were added to the filtrate and mixed. β-carotene was extracted twice in the hexane 148 phase and the collected extract was dried under a stream of liquid nitrogen. Dried extract 149 was solubilized in 0.2 mL methanol. β-carotene content was determined and quantified by 150 HPLC. The HPLC (Jasco, Cremella, Italy) equipment consisted of a ternary pump (Jasco PU- 1580 HPLC pump), a gradient generator (LG-1580-02 Ternary Gradient Unit), 151 152 Ultrabase-C18 column (5 µm, 4.6 x 250 mm) and a UV-visible detector (MD-1510) with a

range of measurement wavelength of 190 to 650 nm. The mobile phase was composed

methanol: acetonitrile: chloroform (47:42:11, v/v/v), volume injection 20 μL and flow rate 1 mL/min. The β -carotene detection was at 436 nm and 25 °C (Munzuroglu et al., 2003). Standard curve of this reference compound (Fluka-Biochemika, Milwaukee, WI, USA) was used to quantify. The results were expressed as mg of β -carotene per 100 grams of fresh sample, considering the percentage of fresh grapefruit in the sample. Changes in this compound along storage were expressed as the compound variation (Δ M_i) referred to the fresh grapefruit content, according to equation (1):

$$\Delta M_{i} = \frac{\left(M_{i}^{t} - M_{i}^{0}\right)}{M_{i}^{FG}} \tag{1}$$

where: M_it: mass of compound i in the sample / g fresh grapefruit at storage time t, M_io:

mass of compound i in the sample / g fresh grapefruit at storage time 0 and M_iFG: mass of

compound i / g fresh grapefruit.

2.4.2. Flavonoids

The extraction of flavonoids was carried out following the procedure proposed by Tomás-Barberán et al. (2001). It consisted of homogenizing 35 g of the sample (T25 Janke and Kunkel turrax) for 5 min with 40 mL of methanol, 10 mL of double distilled water and NaF to inactivate polyphenol oxidases and to prevent phenolic degradation. The homogenate was centrifuged (Selecta Medifriger-BL, 10,000 rpm, 10 min, 4 °C) to obtain the supernatant that was filtered through a 0.45 μ m membrane filter. HPLC method and instrumentation was: Ultrabase-C18, 5 μ m (4.6x250 mm) column (Análisis Vínicos, Tomelloso, Spain); mobile phase was composed of methanol and water and a linear gradient elution was performed starting at 30:70 to reach 100:0 at 70 min, volume injection 25 μ L and flow rate 1 mL/min. Chromatograms were recorded at 286, 284 and 254 nm and at 25 °C. The standard curves of the reference flavonoids, narirutin (NAT), naringin (NAR), hesperidin (HES), neohesperidin (NEOH), didymin (DID), poncirin (PON), naringenin

(NAG) and quercetin (QUER) (Extrasynthese, France) were used to quantify the flavonoids. Naphthalene was used as internal standard (Peiró, 2007; Igual et al., 2011b). The results were expressed as mg of each flavonoid per 100 grams of fresh sample, considering the percentage of fresh grapefruit in the sample. Changes in each compound along storage were expressed as the compound variation (ΔM_i) referred to the fresh grapefruit content, according to equation (1).

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2.5. Statistical analysis

Significant differences among treatments and storage time were evaluated by means of the corresponding analysis of variance (ANOVA) performed by using Statgraphics Plus 5.1. Values of p<0.05 were considered to represent a significant effect. A Principal Component Analysis (PCA) with varimax rotation was applied to the values of the flavonoid content, using SPSS program version 16.0.

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3. Results and Discussion

194 Significant differences were found among water activity of all the jams, the values being 0.945, 0.942, 0.924 and 0.922 (standard deviation 0.003 in all the cases) for ODJ, 195 196 OD+MWJ, MWJ and CJ, respectively. As regards pH (standard deviation 0.02 in all the 197 cases), no significant differences were found between ODJ, OD+MWJ (3.39 and 3.40, 198 respectively) while it was significantly different from that of MWJ (3.27) and CJ (3.25). 199 Some authors have indicated that freezing, pasteurization, boiling and microwave cooking 200 generally reduce the antioxidant capacity of fruits (Aziz et al., 1998; Gil-Izquierdo et al., 201 2002; Guyot et al., 2003). Phenolics and carotenoids have been described as antioxidant 202 compounds. Processing of fruits normally leads to a decrease in the concentration and a 203 change in the composition of phytochemicals including flavonoids (Tsao et al., 2006). 204 Carotenoids are lost between 5 and 40%, depending on the conditions of food preparation

and preservation (Belitz & Grosch, 1997, Eitenmiller & Laden, 1999). The impact of the different processes carried out in the present work to obtain jam on these compounds is shown in Table 1, where the mean values of β-carotene and flavonoids content of FG, ODG and jams, all of them referred to 100 g of fresh grapefruit, appear. Table 2 shows the loss of each analyzed compound, compared to the content present in the fresh fruit, due to processing and storage. The β-carotene is the major dietary precursor of vitamin A (Xu et al., 2006), becoming retinol inside the human body (Belitz & Grosch, 1997). Besides its function as pro-vitamin A, the functional significance of this carotenoid is also due to its antioxidant action (Bushway, 1986). As is shown in Table 1, in this study FG showed values in the same order to those obtained in previous studies for red grapefruit of the same variety (0.2-1.3 mg/100 g; Ladaniya, 2008; Rojas, 2004; Rouseff et al., 1992). After osmotic-dehydration, the sample retained the β-carotene content. When comparing the jams, ODJ was the only one that completely preserved the β-carotene content showing only 4.19% loss of this compound. Nevertheless the sample subjected to combined treatment (OD+MWJ) presented the greatest loss (29 g β-carotene loss/100 g β-carotene present in the fresh fruit, Table 2). The jams obtained by applying an intense thermal treatment (CJ and MWJ) showed similar values of this compound, with about 17.5 g β-carotene loss/100 g βcarotene present in the fresh fruit (Tables 1 and 2). In general, β-carotene is sensitive to oxygen and light, oxidation losses occurring especially at high temperatures (Lesková et al., 2006). On the other hand, in the absence of these two factors, β-carotene is quite stable at elevated temperatures (cooking), producing in this case isomerization and fragmentation. Figure 1 shows the β-carotene change in the jams during storage period, referred to the content in the fresh sample. β-carotene losses were faster during the first week in the case of ODJ and during the first 15 days in the rest of the jams. From that moment onwards, the

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β-carotene content remained constant until the end of storage in all the jams. After 3 months, the samples that were subjected to more intense heat treatments during jam preparation presented a loss between 33 and 38% (Table 2); lower values than these were observed for jams made from osmodehydrated fruit (55-56%). Although the OD treatment maintained the β-carotene content of the fresh grapefruit, greater losses during storage were observed in OD and OD+MWJ samples. This could be related to the greater aw and pH of the jams obtained from OD fruit. As it can be observed in Table 1, the most abundant flavonoid in the fresh grapefruit was NAR followed by NAT, QUER and NAG, results that closely agree with other studies (Gorinstein et al., 2006; Igual et al., 2011b; Peterson et al., 2006a, Ross et al., 2000; Vanamala et al., 2006). In general, osmotic dehydration of the fruit caused no changes in the concentration of the studied flavonoids. Only a significant HES decrease was detected. All the processes carried out to obtain jams significantly (p<0.05) decreased the content of NAT, PON, NAG and QUER. NAR remained stable during all treatments without showing significant (p>0.05) differences with the fresh grapefruit. Jams obtained by heating (CJ, MWJ and OD+MWJ) showed significant (p<0.05) lower values of NAG, DID and QUER as compared to ODJ while NAT, HES and NEOH were worse preserved in ODJ. The loss of each compound due to the jam elaboration process appears in Table 2. As regards the total flavonoids in the samples, calculated as the sum of the individual analysed flavonoids, fresh and OD grapefruit contained about 140 mg/100g fresh fruit and all the jams presented a significant (p<0.05) lower content, ODJ followed by MWJ being the ones with more flavonoids (about 124 mg/100g fresh fruit). A total flavonoids loss caused by processing of 9-18 g/100g total flavonoids present in the fresh fruit was quantified (Table 2). The change in content of flavonoids in the obtained jams during storage appears in Figures 2 and 3. In general, losses of all the studied flavonoids, except in the case of PON, could be observed. In all the jams, PON remained stable during the first month of storage

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and thereafter, it increased. This increase can be attributed to a chemical transformation of NAG and NAR (Igual et al., 2011b). During the first 45 days of storage, NAT and NAG remained stable and from that moment onwards, its content decreased until the end of storage. The greatest loss of HES, NEOH and DID occurred in all the samples during the first 15 days. From that moment, the content of these flavonoids remained stable until the end of storage. Intensive thermal treatments (CJ and MWJ) lead to greater losses in NAR, HES and NEOH during storage, while jams made from osmodehydrated fruit lost more QUER and NAG in this period (Table 2). Figure 4 shows the variation in the sum of all the flavonoids considered referred to the content in the fresh fruit, during 3 months of storage. Jams presented losses during the storage period in the range of 21.5-29.3 g total flavonoids/100g total flavonoids present in the fresh fruit (Table 2). These losses were more marked from day 45 (Figures 2 and 3). The more intensively treated samples (CJ and MWJ) showed the greatest loss during the studied period. The evolution of flavonoids content can be easily observed by means of the PCA carried out with the values corresponding to all the jams at different storage times (Figure 5). The first two factors showed eigenvalues higher than 1. The consideration of both factors accounted for 83.79 % of the total variability. The first factor (F1), explaining 51.14% of the variability, was associated with DID (r=0.94), NEOH (r=0.93), NAR (r=0.93), HES (r=0.90), QUER (r=0.75) and NAG (r=0.74) values. The second factor (F2) accounted for 26.65% of the variability and it was mainly associated with PON (r=0.94) and NAT (r=0.91) values. All the grapefruit jams newly processed showed a higher content of the flavonoids associated with F1 and of NAT but low of PON. During the first month of storage, PON and NAT remained stable while the rest of the flavonoids decreased. From this moment onwards, NAT decreased and PON increased while the other flavonoids did not showed additional changes. Applying a multifactor ANOVA to the values of F1 and F2, it can be observed that both factors are affected by the interaction of the progress of time and the treatment

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applied to obtain jams. F1 decreased more sharply during the first month in CJ and MWJ as compared to ODJ and OD+MWJ while F2 decreases faster during the last two months in the samples ODJ and OD+MWJ when compared to CJ and MWJ.

As can be observed in Table 2, in general the losses of the analyzed compounds in jams caused by processing were lower than those provoked by storage period. As regards the total losses occurred due to both processing and storage, the β -carotene loss when compared to its content in the fresh fruit was between 53 and 86 %, being the combined treatment (OD+MWJ) especially less recommendable to preserve this compound. In the case of flavonoids, these loosses were between 33 and 47 %, the greatest ones being showed by the more intense thermally treated jams, especially the one obtained by using the conventional procedure.

4. Conclusion

Flavonoids of grapefruit are better retained than β -carotene in jams. The greatest losses of the analyzed compounds occurred during jam's storage and not during the production processing. Taking into account the obtained results, microwave heating may be proposed as a good process, better than osmotic dehydration or conventional heating, to obtain a stable jam. This procedure can best fulfill the commitment process time-functional quality of the stored obtained product. Osmotic dehydration would only be recommended if a ready to eat jam is wanted to be obtained.

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473 **FIGURE CAPTIONS**

- 474 **Figure 1.** β-carotene variations of studied jams along 3 month of storage.
- Figure 2. Narirutin (NAT), naringin (NAR), naringenin (NAG) and quercetin (QUER)
- variations of studied jams along 3 month of storage.
- Figure 3. Hesperidin (HES), neohesperidin (NEOH), didymin (DID) and poncirin (PON)
- variations of studied jams along 3 month of storage.
- 479 Figure 4. Principal Component Analysis (PCA) with varimax rotation of the values of
- flavonoid content corresponding to all the grapefruit jam samples. D0, D30, D60 and D90
- indicate storage days.
- Figure 5. Total flavonoids variations of studied jams along 3 month of storage.

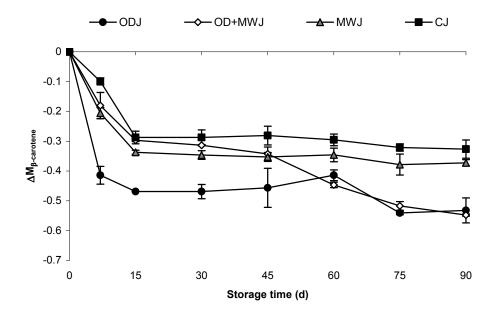


Figure 1

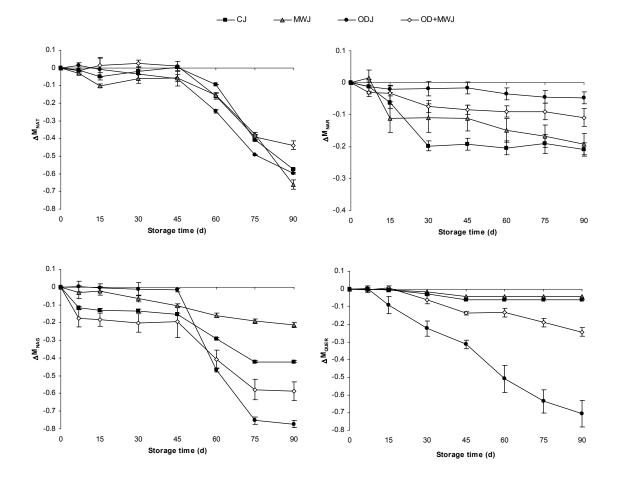


Figure 2

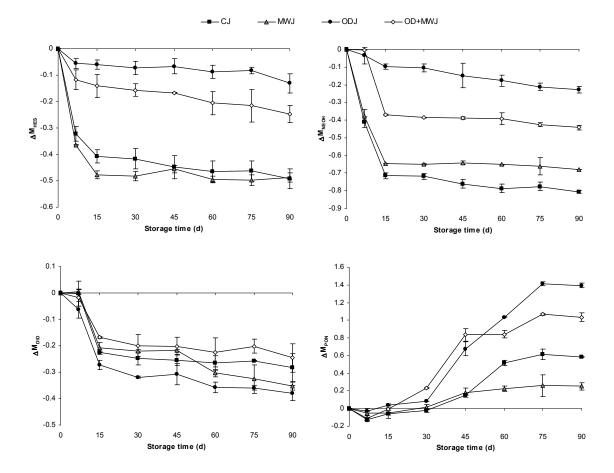


Figure 3

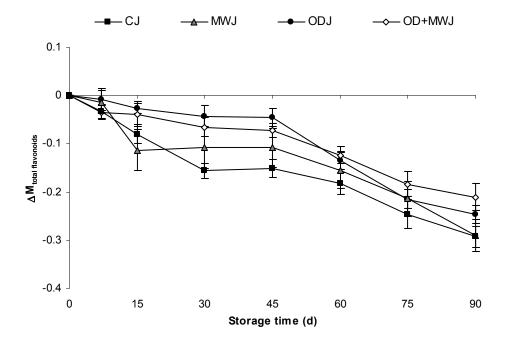


Figure 4

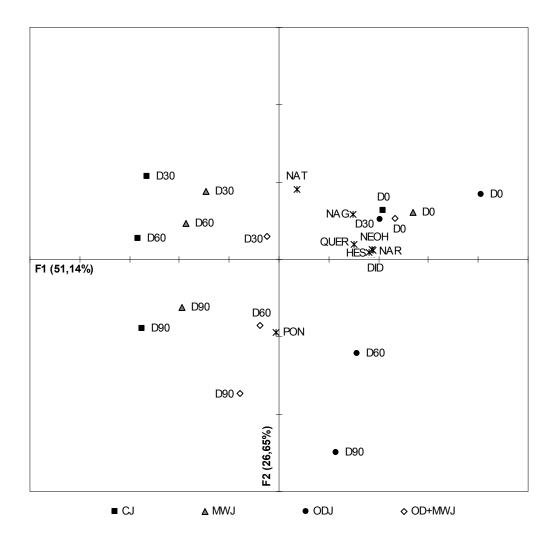


Figure 5

Table 1. Mean values (with standard deviation) of β -carotene and flavonoids content (mg / 100 g fresh fruit) in fresh grapefruit (FG), osmodehydrated grapefruit (ODG) and jams obtained by convencional processing (CJ), microwave (MWJ), from osmodehydrated grapefruit (ODJ) and by combined treatment (OD+MWJ).

Compound	FG	ODG	CJ	MWJ	ODJ	OD+MWJ
β-carotene	2.58 (0.11) ^a	2.60 (0.14) ^a	2.05 (0.02)b	2.22 (0.06)b	2.48 (0.05) ^a	1.83 (0.02) ^c
NAT	29.4 (0.4) ^a	28.7 (0.2) ^a	24.2 (0.3)b	25.3 (0.6)b	22.2 (0.5) ^c	20.0 (0.8) ^d
NAR	84 (3) ^a	82 (2) ^a	81 (5) ^a	85 (3) ^a	81 (2) ^a	84 (6) ^a
HES	2.40 (0.06) ^a	1.98 (0.09) ^b	2.09 (0.05)b	2.24 (0.04) ^a	1.76 (0.05) ^c	1.72 (0.07) ^c
NEOH	2.92 (0.07) ^{ab}	2.94(0.2) ^a	3.13 (0.05) ^a	3.09 (0.09) ^a	2.6 (0.2) ^c	2.64 (0.07)bc
DID	1.42 (0.03) ^a	1.5 (0.2) ^a	0.95 (0.03) ^c	1.10 (0.02)bc	1.46 (0.02) ^a	1.16 (0.05) ^b
PON	1.921 (0.010) ^a	2.06 (0.12) ^a	0.47 (0.02) ^b	0.47 (0.09)b	0.52 (0.02)b	0.60 (0.03) ^b
NAG	8.3 (0.2) ^a	8.5 (0.4) ^a	3.49 (0.04) ^d	3.55 (0.04) ^d	6.8 (0.4) ^b	4.9 (0.5) ^c
QUER	11.4 (0.2) ^a	11.34 (0.03) ^a	0.70 (0.02) ^d	0.50 (0.04) ^d	8.6 (0.8) ^b	3.1 (0.2) ^c
Total Flavonoids	141 (3) ^a	139.4 (0.8) ^{ab}	116 (5) ^d	119 (7) ^{cd}	129 (4) ^{bc}	115 (3) ^d

The same letter in superscript within rows indicates homogeneous groups established by the ANOVA (p<0.05).

NAT: narirutin, NAR: naringin, HES: hesperidin, NEOH: neohesperidin, DID: didymin, PON: poncirin, NAG: naringenin y QUER: quercetin

Table 2. Loss of each analyzed compound, compared to the content of the correponding compounds present in the fresh fruit, due to the process and also to the storage in jams obtained by convencional processing (CJ), microwave (MWJ), from osmodehydrated grapefruit (ODJ) and by combined treatment (OD+MWJ).

Compound	g component lost during processing / 100g component present in fresh grapefruit				g component lost during storage / 100g component present in fresh grapefruit				g component lost during processing and storage / 100g component present in fresh grapefruit			
	CJ	MWJ	ODJ	OD+MWJ	CJ	MWJ	ODJ	OD+MWJ	CJ	MWJ	ODJ	OD+MWJ
β-carotene	20.83	14.13	4.19	29.19	33.63	38.36	54.82	56.42	54.46	52.49	59.01	85.61
NAT	17.75	14.20	24.42	31.92	57.47	66.08	59.79	44.27	75.21	80.28	84.21	76.19
NAR	3.11	0.009	-1.87	3.03	20.98	19.31	4.80	11.20	24.09	19.32	2.93	14.23
HES	13.04	6.50	26.61	28.16	49.25	48.63	12.97	25.25	62.29	55.13	39.57	53.42
NEOH	-7.45	-6.01	12.01	9.60	80.57	67.89	22.90	44.98	73.12	61.89	34.91	54.58
DID	33.32	22.75	-2.90	18.19	28.18	35.30	37.89	24.98	61.50	58.05	34.98	43.17
PON	75.38	75.38	73.13	68.98	-58.39	-25.47	-139.33	-103.15	16.99	49.91	-66.19	-34.16
NAG	57.78	57.04	18.20	39.72	42.22	21.37	77.26	59.67	100.00	78.41	95.45	99.39
QUER	93.85	95.58	24.53	73.36	6.15	4.42	70.67	24.13	100.00	100.00	95.20	97.48
Total Flavonoids	17.87	15.21	8.67	18.45	29.33	29.02	24.66	21.47	47.20	44.23	33.33	39.92

NAT: narirutin, NAR: naringin, HES: hesperidin, NEOH: neohesperidin, DID: didymin, PON: poncirin, NAG: naringenin y QUER: quercetin