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Stability of micronutrients and phytochemicals of grapefruit jam as affected by the obtention process

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Abstract

Fruits are widely revered for their micronutrient properties. They serve as a primary source of vitamins and minerals as well as of natural phytonutrients with antioxidant properties. Jam constitutes an interesting way to preserve fruit. Traditionally, this product is obtained by intense heat treatment that may cause irreversible loss of these bioactive compounds responsible for the health-related properties of fruits. In this work, different grapefruit jams obtained by conventional, osmotic dehydration (OD) without thermal treatment and/or microwave (MW) techniques were compared in terms of their vitamin, organic acid and phytochemical content and their stability through 3 months of storage. If compared with heating, osmotic treatments lead to a greater loss of organic acids and vitamin C during both processing and storage. Microwave treatments permit jam to be obtained which has a similar nutritional and functional value than that obtained when using a conventional heating method, but in a much shorter time.

Keywords

Grapefruit, osmotic dehydration, microwave, vitamins, organic acids, carotenoids, phenols, storage

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Introduction

Fruits have historically been considered rich sources of some essential dietary micronutrients and fibers. More recently, they have been recognized as important sources for a wide array of phytochemicals that individually, or in combination, may benefit health (Rechkemmer, 2001). These naturally occurring compounds possess anticarcinogenic and other beneficial properties, so that they are referred to as chemopreventives. Therefore, some people have conferred on fruits and vegetables the status of "functional foods" (Yahia, 2010). One of the predominant mechanisms of the protective action of phytochemicals is their antioxidant activity and the capacity to scavenge free radicals. Among the most widely investigated chemopreventives are to be found some vitamins, polyphenols and pigments, such as carotenoids, chlorophylls, flavonoids and betalains (Yahia, 2010). Grapefruit is a citrus fruit which presents high amounts of vitamins, phenolic compounds and carotenoids (Rouseff et al., 1992; Xu et al., 2008; Igual et al., 2010a). However, its bitter taste limits its consumer popularity as fresh fruit. An alternative way to consume and preserve fruits has traditionally been as jam. The usual application of prolonged heating treatments of the fruit in the conventional jam elaboration process can lead to an important loss of its beneficial properties. Osmotic dehydration at a mild temperature (30-40 ºC) is a technique that can be used to obtain jam without being so aggressive with the antioxidant compounds of fruits (García-Martínez et al., 2002; Igual et al., 2010b). Osmotic dehydration is a concentration technique, in which the fruit is immersed in a highly concentrated solution in order to promote water loss from the fruit cells. Osmo-dehydrated fruit ground together with a part of the osmotic solution used and some fibre allow to obtain a jam. On the other hand, the use of microwave energy has been proposed as an alternative to traditional heat, since a shorter process time is required due to the volumetric heating of the product. In this sense, the thermolabile nutrients of fresh fruit may be better preserved and a higher quality final product may be

achieved (Picouet et al., 2009, Igual et al., 2010b, García-Martínez et al., 2012). Drying, cooking, blanching, pasteurization, thawing and tempering, microwave vacuum-drying and microwave freeze-drying are some of the commercially proven applications of this technology (Hebbar & Rastogi, 2012; Salazar-González et al., 2012; Vadivambal & Jayas, 2010). In the case of jam making the fresh fruit is previously precooked in the microwave, then mixed with sugar and finally the mixture is cooked again in the microwave. A combined osmotic–microwave process has also been proven suitable to provide a jam with adequate physicochemical and sensorial properties (Igual et al., 2013).

The aim of this work was to evaluate the influence of different jam-making processes, such as osmotic dehydration, microwave energy application and conventional heating, on the organic acid, vitamin and phytochemical content and on the antioxidant capacity of grapefruit jam during storage.

Materials and methods

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 jams. In the case of the jam obtained by osmotic dehydration, an osmotic solution (OS) was prepared by mixing an amount of sucrose with distilled water until it was completely dissolved, forming a 65 ºBrix syrup. In this case, citrus peel pectin (60% degree of esterification, Fluka Biochemika, Switzerland) was added as a gelling agent.

Jams preparation procedures

The following processes were applied to obtain a 40-60 ºBrix 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 were left to gel at 20 ºC for 24 h till analysis.

Conventional process. Fresh fruit (67 g grapefruit/100 g mixture) was pre-cooked at 85 ºC 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 ºC for 20 min longer. An electric food processor (Thermomix TM 21, Vorwerk, Spain) was used for the treatment. The obtained jam was named Conventional.

Microwave process. Fresh fruit (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 (Moulinex 5141 AFW2, Spain) was used to obtain this jam, named MW.

Osmotic process. Half slices of peeled grapefruit were placed in a 65 ºBrix OS (ratio OS:fruit 5:1) for 10 min at 20 ºC and 50 mbar pressure. Afterwards, the atmospheric pressure was restored for 10 min longer in order to promote the impregnation of the fruit with the OS. Finally, samples immersed in the OS were heated to 40 °C (water bath P-Selecta Precisterm, Barcelona, Spain) under continuous stirring (200 rpm, Heidolph Instruments, RZR 2020, Schwabach, Germany) for 3 h, to reach ≈30 ºBrix following a previous kinetic study (Igual et al., 2010b). Osmo-dehydrated grapefruit pieces (ODG) (53 g), potassium sorbate (0.01 g/100 g mixture) and pectin (1 g/100 g mixture) were ground with the required part of the OS (40 g) used for fruit dehydration to obtain jam with 60 g fresh fruit/100 g jam, taking into account ºBrix of ODG and ºBrix of the OS. The jam thus obtained was referred to as OD.

Combined osmotic-microwave process. 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+MW samples.

Storage conditions

Jams were stored for 3 months at 20 ºC, except the OD one which was stored at 4 °C. According to previous studies, OD jams obtained in the absence of thermal treatment need refrigerated storage at 4 ºC to ensure the same shelf life as thermally treated ones (García-Martínez et al., 2002). Analyses were carried out after 1, 7, 15, 30, 45, 60, 75 and 90 days of storage from the day where the jams were obtained.

Analysis

Physicochemical properties. Moisture content (x_w), ºBrix and water activity (a_w) were determined for fresh grapefruit, ODG and all the jams. The x_w was determined by drying the sample to constant weight at 60 ºC in a vacuum oven (AOAC method 934.06, 2000). ºBrix were measured in previously homogenized samples using a refractometer at 20 ºC (Zeiss, ATAGO model NAR-3T refractometer, Japan). A dew point hygrometer (FA-st Lab, GBX, France) was used to measure a_w. pH was measured by means of a CRISON pH-meter (Belgium). Each analysis was carried out in triplicate.

Organic acids. The determination and quantification of tartaric (TA), malic (MA) and citric acid (CA) was performed by high performance liquid chromatography (HPLC) according to Cen et al. (2007). Samples were centrifuged (Selecta Medifriger-BL, Spain) at 9167xg for 15 min and filtered by 0.22 μm membrane. The HPLC equipment (Jasco, Italy) consisted of a ternary pump (Jasco PU-1580 HPLC pump, Italy), a gradient generator (LG-1580-02

Ternary Gradient Unit), Ultrabase-C18 column (5 µm, 4.6x250 mm) and a UV-visible detector (MD-1510) with a range of measurement wavelength from 190 to 650 nm. The mobile phase was 0.01mol/L potassium dihydrogen phosphate solution, volume injection 20 µL flow rate 1mL /min and detection at 215 nm at 25 ºC. Standard curves of each reference acid (tartaric, malic and citric) (Panreac, Spain) were used to quantify. The storage induced variation in each compound (ΔM_i) was expressed as the change in the amount of the compound referred to the fresh grapefruit content, according to equation (1):

$$\Delta M_i = \frac{(M_i^t - M_i^0)}{M_i^{FG}} \quad (1)$$

where: M_i^t: mass of compound *i* in the sample obtained from 1 g fresh grapefruit at storage time t, M_i^0: mass of compound *i* in the sample obtained from 1 g fresh grapefruit at storage time 0 and M_i^{FG}: mass of the compound *i* in 1 g fresh grapefruit.

Ascorbic acid and total vitamin C. Ascorbic acid (AA) and total vitamin C (ascorbic acid + dehydroascorbic acid) were determined by HPLC (Jasco, Italy). To determine the ascorbic acid, the sample (1 g) was extracted with oxalic acid (Xu et al., 2008). The procedure employed to determine total vitamin C (0.5 mL sample) was the reduction of dehydroascorbic acid to ascorbic acid, using DL-dithiothreitol as the reductant reagent (Sánchez-Moreno et al., 2003). Afterwards, the same procedure as that used for the ascorbic acid method was performed. The HPLC conditions were: Ultrabase-C18, 5 µm (4.6x250 mm) column (Análisis Vínicos, Spain); mobile phase 0.1 % oxalic acid, volume injection 20 µL, flow rate 1mL /min, detection at 243 nm and at 25 ºC. AA standard solution (Panreac, Spain) was prepared. The variation in each compound brought about by storage (ΔM_i) was expressed as the change in the amount of the compound referred to the fresh grapefruit content, according to equation (1).

Vitamins A and E. Ethanol (4 mL) was added to 2 g homogeneous sample and the mixture was centrifuged (Selecta Medifriger-BL, Spain) at 366xg for 3 min at 4 ºC. The supernatant was filtered through a Whatman No.1 paper and 0.5 mL of n-hexane were added and mixed. Vitamins A and E were extracted twice in the hexane phase and the collected extract was dried under a stream of liquid nitrogen. The dried extract was solubilized in 0.2 mL methanol. HPLC conditions were: Ultrabase-C18, 5 μm (4.6x250 mm) column (Análisis Vínicos, Spain); methanol/ acetonitrile/ chloroform (47:42:11, v/v) as mobile phase, volume injection 20 μL, flow rate 1 mL/min, detection at 326 and 296 for vitamins A and E, respectively (Munzuroglu et al., 2003) at 25 ºC. Standard curves of reference vitamins A and E (Fluka-Biochemika, USA) were used for quantification purposes. The variation in each compound brought about by storage (ΔM_i) was expressed as the change in the amount of the compound referred to the fresh grapefruit content, according to equation (1).

Total carotenoids. The total quantity of carotenoids (TC) present in the samples (5 g) was extracted with hexane/acetone/ethanol following Olives et al.'s (2006) methodology. The spectrophotometric reference method of AOAC (2000) was used for quantification. Sample absorbance was measured at 446 nm in a UV-visible spectrophotometer (Thermo Electron Corporation, USA). The total carotenoid content was expressed as mg of β-carotene (Fluka-Biochemika, USA) per 100 grams of fresh sample. The variation in each compound brought about by storage (ΔM_i) was expressed as the change in the amount of the compound referred to the fresh grapefruit content, according to equation (1).

Total phenols (TP)***.*** Phenols (35 g sample) were extracted with methanol, HCl (6 N) and NaF and analysed following the Folin-Ciocalteu method, as reported by Selvendran and

Ryden (1990) absorbance was measured at 765 nm in a UV-visible spectrophotometer (Thermo Electron Corporation, USA). The total phenolic content was expressed as mg of Gallic Acid Equivalents (GAE) (Sigma-Aldrich, Germany) per gram of sample, using a standard curve range of 0-800 mg of gallic acid /mL. The variation in each compound brought about by storage (ΔM_i) was expressed as the change in the amount of the compound referred to the fresh grapefruit content, according to equation (1).

Antioxidant capacity. Antioxidant capacity (AOC) was assessed using the free radical scavenging activity of the samples (0.1 mL) evaluated with the stable radical DPPH (Sánchez-Moreno et al., 2003). At 25 ºC, a Thermo Electron Corporation spectrophotometer (USA) was used to measure the absorbance at 515 nm at 0.25 min intervals until the reaction reached the steady state. Appropriately diluted jam samples were used on the day of preparation. The percentage of DPPH (% DPPH) was calculated following equation (2):

$$\%\ \text{DPPH} = \frac{(A_{control} - A_{sample})}{A_{control}} \times 100 \qquad (2)$$

where $A_{control}$ is the absorbance of the control (initial time) and A_{sample} the absorbance of the sample at the steady state.

Statistical analysis

Significant differences among treatments and storage time were evaluated by means of the analysis of variance (ANOVA). Values of $p<0.05$ were considered to represent a significant effect. Furthermore, a correlation analysis was carried out between the antioxidant activity and all the studied components with a 95 % significance level. These statistical analyses were performed using Statgraphics Plus 5.1. To study the relationships

between the samples and their initial bioactive compound content, a Principal Component Analysis (PCA) was applied using SPSS program version 16.0.

Results and Discussion

Effect of treatment on analysed compounds

Table 1 shows the physicochemical and compositional parameters of fresh fruit, osmodehydrated grapefruit and jams. In general, the values of *Star Ruby* grapefruit used as raw material in this study were similar to those obtained by other authors (Rouseff et al., 1992; Peiró et al., 2006; Chun et al., 2006; Kirit and Ozdemir, 2007; Xu et al., 2008; Igual et al., 2010a). To explore the main relationships between the studied samples and the analyzed compounds, a PCA was carried out (Figure 1). The first two components accounted for about 85% of the overall variance. The first component (C1), explaining 52.98% of the variability, was associated with vitamins A (r=0.96), and E (r=0.97), AA (r=0.96), TP (r=0.89), TC (r=0.84) and AOC (r=0.90) values. The fresh fruit showed the highest values of all these compounds associated with C1, followed by ODG. All the jams appeared on the left-hand side of the plot, due to the fact that they have a smaller quantity of these compounds, especially the OD+MW jam. The second component (C2) accounted for 32.20 % of the variability and it was mainly associated with CA (r=0.97), MA (r=0.87), TA (r=0.53) and vitamin C (r=0.89) values. In this case, the jams submitted to a more intense thermal treatment (conventional and MW) appeared in the upper part of the plot mainly due to the higher values of CA, MA and TA. These jams contained a greater quantity of these compounds than even the fresh fruit. In this sense, heating has been described as a means of enhancing the release of bound compounds, leading to a higher content after processing if compared to fresh commodities (Leong and Oey, 2012; García-Martínez et al., 2012). Osmodehydrated grapefruit and the jams obtained from it, OD and

OD+MW, were placed in the opposite part of the plot with the lowest values of the associated compounds.

As shown in Table 1, the grapefruit reached ≈30 ºBrix after the osmotic treatment. ODG showed a significantly ($p<0.05$) lower content of hydrosoluble compounds (TA, MA, CA, AA, vitamin C) when compared to the fresh fruit, probably as a result of the flow of these compounds into the osmotic solution (Peiró et al., 2006). As a consequence, the jams obtained from OD fruit (OD and OD+MW ones) were the ones that presented significantly ($p<0.05$) lower values of MA, CA, AA and vitamin C with respect not only to the other MW and Conventional jams but also if compared with fresh fruit and ODG. The AA degradation during the processing depends on the warming degree (temperature and time), the outflow of fruit, the pH and the presence of metals and oxygen (Eitenmiller and Laden, 1999). AA is easily oxidized to DHAA which also has vitamin C activity and, furthermore, this reaction is reversible. DHAA is less stable than AA but its nutritional value is essentially the same (Russell, 2004), so the sum of AA and DHAA content is assumed as vitamin C. When compared to grapefruit samples (fresh fruit, ODG), the greater difference between vitamin C and AA content in jams indicates that jam processing provoked the oxidation of AA to DHAA. Of the jams, OD and OD+MW showed the highest a_w and pH values and this fact could be connected to the greater AA oxidation observed (Lesková et al., 2006). As expected, there was no observed loss of carotenoids caused by their outflow from the fruit into the OS, since these compounds are not hydrosoluble. Nevertheless, when a thermal treatment was applied to obtain jams (conventional, MW and OD+MW) significant ($p<0.05$) carotenoid losses (≈50%) were quantified. As other authors have observed, the carotenoid's instability is mainly due to its oxidative degradation (Meléndez-Martínez et al., 2004). In this sense, factors such as grinding, thermal treatments, light and oxygen exposition or pH can provoke important changes in these compounds (Rodríguez-Amaya, 1999). As regards TP, the observed loss in the jams was similar to that observed by other

authors working with blueberry, raspberry and blackberry jams (Amakura et al., 2000). The cell disintegration that occurred during the jam making processes can facilitate the oxidation reactions of these compounds (Pinto et al., 2007) although, in this case, the heat treatment seemed to be mainly responsible for the observed losses.

In this way, Fig. 1 shows how the studied compounds allow two groups of jams to be established, one including MW and Conventional ones and the other one with OD and OD-MW jams. From these results, the MW process can be proposed as a good alternative to conventional heating as a means of obtaining jams. The process is shorter in time and the nutritional and functional value of the product is maintained. Nevertheless, despite the absence or the milder thermal treatment applied when osmodehydrated fruit is used, this technique would not be recommended due to the greater loss in the nutritional and functional values of the obtained product, due to the hydrosoluble character of most of the studied compounds.

Evolution of the analysed compounds of obtained jams during storage

The water content, ºBrix, pH and water activity during the storage period oscillated between 0.50 - 0.54 $g_{water}/g_{product}$, 46 - 49 $g_{soluble\ solids}/g_{product's\ liquid\ phase}$, 3.22 - 3.40 and 0.916 - 0.946, respectively. No significant ($p>0.05$) change in the analyzed physicochemical parameters was observed during storage. The evolution of TA, MA, CA and AA content, referred to fresh fruit, during storage is presented in figure 2. The jams obtained by applying a more intense thermal treatment in their preparation (conventional and MW) showed greater TA and MA losses during storage than the rest of the jams. However, only the OD+MW sample showed CA loss at the end of storage.

As regards AA loss, all the jams followed the same trend. At the beginning of storage (1-2 weeks), a sharp decrease in AA was observed. At the end of storage, the MW jam showed the lowest AA loss (26%) and the jams obtained with osmodehydrated fruit the greatest,

38% and 34% for OD and OD+MW, respectively. Quenzer and Burns (2006) also observed a higher AA retention in microwaved spinach samples than when treated in the traditional way. The vitamin C content during the storage of fruit-based products depends on the storage conditions, mainly temperature and the presence of oxygen and light (Klimczak et al., 2007). A similar trend was observed when comparing the vitamin C loss during storage (Figure 3) with the AA loss for this period (Figure 2). Vitamin C also showed a sharp decrease in the first week then stabilizing until the end of the storage period.

The change that took place in the vitamin A content of jams during storage (Figure 3) was similar in every sample. During the first 45 days, no important changes in this vitamin were observed, only an increase in the conventional one. From that moment onwards, a significant ($p<0.05$) decrease in all the samples was observed until the end of storage. The loss of vitamin A in jams caused by storage was between 3 and 7%. As far as the loss of vitamin E was concerned (Figure 3), an early decrease in the first 7 days was observed in OD and OD + MW jams. At the end of the storage period, the conventional jam lost the lowest amount of this vitamin while the OD jam was observed to lose the highest amount. Considering the three vitamins together, conventional and MW jams, which is to say those obtained by applying intense heating treatments, presented the lowest vitamin loss after storage. Sánchez-Moreno et al. (2003) point to the important role played by the heating treatment in vitamin C stability, due to the inactivation of enzymes that degrade this vitamin during storage, such as ascorbate oxidase. Something similar seems to occur with vitamin E. Vitamin A was shown to be the most stable during storage.

Figures 4 to 6 show the loss of TC, TP and antioxidant capacity in the different jams during storage. In Figure 4 it can be observed that, there was a greater loss of TC in the samples elaborated from osmo-dehydrated fruit (61% to 37% for OD and OD + MW, respectively) during the first week of storage, while in the conventional and MW jams this occurred after 15 days (30%) and 1 month (43%), respectively. After that, the TC content remained

constant until the end of the study, except for OD jam that again showed a significant ($p<0.05$) loss of carotenoids from day 45 to 60 and until the end of storage. A gradual loss of phenolic compounds during the storage period was observed for all the jams (Figure 5). At the end of the storage, the greatest TP loss was observed in OD jam while no significant ($p>0.05$) differences were found among the other jams. All the samples suffered an antioxidant capacity loss during the storage period (Figure 6). OD jam was the most stable, with a DPPH reduction of 21%, followed by Conventional (35 %), OD+MW (46%) and MW (49%).

A statistical correlation was carried out to explain the relationships among the phytochemical constituents quantified in the samples and also with the antioxidant capacity. Table 2 shows the Pearson correlation coefficients between each pair of variables. All correlation coefficients were statistically significant ($p<0.05$) in most cases and positive, except that of the pair of variables CA-total phenols. All the analyzed compounds, except the TA, showed a significant ($p<0.05$) correlation with the antioxidant capacity. The greatest contribution to the antioxidant capacity was provided by the vitamin A content, followed by the vitamin E, AA and the total carotenoids. Although there is some controversy about the influence of the phytochemicals present in fruits and vegetables with their antioxidant capacity (Guo et al., 2003), the results obtained in this work are consistent with some other studies where the contribution of AA and the phenolic compounds to the antioxidant capacity has been described (Xu et al., 2008; Tavarini et al., 2008). In addition, a significant ($p<0.05$) linear relationship was found between malic and citric acids and antioxidant capacity. The organic acids can behave like synergistic antioxidants, acting as complexing agents on inorganic metal ions, which in turn can catalyse the degradation, preventing or slowing antioxidants degradation and increasing its stability (Biolatto et al., 2005).

Conclusion

From this study, when compared to the conventional process, microwave energy could be recommended as a means of obtaining jam, since its application represents a 50% reduction in processing time and allows a product to be obtained which has a very similar nutritional and functional value. As a technique for concentrating the product, fruit osmodehydration is not recommended due to the hydrosoluble nature of most of the nutritive compounds, which will be lost during the process. Moreover, thermally treated jams are better at retaining vitamins and phenolic compounds during storage.

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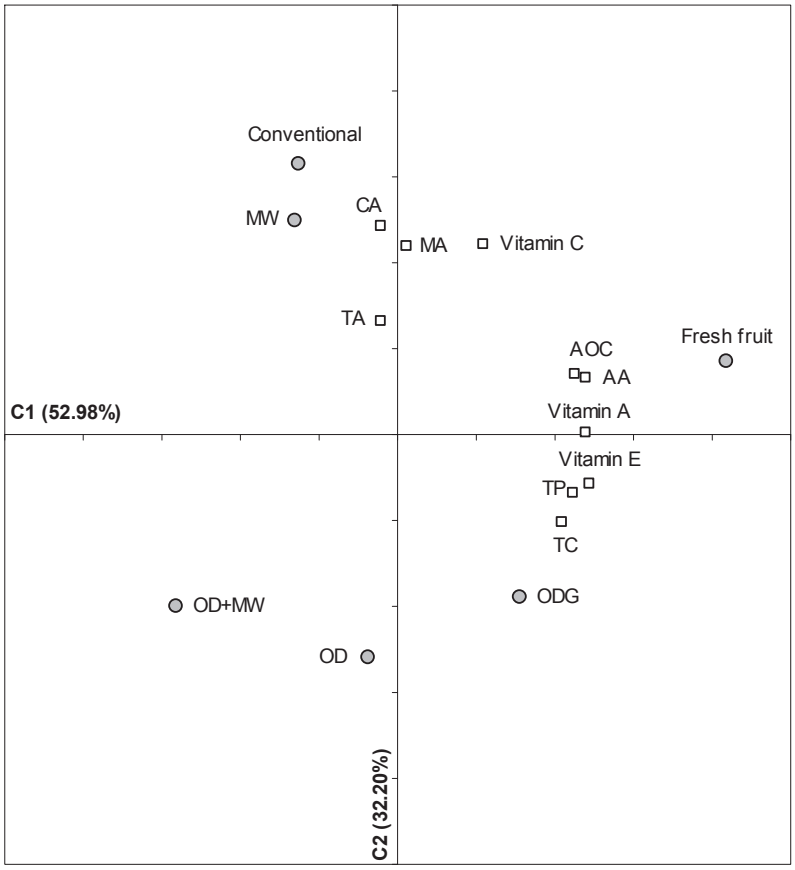

Figure 1. Principal Component Analysis (PCA) of tartaric acid (TA), malic acid (MA), citric acid (CA), ascorbic acid (AA), vitamins A,C and E, total phenols (TP), total carotenoids (TC) and antioxidant capacity (AOC) of fresh fruit, osmodehydrated fruit (ODG) and jams newly processed by osmotic dehydration (OD), microwave (MW), combination of osmotic dehydration and microwave (OD+MW) and conventional process.

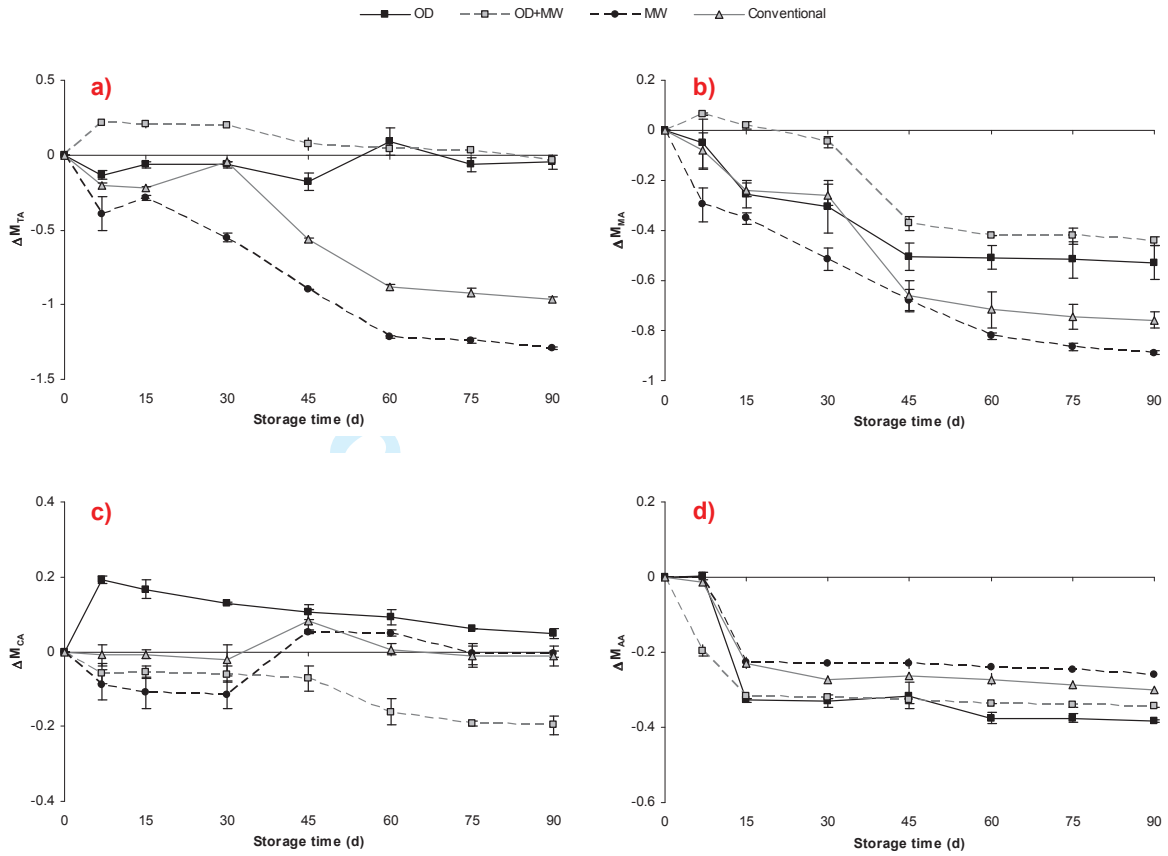

Figure 2. Change in tartaric acid (TA) (a), malic acid (MA) (b), citric acid (CA) (c), ascorbic acid (AA) (d) content of jam processed by osmotic dehydration (OD), microwave (MW), combination of osmotic dehydration and microwave (OD+MW) and conventional process during 3 month of storage.

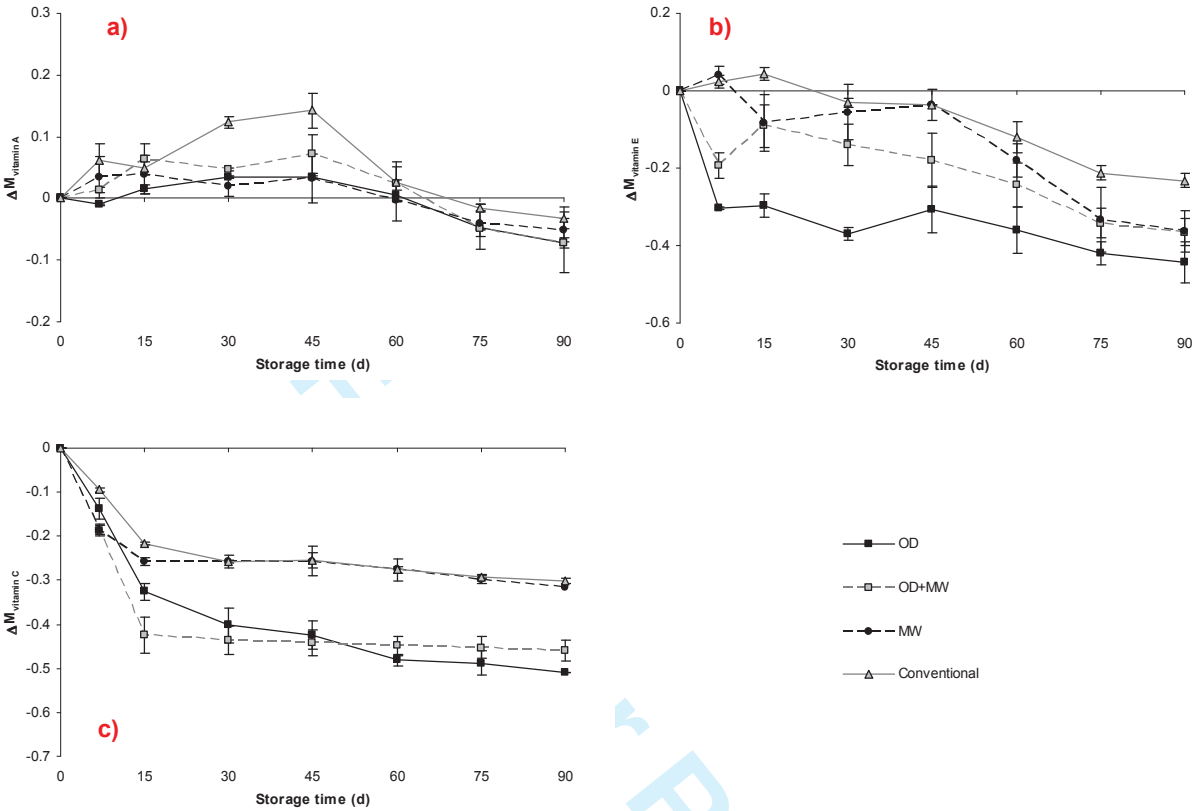

Figure 3. Change in content of vitamins A (a), C (b) and E (c) of jam processed by osmotic dehydration (OD), microwave (MW), combination of osmotic dehydration and microwave (OD+MW) and conventional process during 3 month of storage.

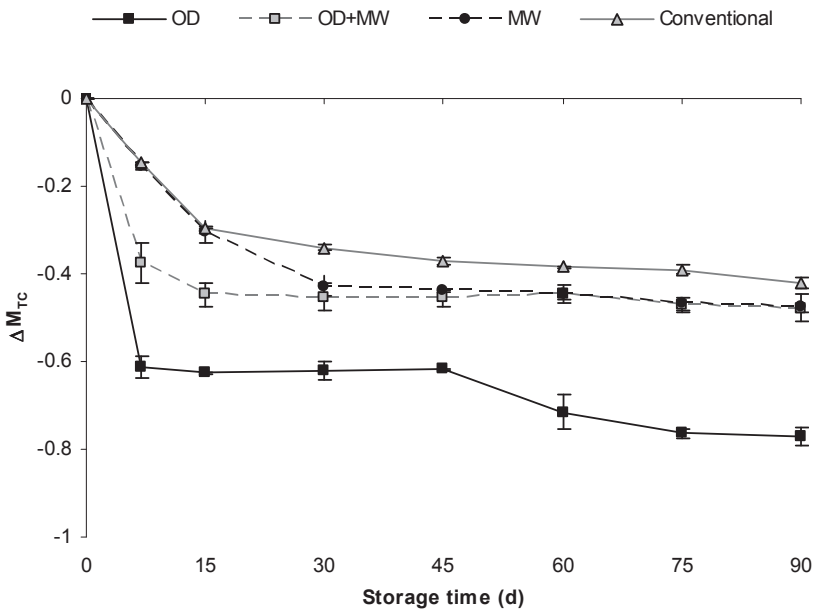

Figure 4. Change in total carotenoids (TC) of jam processed by osmotic dehydration (OD), microwave (MW), combination of osmotic dehydration and microwave (OD+MW) and conventional process during 3 month of storage.

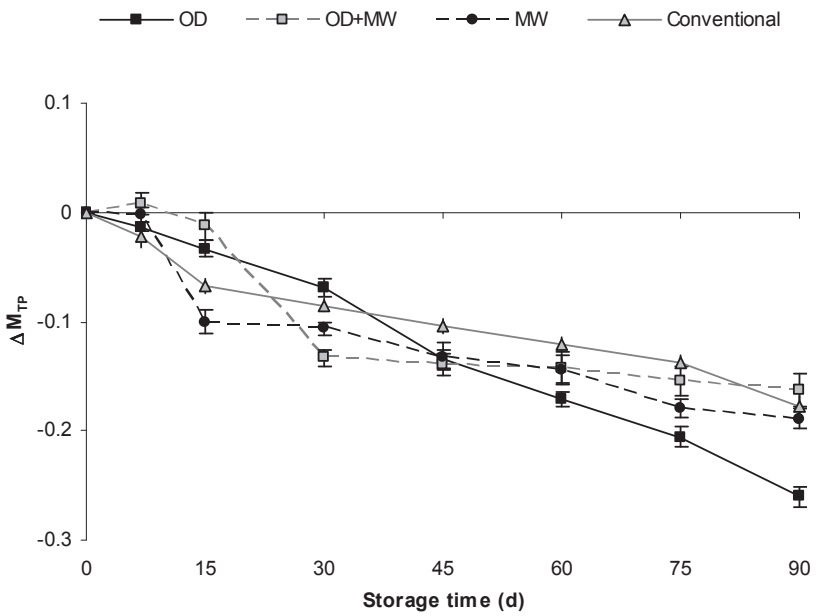

Figure 5. Change in total phenols (TP) of jam processed by osmotic dehydration (OD), microwave (MW), combination of osmotic dehydration and microwave (OD+MW) and conventional process during 3 month of storage.

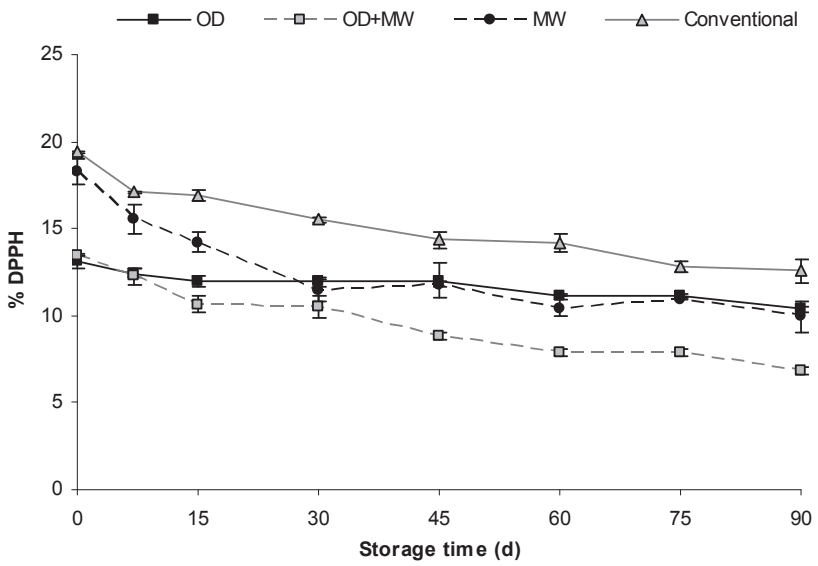

Figure 6. Change in the antioxidant capacity (%DPPH) of jam processed by osmotic dehydration (OD), microwave (MW), combination of osmotic dehydration and microwave (OD+MW) and conventional process during 3 month of storage.

Table 1. Mean values (with standard deviation) of ºBrix, a_w, x_w, pH, tartaric acid (TA), malic acid (MA), citric acid (CA), ascorbic acid (AA), vitamins A,C and E, total phenols (TP), total carotenoids (TC) and %DPPH in fresh fruit, ODG and obtained jams.

	Fresh fruit	ODG	Jams			
			OD	OD+MW	MW	Conventional
ºBrix[1]	12.0 (0.2)^{f}	29.6 (0.2)^{e}	46.1 (0.2)^{c}	46.0 (0.2)^{c}	47.7 (0.2)^{b}	48.5 (0.2)^{a}
a_w	0.989 (0.003)^{a}	0.972 (0.003)^{b}	0.945 (0.003)^{c}	0.942 (0.003)^{d}	0.924 (0.003)^{e}	0.922 (0.003)^{f}
x_w[2]	0.882 (0.002)^{a}	0.703 (0.002)^{b}	0.541 (0.002)^{c}	0.537 (0.002)^{d}	0.529 (0.002)^{e}	0.526 (0.002)^{f}
pH	3.27 (0.02)^{b}	3.28 (0.02)^{b}	3.39 (0.02)^{a}	3.40 (0.02)^{a}	3.27 (0.02)^{b}	3.25 (0.02)^{c}
TA[3]	314 (10)^{b}	246 (2)^{c}	301 (4)^{b}	260 (3)^{c}	223 (12)^{d}	553 (3)^{a}
MA[3]	558.7 (0.2)^{c}	412 (10)^{e}	476 (34)^{d}	336 (12)^{f}	747 (6)^{a}	632 (22)^{b}
CA[3]	1318 (5)^{b}	1159 (12)^{c}	888 (9)^{d}	1049 (25)^{d}	1720 (15)^{a}	1726 (12)^{a}
AA[3]	35.6 (0.4)^{a}	29.2 (0.8)^{b}	23.4 (0.6)^{e}	20.0 (0.3)^{f}	24.6 (0.2)^{d}	26.1 (0.2)^{c}
Vitamin A[3]	1.21 (0.12)^{a}	0.89 (0.04)^{b}	0.30 (0.05)^{cd}	0.24 (0.02)^{d}	0.29 (0.03)^{cd}	0.393 (0.012)^{c}
Vitamin C[3]	36.8 (0.3)^{a}	31.8 (0.2)^{c}	30.3 (0.7)^{d}	30.0 (0.4)^{d}	35.9 (0.4)^{ab}	35.5 (0.3)^{b}
Vitamin E[3]	0.161 (0.003)^{a}	0.134 (0.003)^{b}	0.113 (0.006)^{c}	0.084 (0.006)^{d}	0.088 (0.010)^{d}	0.086 (0.002)^{d}
TP[3]	136 (2)^{a}	133.4 (0.8)^{ab}	132.9 (0.5)^{b}	118 (1)^{d}	122.2 (0.9)^{c}	122.8 (0.9)^{c}
TC[3]	8.70 (0.04)^{a}	8,42 (0,01)^{ab}	8.32 (0.01)^{b}	4.6 (0.3)^{e}	5.4 (0.1)^{c}	5.1 (0.2)^{de}
%DPPH	43.7 (0.4)^{a}	23 (1)^{b}	13.1 (0.4)^{d}	13.45 (0.02)^{d}	18.3 (0.7)^{c}	19.39 (0.03)^{c}

The same letter in superscript within rows indicates homogeneous groups established by the ANOVA ($p<0.05$).
Units: [1]g soluble solids / 100 g liquid phase of sample, [2]g water / g sample, [3]mg / 100 g fresh fruit.

Table 2. Pearson correlation coefficients among studied compounds (TA: tartaric acid; MA: malic acid; CA: citric acid; AA: ascorbic acid; TP: total phenols) and antioxidant capacity (% DPPH).

	TA	MA	CA	AA	Vitamin A	Vitamin C	Vitamin E	TP	TC
% DPPH	0.1833	0.5803*	0.2519*	0.8106*	0.8945*	0.6613*	0.8313*	0.631*	0.7537*
TA		0.5787*	0.4356*	0.2625*	0.1419	0.3531*	0.3148*	0.1283	0.0684
MA			0.4565*	0.7683*	0.3679*	0.7989*	0.7003*	0.6693*	0.6306*
CA				0.4461*	0.2176	0.6551*	0.2119	-0.0470	0.0506
AA					0.6703*	0.9144*	0.8251*	0.7100*	0.8506*
Vitamin A						0.5077*	0.8020*	0.5298*	0.6374*
Vitamin C							0.7135*	0.5496*	0.6918*
Vitamin E								0.767*	0.8533*
TP									0.8224*

*significant differences at the 0.05 level