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Effect of the re-use of the osmotic solution on the stability of osmodehydro-refrigerated grapefruit

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Abstract

Osmotic dehydration (OD) of grapefruit (55ºBrix sucrose solution, 30 ºC) was carried out to obtain ~ 75 g water/100 g sample in the final product. Although the grapefruit was replaced each time, the osmotic solution (OS) was reused for five OD cycles, with or without pasteurization. The samples obtained in cycles 1, 3 and 5, were stored at 10 ºC. Changes in ºBrix, water content, water activity, pH, total acidity, ascorbic acid content, cation concentration, respiration rate and total microbial counts at different storage times were analysed and compared to fresh-cut grapefruit stored under the same conditions. During OD, a partial loss of the natural soluble substances present in the fruit was observed. In terms of the dehydration level reached by the fruit, it is possible to reuse the OS in up to 5 OD, without any reconcentration treatment. Nevertheless, it is advisable to pasteurize the OS before

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each cycle in order to obtain a product with a shelf-life of between 7 and 12 days in refrigeration, depending on the number of cycles.

Keywords: *osmotic dehydration, ascorbic acid, shelf-life, microbial stability, respiration rate.*

Runnig tittle: Stability of osmodehydrated grapefruit

1. Introduction

Over the last few years, consumer demand for fresh, ready-to-use products has led to an increased interest in minimally processed fruits and vegetables, as these products combine freshness and convenience. Moreover, the role played by the antioxidant properties of many fruits and vegetables in the prevention of degenerative diseases is widely acknowledged (Kaur & Kapoor, 2001). Of the antioxidant vitamins, vitamin C plays a relevant role in human health and citrus fruits are an important source of this vitamin (Biolatto, Salitto, Cantet & Pensel, 2005; Igual, García-Martínez, Camacho & Martínez-Navarrete, 2010). Besides ascorbic acid, grapefruit contains flavanone glycosides, such as hesperidin, narirutin and naringin, and other compounds with antioxidant capacity (Del Caro, Piga, Vacca & Agabbio, 2004; Gil-Izquierdo, Gil, Ferreres & Tomás-Barberán, 2001; Peiró, Dias, Camacho & Martínez-Navarrete, 2006).

The preliminary operations needed to obtain minimally processed foods, such as peeling or cutting, result in cell breakdown with subsequently increased enzyme activity and the acceleration of physiological reactions while, at the same time, providing favourable conditions for microbiological growth depending on the water content. Quality loss occurs due to enzymatic browning, firmness reduction, off-flavour development, a decrease in nutritional value and microbiological growth (Pretel, Fernández, Romojaro & Martínez, 1998; Watada, Abe & Yamauchi, 1990), all of which depend on the storage time and temperature and also on the packaging used, such as passive or active modified atmosphere packaging and the use of edible coatings (Gunes & Chang Lee, 1997; Zagory & Kader, 1988).

The reduction of water activity (a_w) has been proposed as a preservation method to obtain minimal processed fruits. Nevertheless, this reduction must be carefully controlled to preserve the fresh-like quality demanded for the product. In the range of high water activity, a small decrease of a_w supposes a very important decrease in the in the relative rate of all deteriorative reactions and microbial growth. In this sense, many minimal processed fruits have an a_w of 0.98 or above (Willey, 1994). Osmotic dehydration at mild temperatures has been widely accepted as a technique for obtaining, in reasonable process times, processed fruits that somewhat preserve their fresh-like characteristics prolonging its shelf-life. The use of vacuum in osmotic dehydration improved mass transfer kinetics (Fito & Chiralt, 1997). This operation implies a two-way mass transfer process: mainly water, but also some natural soluble substances such as vitamins, organic acids or phytochemicals flow out from the fruit to the OS (García-Martínez, Martínez-Monzó, Camacho & Martínez-Navarrete, 2002; Peiró et al., 2006; Peiró-Mena, Camacho & Martínez-Navarrete, 2007; Valdez-Fragoso, Welti-Chanes & Giroux, 1998), while soluble solutes are transferred from the solution to the fruit, which may change product taste and acceptability. This method has received considerable attention due to the low amount of energy required (Taiwo, Angersbach, Ade-Omowaye & Knorr, 2001) and the improvement in fruit quality (Panagiotou, Karathanos & Maroulis, 1998). As no high temperatures are normally used in OD processes and no water phase changes occur, the changes in sensory attributes, such as colour, aroma, flavour and texture, are minimised (Chiralt et al., 2001; Escriche, Chiralt, Moreno & Serra, 2000; Raoult-Wack, 1994; Talens, Escriche, Martínez-Navarrete & Chiralt, 2002).

One limitation of the OD process is the management of the osmotic solution. To solve this problem, the reuse or recycle of OS in successive dehydration cycles

without any reconcentration treatment may be proposed. The number of cycles will be mainly limited by its dilution related to the dehydration level of the obtained fruit and also to microbiological aspects. In this way, OD could become a more economical, environmentally friendly process, obtaining products with the maximum nutritional and functional values.

The objective of this work was to assess the effect of the reuse of the OS on the stability of the osmodehydrated grapefruit during refrigerated storage, measured through changes in composition (soluble solids, water content, total acidity, ascorbic acid content, cation concentration), water activity, pH, respiration rate and microbial growth.

2. Materials and methods

2.1. Raw material

Grapefruits (*Citrus paradise*), of the cultivar Star Ruby, were purchased in a local market in Valencia (Spain). Whole grapefruits were selected on the basis of a similar degree of ripeness (ratio ºBrix/acidity ≈ 9) and apparent fruit quality (color and firmness). They were stored in refrigerated chambers at 10 ºC and at 85-90% relative humidity until they were used (less than 24h). Prior to treatments, whole grapefruits were washed and peeled and cut into 1cm thick slices, which were then cut in half.

Sucrose (food grade commercial sugar) was used to prepare a 55ºBrix osmotic solution, used as osmotic agent. The sugar was mixed with heated (30 ºC) distilled water until total dissolution.

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103 *2.2. Osmotic dehydration*

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105 For the OD process, the grapefruit samples were immersed in a plastic beaker
106 filled with 55ºBrix sucrose syrup. A plastic screen was placed on the beaker to keep
107 the slices totally immersed in the solution and separated from the stirrer working at
108 250 rpm (Heidolph Instruments, RZR 2102 control, Schwabach, Germany). OD was
109 carried out for 3 h placing the beaker in a temperature-controlled water bath at 30 ºC
110 (J.P. Selecta S.A., Precisterm S-141, Barcelona, Spain) and 50 mbar pressure for the
111 first 10 min of the process, afterwards restoring atmospheric pressure in order to
112 promote the sample's vacuum impregnation with the OS. Dehydration time was
113 selected based on the results obtained in previous osmotic dehydration kinetics
114 studies (Moraga, Moraga, Fito & Martínez-Navarrete, 2009), to obtain samples with
115 75 g water/100 g.

116 The ratio of osmotic solution to fruit was 10:1. Five consecutive OD cycles
117 were carried out using the same OS, not re-concentrated, but having renewed the
118 fruit for each OD cycle. Two series of OD were carried out, with and without a mild
119 thermal treatment of the OS to pasteurize it before each OD cycle. The thermal
120 treatment consisted of heating the solution from 30 to 72 ºC in 7 min, maintaining the
121 last temperature for 15 seconds. Both series of samples were analyzed for microbial
122 growth. Additionally, only samples obtained when OS was thermally treated were
123 also submitted to the rest of the analysis described as follows.

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125 *2.3. Analysis*

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Fresh-cut and osmodehydrated grapefruit pieces obtained after the first, third and fifth consecutive OD cycles, with and without the previously described thermal treatment of the OS, were analyzed as to their microbiological stability. Samples (stored in PET packages at 10 ºC) were analyzed in duplicate as to their total microbial count and yeasts and moulds, using Plate Count Agar (Scharlab, Barcelona, Spain) for 48–72 h at 30 ºC and Sabouraud Chloramphenicol Agar (Scharlab, Barcelona, Spain) for 3–5 days at 30 ºC, respectively. Sample dilutions were prepared and, after the incubation period, Petri dishes with a number of colonies of between 30 and 300 for total count and 0 and 30 for moulds and yeast were considered. Results were expressed as colony forming units (cfu) per g of sample.

Fresh-cut (FG) and osmodehydrated grapefruit obtained after the first, third and fifth consecutive OD cycles (ODG1, ODG3 ODG5, respectively), with the thermal treatment applied to the OS before each OD cycle, were stored at 10 ºC in PET packages and analyzed at different storage times (less than 15 days) to determine the water activity, ºBrix, water content, pH, titrable acidity, ascorbic acid content, cation (Ca^{+2}, Na^{+}, Mg^{+2}, K^{+}) concentration and respiration rate. In order to determine the ascorbic acid content, each grapefruit was identified and analyzed before and after each OD cycle (FG1, FG3, FG5, respectively) and also during storage, to better control the changes in the amount of this compound. The OS was also analyzed as to ºBrix, ascorbic acid content and cation (Ca^{+2}, Na^{+}, Mg^{+2}, K^{+}) concentration after each OD cycle.

The a_w was measured using a dew point hygrometer (Decagon, AquaLab CX-2, Washington, U.S.A.), the total soluble solids (ºBrix) with a 20 ºC thermostated refractometer (ATAGO CO., ABBE 3T, Tokyo, Japan), the water content (x_w) by

drying in a vacuum oven at 60 ºC till constant weight was reached (AOAC method 934.06 (2000)), the pH using a Crison micropH 2001 pHmeter, the titrable acidity (referred to as citric acid) by using AOAC method 942.15, (2000) and the ascorbic acid (AA) content by using the 2,6-dichloroindophenol titrimetric method (AOAC method 967.21, 2000). In all cases, grapefruit samples were previously homogenized at 8000 rpm (IKA®, ULTRA-TURRAX T25, Staufen, Germany). To determine the ascorbic acid content, the juice of the homogenized samples was previously extracted by centrifugation (J.P. Selecta S.A., Medifriger-BL, Barcelona, Spain) for 10 min at 10000 rpm. Measurements were taken in triplicate.

Cation quantification was carried out by means of an ion chromatograph (Methrom Ion Analysis, Herisau, Switzerland), using a universal standard column (Metrosep C2-150, 4.0 x 150 mm) along with an eluent composed of tartaric acid (4.0 mmol/L) and dipicolinic acid (0.75 mmol/L), equipped with electronic detectors. In every case, the fruit samples were previously homogenized and centrifuged (J.P. Selecta S.A., Medifriger-BL, Barcelona, Spain) for 10 min at 12000 rpm, to remove 1 mL of supernatant. Measurements were taken in duplicate.

A closed or static system was chosen to measure the respiration rate. Samples (≈ 150 g) were placed in 884 mL hermetic glass containers provided with a septum and stored in a temperature controlled chamber (J.P. Selecta S.A., Hot-Cold M, Barcelona, Spain) at 10 ºC for 6 days. Two replicates were performed in each sample. Volume samples of air from the headspace were withdrawn, at different times, with a needle connected to a gas analyzer. A head-space-gas analyzer, (PBI Dansensor A/S, CheckMate 9900, Ringsted, Denmark), was used to determine the O_2 and CO_2 contents inside the hermetic glass containers. Gas sampling was carried out every 30 or 60 min during the first two hours and every 60 or 90 min until the 8h

measurement period was up. After this, the containers were opened to renew the ambient air of the headspace. The respiration rate, expressed as CO_2 production rate ($RRCO_2$, $mLCO_2 \cdot kg^{-1} \cdot h^{-1}$), was calculated from equation 1.

$$y_{CO_2}^{t} = y_{CO_2}^{t_o} + \left[100\ RR_{CO_2}\frac{M}{V}\right]t \quad (1)$$

where $y_{CO_2}^{t_o}$ is the gas concentration in the headspace (mL CO_2 /100mL) at the beginning of the experiment and $y_{CO_2}^{t}$ after each time of measurement (t), M is the mass of the fresh-cut samples (kg) and V the volume (mL) of headspace. V was calculated from the volume of the glass and the volume of samples obtained from its mass and density. $RRCO_2$ values were referred to fresh-cut sample mass (M) to make comparisons possible.

Analyses of variance (ANOVA) were applied to evaluate the differences among treatments, using Statgraphics®Plus 5.1. software.

3. Results and discussion

3.1. Microbial growth

Figure 1 shows both total microbial counts and yeasts and moulds analysed, during refrigerated storage, in fresh-cut and osmodehydrated grapefruit after 1, 3 and 5 consecutive OD cycles, without submitting the OS to a thermal treatment before any OD cycle.

Due to the low pH of citrus fruits, most of the microbial alterations are due to the yeasts and some just some ones are due to moulds, without the existence of pathogenic microorganisms. The established limit of microbiological growth used to determine the shelf-life of each sample was one of the most restrictive found in foods (Pascual & Calderón, 2000): that of total microbial counts was 10^4 cfu/g and that of yeasts and moulds, 10^2 cfu/g. In all cases, the limit of 10^2 cfu/g of yeasts and moulds was reached quicker than the limit for total counts, so the first one was used to establish the microbiological shelf-life of samples.

In this sense, the fresh-cut and the osmodehydrated grapefruit obtained in the first use of the OS reached the limit for yeasts and moulds after 5 and 7 storage days, respectively (Fig. 1b). The reuse of the OS supposed an increase in the microbial load.

After the third OD cycle, the microbiological shelf-life of the dehydrated samples was reduced from 7 to 2 days. Samples dehydrated with the OS that had been reused for 5 OD cycles presented a microbial growth which exceeded the limit selected immediately after the treatment.

As expected, applying the thermal treatment to the OS before each OD cycle was recommended. The microbial growth of the osmodehydrated grapefruit samples after 1, 3 and 5 OD cycles (ODG_1, ODG_3 and ODG_5), when a thermal treatment was applied to the OS between cycles, is shown in Figure 2 as a function of storage time. Applying the previously mentioned limit for the counts of moulds and yeasts (10^2 cfu/g), the shelf-life of osmodehydrated samples was 6, 12 and 9 days in the samples ODG1, ODG3 and ODG5, respectively. In the first cycle, the microbial growth in the dehydrated fruit was practically the same as that observed when no treatment was applied, probably due to the low microbial load in the initial OS. Despite the thermal

225 treatment applied to the OS before each OD cycle was a mild one, in order to avoid
226 losses in functional compounds, successive thermal treatments associated to each OD
227 cycle seem to affect the microbial count of the OS implying an improvement of
228 microbiological fruit quality. On the other hand, the introduction of each fruit batch
229 supposes an increase in the microbial load. Both aspects, together with the lower pH
230 of the fruit obtained in the third cycle (Table 1), could contribute this sample to be
231 the best preserved during storage.

232 On the basis of the aforementioned results, the reuse of the OS applying a
233 mild thermal treatment before each OD cycle is recommended from the microbial
234 point of view. Samples osmotically dehydrated by a heat-treated OS were used in the
235 rest of the analysis.

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237 *3.2. Water content, ºBrix and water activity*

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239 As has been discussed, another limit to the reuse of the OS in successive OD
240 cycles without any reconcentration treatment will be its dilution, which could affect
241 the dehydration level reached by the fruit. In this sense, in order to evaluate the
242 possibility of reusing the OS up to 5 OD cycles, the compositional changes in x_w,
243 ºBrix and a_w were analysed in grapefruit before (FG) and after dehydration, using the
244 OS for 1, 3 and 5 OD cycles (ODG1, ODG3 and ODG5 samples, respectively)
245 (Table 1). Table 1 also shows the evolution of x_w, ºBrix and a_w during cold storage
246 for 10 days.

247 The mean x_w, ºBrix and a_w of the fresh grapefruit batch used in this study was
248 87.20 ± 0.06, 12.27 ± 0.06 and 0.987 ± 0.003, respectively. After the OD treatment,
249 the expected reduction in x_w, the increase in ºBrix and, therefore, the decrease in the

a_w of the samples were observed. Comparing the newly dehydrated samples (storage time = 0) after the different OD cycles (1, 3 and 5), a significant effect ($P<0.05$) of the reuse of the OS on the dehydration level reached by the fruit was observed (Table 1). Samples dehydrated with OS which had not been previously used (ODG1) showed significantly ($P<0.05$) lower x_w and a_w and higher ºBrix than samples dehydrated with the reused OS (ODG3 and ODG5), although the differences were not as marked during their storage. Moreover, the dehydration level was higher in ODG5 than in ODG3, which is not coherent with the progressive dilution of the osmotic agent. In this sense, the significant differences in the composition of OD samples seem to be more closely related to the natural variability of this kind of raw material than to the effect of the OS dilution (Peiró et al., 2006). The evolution of the ºBrix in the OS, during its reuse up to 5 OD cycles, showed a linear behaviour from an initial value of 55.05 ± 0.07 to 51.85 ± 0.07 ºBrix (Fig. 3). From this point of view, the OS could be reused during 5 OD cycles without any reconcentration treatment. The results coincide with those reported by other authors when the OS was reused under the same experimental conditions during the OD of kiwifruit, pineapple and grapefruit (García-Martínez et al., 2002; Peiró et al., 2006; Peiró-Mena et al., 2007).

During the storage period, significant differences ($P<0.05$) in x_w, ºBrix and a_w were obtained in all the studied samples (Table 1). Nevertheless, there was no clear attributable tendency of the changes to the storage time, and they can also be caused by the natural variability of the raw material more than by the storage effect.

3.3. Titrable acidity, pH and ascorbic acid

The dehydration treatment also implied a significant ($P<0.05$) decrease in the acidity of the samples, expressed as the citric acid (CA) content, the major organic acid in grapefruit (Table 2). In dehydrated samples, the results have also been referred to the corresponding fresh sample in order to compare differences and to calculate the losses experienced during the OD treatments, expressed as mg of CA lost by each 100 mg of CA present in the corresponding fresh grapefruit. These losses in CA were lower when the OS was reused, ranging from 34 to 23%, probably due to the enrichment of the OS in the natural acids extracted from fruit in the successive OD cycles, thus reducing concentration gradients that favour the leaching out of these compounds.

During the storage period, significant changes ($P<0.05$) in the titrable acidity and pH of samples were observed (Table 2). The fresh-cut grapefruit presented losses in the CA content, ranging from 16 to 30%, as well as a slight pH increase. During the storage, the CA losses were, in general, much lower in dehydrated samples than in FG. Nevertheless, considering the global effect of the treatment and the storage, the losses in ODG samples were in the same order as those found for the fresh-cut grapefruit at the end of the storage period.

Table 3 presents the results of the ascorbic acid analysis. The different fresh grapefruit samples used presented an AA concentration similar to that found by other authors (Gorinstein et al., 2004). Nevertheless, significant differences ($P<0.05$) between fresh samples were found, due to the natural variability of the fruit.

During the first 24 hours, a sharp decrease in the AA content of FG samples was observed (~ 50%). However, longer storage times did not lead to greater AA losses, the contents being constant from 1 to 6 days of storage. Other studies, such as the one reported by Del Caro et al. (2004), found significant decreases in the AA

analyzed in citrus segments of mandarin and orange during storage at 4 ºC. Nevertheless, Red blush grapefruit juices did not show any significant differences throughout 15 days of storage at the same temperature.

In all cases, the osmotic treatment caused significant losses in the AA content of samples, ranging from 24 to 43%. An additional decrease was observed during storage, especially after 8 days. Two independent mechanisms could be considered to explain these AA losses: losses by diffusion from the fruit tissue into the OS during dehydration and losses due to chemical degradation during processing and storage.

The reaction mechanism of ascorbic acid decomposition in foods has been extensively studied. When oxygen is present, AA degradation occurs simultaneously by oxidative and anaerobic mechanisms, the latter pathway being slower than the oxidative one (Rojas & Gerschenson, 2001). The oxidative degradation of AA is related to ascorbinase activity and by indirect degradation throught polyphenol oxidase, cytochrome oxidase and peroxidase activity (Lee & Kader, 2000). During the first two weeks of storage, the predominant effect is that of the oxidation of the AA to L-dehydroascorbic acid (DHA). From that storage time, the anaerobic degradation becomes predominant (Wong, Stanton & Burns, 1992). The storage time considered in this study was 12 days, with the most important route of AA degradation expected to be the oxidative one.

As was observed in CA, considering the global effect of the treatment and the storage, the AA losses in dehydrated samples were in the same order as those found for the fresh-cut grapefruit after 6 days of storage.

The AA content of the OS slightly increased up to the third OD cycle, subsequently staying constant till the fifth cycle (Figure 3). Therefore, at least a part

of the AA lost by the grapefruit during the osmotic process remains incorporated in the OS, imparting it added value.

3.4. Cation concentration

The analysis of the obtained chromatograms allowed us to obtain the content of major cations (Ca^{2+}, Na^{+}, Mg^{2+} and K^{+}) present in the grapefruit, before and after the dehydration treatments (Table 4). In dehydrated samples, results have also been referred to the corresponding fresh sample so as to compare differences.

The content in calcium, sodium, magnesium and potassium of the fresh and dehydrated grapefruit samples were similar to data reported by other authors (Peiró et al., 2006). As can be observed, and as is typical in fruits, the potassium content was almost 10 times higher than the magnesium or calcium content. Sodium was present in a very low quantity. The osmotic dehydration treatment caused significant losses ($P<0.05$) in all the cations, as has been described by other authors working on different fruits (Peiró et al., 2006; Peiró et al., 2007), except in the case of Na^{+} ($P>0.05$). Although, in general, the losses seemed to be lower when the osmotic solution was reused, no cycle-dependent significant differences ($P>0.05$) were found in the cation content of the dehydrated samples.

In the OS, all the cations (Ca^{2+}, Na^{+}, Mg^{2+} and K^{+}) were seen to be present (Table 5). A linear increase, related to the reuse of the OS, was observed in the concentration of the major grapefruit cation, the potassium, following the equation: $y=2.2575x+1.6808$, $R^2 = 0.9993$. For the remaining cations, except in the case of Mg^{2+}, the increase was not significant ($P>0.05$), probably due to the low concentration present in the OS.

3.5. Respiration rate

Figure 4 represents the respiration rate, in terms of CO_2 generation, of all the samples under consideration and their evolution throughout the storage period, which can be considered as an indicator of the physiological alterations caused by treatments.

During the storage of climacteric fruits, a very steep increase in the CO_2 and ethanol production takes place. In non-climacteric fruits, such as grapefruit, this increase is not so important and only a slight increase in the respiration rate is produced at the arrival of senescence. Nevertheless, if a severe wound is produced in the tissue (like a cut), the stress induces the CO_2 production and, in some cases, the production of ethanol (Brecht, 1995; Taiz & Zeiger, 1991). This can be observed in the evolution of the CO_2 production of the fresh-cut grapefruit samples (Fig. 4), which presented an initially high value, probably in response to the stress generated by the cut, and an abrupt reduction after 24h of storage, increasing from the third day onwards, as a consequence of the arrival of fruit senescence. In the grapefruit samples dehydrated with the osmotic solution that had not been re-used (ODG1), the CO_2 production also presented an initially high value, although it was lower than that observed in the fresh-cut grapefruit. Similar results were observed in strawberry and apple which had been osmotically dehydrated in similar conditions, explained on the basis of the development of fermentative metabolisms associated with cellular alteration during the process (Castelló, Igual, Fito & Chiralt, 2009; Castelló, Fito & Chiralt, 2010). It is known that anaerobic respiration in fruit tissue is characterized by increases in ethanol, ethyl acetate, ethyl butanoate and acetaldehyde during

storage. These compounds seem to reduce the shelf-life of fresh-cut fruits (Dea, Brecht, Nunes & Baldwin, 2010). The above mentioned effect was not observed in the grapefruit dehydrated with the osmotic solution that had been re-used throughout 3 and 5 successive cycles of dehydration. This apparent absence of fermentative processes might be related to the more extended shelf life of these samples (Fig. 2).

The effect of different osmotic dehydration treatments on the respiratory pathway of fruits has been widely studied and related to the different alterations of the cells as a consequence of the structural damage that the dehydration provokes in the cells next to the surface of the cut, as well as to the presence of concentration profiles that lead to profiles of physiological alteration (Castelló, Fito & Chiralt, 2006; Castelló et al., 2009, 2010; Torres, Castelló, Escriche & Chiralt, 2008). Depending on the intensity of the osmotic treatment and the application or not of sub-atmospheric pressures, the number of altered or non-viable cells will vary (Ferrando & Spiess, 2001). The altered cells will present a different respiratory pattern due to the induced stress, whereas the non- viable ones will present no respiratory activity.

In grapefruit samples, the dehydration treatment applied caused an initial decrease in the CO_2 production that was subsequently maintained during storage. This can be a consequence of the reduction in the number of viable cells in the tissue, producing a reduction of the net flow of the cell generation and degeneration gases, and may also be due to the barrier effect of the external collapsed cells and pores. The lowest levels of RR_{CO2} belonged to ODG3 samples, which presented the longest microbiological shelf life (Fig. 2).

4. Conclusions

Osmodehydration treatments (till a_w ~ 0.978) extend the microbiological shelf-life of grapefruit in refrigerated storage conditions. It is possible to reuse the OS in successive OD cycles, without any re-concentration treatment, as the stability of the obtained fruit, related to composition, is not affected by the dilution that takes place in the OS. Nevertheless, a mild thermal treatment is required to ensure the microbiological quality of the osmodehydrated fruit. The benefits of reusing the osmotic solution in successive OD cycles could be deduced not only in economic terms but also in terms of a better preservation of the citric acid in the samples and a prolonged product shelf-life, from 5 days (fresh-cut grapefruit) to 7-12 days, depending on the number of OD cycles.

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524

Figure captions

Figure 1. **(A)** Total microbial counts and **(B)** yeasts and moulds, at different storage times (10 ºC), in fresh-cut (◆) and osmodehydrated grapefruit after 1 (■), 3 (▲) and 5 (●) consecutive OD cycles, without submitting the OS to any thermal treatment before each OD cycle.

Figure 2. **(A)** Total microbial counts and **(B)** yeasts and moulds, at different storage times (10 ºC), in fresh-cut (◆) and osmodehydrated grapefruit after 1 (■), 3 (▲) and 5 (●) consecutive OD cycles, when a thermal treatment (72 ºC/ 15s) was applied to the OS between cycles.

Figure 3. Mean values of ºBrix (■) and ascorbic acid content (◆) of the osmotic solution as a function of the number of OD cycles.

Figure 4. Respiration rate, in terms of CO_2 generation, at different storage times (10 ºC), for fresh-cut (FC: ◆) and osmodehydrated grapefruit after 1 (ODG1: □), 3 (ODG3: △) and 5 (ODG5: ○) consecutive OD cycles.

Table 1

Table 1. Mean values and standard deviation of water content (x_w) (g water/100 g sample), ºBrix (g soluble solids/100 g sample) and water activity (a_w), at different storage times (10 ºC), for fresh-cut (FG) and osmodehydrated grapefruit after 1 (ODG1), 3 (ODG3) and 5 (ODG5) consecutive OD cycles.

Samples	t(days)	x_w	ºBrix	a_w [(1)]
FG	0	87.20 ± 0.06 (w)	12.27 ± 0.06 (w)	0.987
	3	86.44 ± 0.09 (x)	13.53 ± 0.06 (x)	0.990
	6	87.47 ± 0.02 (v)	12.0 ± 0.0 (v)	0.988
	8	86.20 ± 0.08 (y)	13.77 ± 0.06 (y)	0.990
	10	87.43 ± 0.10 (v)	12.07 ± 0.06 (v)	0.989
ODG1	0	(a) 73.0 ± 0.5 (y)	(a) 26.4 ± 0.0 (x)	(b) 0.978 (v)
	3	76.14 ± 0.06 (v)	23.43 ± 0.06 (w)	0.981 (w)(x)
	6	76.53 ± 0.07 (v)	22.93 ± 0.06 (v)	0.980 (v)(w)
	8	73.89 ± 0.15 (x)	25.37 ± 0.06 (y)	0.979 (v)(w)
	10	75.02 ± 0.12 (w)	24.37 ± 0.06 (y)	0.983 (x)
ODG3	0	(c) 76.2 ± 0.1 (y)	(c) 23.17 ± 0.06 (w)	(c) 0.980 (x)
	3	75.8 ± 0.1 (x)	23.6 ± 0.0 (x)	0.972 (v)
	6	73.7 ± 0.3 (v)	25.47 ± 0.06 (z)	0.975 (v)(w)
	8	74.6 ± 0.1 (w)	24.6 ± 0.0 (y)	0.979 (w)(x)
	10	76.2 ± 0.1 (y)	23.07 ± 0.06 (v)	0.979 (w)(x)
ODG5	0	(b) 75.2 ± 0.4 (v)	(b) 24.47 ± 0.06 (z)	(a) 0.972 (v)
	4	75.1 ± 0.2 (v)	23.6 ± 0.0 (y)	0.978 (w)
	6	76.5 ± 0.0 (w)	23.0 ± 0.0 (x)	0.979 (w)
	8	77.3 ± 0.1 (x)	22.2 ± 0.2 (v)	0.980 (w)
	10	76.5 ± 0.3 (w)	22.73 ± 0.06 (w)	0.980 (w)

(1) Standard deviations were, in all cases, lower than the accuracy of the equipment (0.003).
(a)(b)(c) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor cycle of dehydration.
(v)(w)(x)(y)(z) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor storage time.

Table 2

Table 2. Mean values and standard deviation of pH and citric acid (CA) content, expressed as mg CA/ 100 mg sample and as mg CA/ 100 mg of the corresponding fresh grapefruit (FG), at different storage times (10 ºC), for fresh-cut (FG) and osmodehydrated grapefruit after 1 (ODG1), 3 (ODG3) and 5 (ODG5) consecutive OD cycles.

Samples	t(days)	mgCA/100gDG	mgCA/100gFG	$\Delta CA_{treatment}$ (*)	$\Delta CA_{storage(*)}$	pH
FG	0	-	$1.346 \pm 0.006^{(w)}$	-	-	$3.223 \pm 0.015^{(v)}$
	3	-	$1.13 \pm 0.02^{(x)}$	-	-15.90	$3.247 \pm 0.006^{(w)}$
	6	-	$0.92 \pm 0.03^{(y)}$	-	-32.02	$3.337 \pm 0.010^{(x)}$
	8	-	$0.874 \pm 0.016^{(z)}$	-	-35.05	$3.497 \pm 0.006^{(y)}$
	10	-	$0.94 \pm 0.03^{(y)}$	-	-29.95	$3.503 \pm 0.006^{(y)}$
ODG1	0	$^{(a)}1.01 \pm 0.02$	$^{(a)}0.89 \pm 0.02^{(y)}$	-33.95	-	$^{(a)}3.137 \pm 0.006^{(v)}$
	3	1.12 ± 0.15	$0.99 \pm 0.13^{(x)(y)}$	-	-	$3.17 \pm 0.04^{(w)}$
	6	1.365 ± 0.015	$1.203 \pm 0.013^{(w)}$	-	-	$3.20 \pm 0.01^{(w)}$
	8	1.209 ± 0.015	$1.065 \pm 0.014^{(x)}$	-	-	$3.273 \pm 0.006^{(x)}$
	10	0.802 ± 0.014	$0.707 \pm 0.012^{(z)}$	-	-	$3.357 \pm 0.006^{(y)}$
ODG3	0	$^{(b)}1.100 \pm 0.001$	$^{(b)}0.985 \pm 0.007^{(w)}$	-26.84	-	$^{(c)}2.77 \pm 0.06^{(v)}$
	3	1.10 ± 0.01	$0.9817 \pm 0.0099^{(w)}$	-	-0.32	$3.27 \pm 0.06^{(x)}$
	6	1.001 ± 0.009	$0.897 \pm 0.008^{(y)}$	-	-8.95	$3.33 \pm 0.06^{(x)(w)}$
	8	1.06 ± 0.01	$0.946 \pm 0.013^{(x)}$	-	-3.94	$3.37 \pm 0.06^{(y)}$
	10	1.08 ± 0.02	$0.97 \pm 0.02^{(w)}$	-	-1.73	$2.90 \pm 0.00^{(w)}$
ODG5	0	$^{(c)}1.14 \pm 0.04$	$^{(c)}1.03 \pm 0.04^{(w)}$	-23.46	-	$^{(b)}2.83 \pm 0.06^{(v)}$
	3	0.905 ± 0.007	$0.816 \pm 0.007^{(z)}$	-	-20.77	$3.33 \pm 0.06^{(x)}$
	6	1.037 ± 0.008	$0.935 \pm 0.007^{(y)}$	-	-9.20	$3.4 \pm 0.2^{(x)}$
	8	1.080 ± 0.005	$0.974 \pm 0.005^{(x)}$	-	-5.42	$2.9 \pm 0.0^{(v)}$
	10	1.04 ± 0.03	$0.94 \pm 0.02^{(x)(y)}$	-	-8.60	$3.1 \pm 0.1^{(w)}$

(*) Percentage of CA loss due to the treatment or storage (mg CA lost/ 100mg initial CA).
(a)(b)(c) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor cycle of dehydration.
(w)(x)(y)(z) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor storage time.

Table 3

Table 3. Mean values and standard deviation of ascorbic acid (AA) content, expressed as mg AA/ 100 mg sample and as mg AA/ 100 mg of the corresponding fresh grapefruit (FG), at different storage times (10 ºC), for fresh-cut and osmodehydrated grapefruit after 1 (FG1 and ODG1, respectively), 3 (FG3 and ODG3, respectively) and 5 (FG5 and ODG5, respectively) consecutive OD cycles.

Samples	t(days)	mgAA/100gDG	mgAA/100gFG	$\Delta AA_{treatment}$ (*)	$\Delta AA_{storage}$ (*)
FG	0	-	$30.1 \pm 1.7^{(v)}$	-	-
	1	-	$14.9 \pm 0.7^{(x)}$	-	-50.67
	3	-	$15 \pm 0^{(w)(x)}$	-	-48.00
	6	-	$16.5 \pm 0.7^{(w)}$	-	-45.33
FG1	0	-	31 ± 5	-	-
ODG1	0	$^{(b)}27.2 \pm 1.3$	$23.52 \pm 1.15^{(v)}$	-23.59	-
	3	20.5 ± 1.7	$17.8 \pm 1.5^{(x)}$	-	-24.44
	6	21.4 ± 1.2	$18.51 \pm 1.05^{(x)}$	-	-21.29
	8	24.31 ± 0.98	$21 \pm 0.9^{(w)}$	-	-10.55
	10	14.8 ± 0.8	$12.8 \pm 0.7^{(y)}$	-	-45.48
	12	13.0 ± 0.8	$11.2 \pm 0.7^{(y)}$	-	-52.29
FG3	0	-	42 ± 3	-	-
ODG3	0	$^{(a)}34.40 \pm 1.08$	$26.2 \pm 0.8^{(v)}$	-36.94	-
	3	23.41 ± 0.99	$17.9 \pm 0.8^{(x)}$	-	-31.94
	6	23.1 ± 1.2	$17.6 \pm 0.9^{(x)}$	-	-32.77
	8	27.12 ± 0.98	$20.7 \pm 0.8^{(w)}$	-	-21.17
	10	20.4 ± 0.8	$15.6 \pm 0.6^{(y)}$	-	-40.71
	12	16.7 ± 1.4	$12.73 \pm 1.06^{(z)}$	-	-51.49
FG5	0	-	38 ± 4	-	-
ODG5	0	$^{(b)}25.79 \pm 1.09$	$21.8 \pm 0.9^{(v)(w)}$	-43.20	-
	3	24.1 ± 0.0	$20.4 \pm 0.0^{(w)(x)}$	-	-6.47
	6	266 ± 0.0	$22.5 \pm 0.0^{(v)}$	-	3.04
	8	22.4 ± 1.4	$18.9 \pm 1.2^{(x)}$	-	-13.23
	10	-	-	-	-
	12	17.2 ± 0.8	$14.6 \pm 0.7^{(y)}$	-	-33.11

(*) Percentage of AA loss due to the treatment or storage (mg AA lost/ 100mg initial AA).
(a)(b) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor cycle of dehydration.
(v)(w)(x)(y)(z) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor storage time.

Table 4

Table 4. Mean values and standard deviation of cation (Ca^{+2}, Na^{+}, Mg^{+2}, K^{+}) concentration, expressed as mg cation/ 100 mg sample and as mg cation/ 100 mg of the corresponding fresh grapefruit (FG), for fresh-cut (FG) and osmodehydrated grapefruit after 1 (ODG1), 3 (ODG3) and 5 (ODG5) consecutive OD cycles.

Samples	Ca^{+2}	Na^{+}	Mg^{+2}	K^{+}
FG[a]	17.6 ± 0.8(x)	1.5 ± 0.7(x)	15.5 ± 0.8(x)	158 ± 3(x)
ODG1[a]	11.6 ± 0.4(y)	1.3 ± 1.9(x)	8.7 ± 0.2(y)	85.5 ± 0.3(y)
ODG1[b]	13.1 ± 0.5	1.9 ± 1.6	9.8 ± 0.3	97.0 ± 0.3
Loss[c]	-34.19	-10.74	-44.10	-46.01
ODG3[a]	11.1 ± 0.2(y)	1.39 ± 0.02(x)	10.7 ± 0.9(y)	85 ± 6(y)
ODG3[b]	12.4 ± 0.2	1.55 ± 0.02	12.0 ± 1.1	95 ± 7
Loss[c]	-37.03	-6.62	-31.01	-46.54
ODG5[a]	12.8 ± 0.9(y)	1.457 ± 0.014(x)	10.3 ± 0.9(y)	93 ± 5(y)
ODG5[b]	14.2 ± 1.1	1.62 ± 0.02	11.36 ± 1.08	104 ± 6
Loss[c]	-27.36	-1.77	-33.94	-40.98

[(a)] mg mineral/100g fresh sample.
[(b)] mg mineral/100g osmodehydrated sample.
[(c)] mg mineral loss/100g mineral fresh sample.
(x)(y) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor cycle of dehydration.

Table 5

Table 5. Mean values and standard deviation of cation (Ca^{+2}, Na^{+}, Mg^{+2}, K^{+}) concentration in the osmotic solution (mg cation/100g OS) after 1, 3 and 5 consecutive OD cycles.

Cycles	Ca^{+2}	Na^{+}	K^{+}	Mg^{+2}
1	$3.9 \pm 1.2^{(a)}$	$1.1 \pm 0.4^{(a)}$	$3.87 \pm 1.05^{(a)}$	$0.13 \pm 0.12^{(a)}$
3	$3.1 \pm 0.3^{(a)}$	$0.9 \pm 0.2^{(a)}$	$8.59 \pm 0.05^{(a)(b)}$	$1.0 \pm 0.3^{(b)}$
5	$5 \pm 2^{(a)}$	$1.08 \pm 0.07^{(a)}$	$12.9 \pm 0.6^{(b)}$	$1.2 \pm 0.2^{(b)}$

(a)(b) The same letter indicates homogeneous group established by the ANOVA ($P<0.05$) with the factor cycle of dehydration.

Figure 1

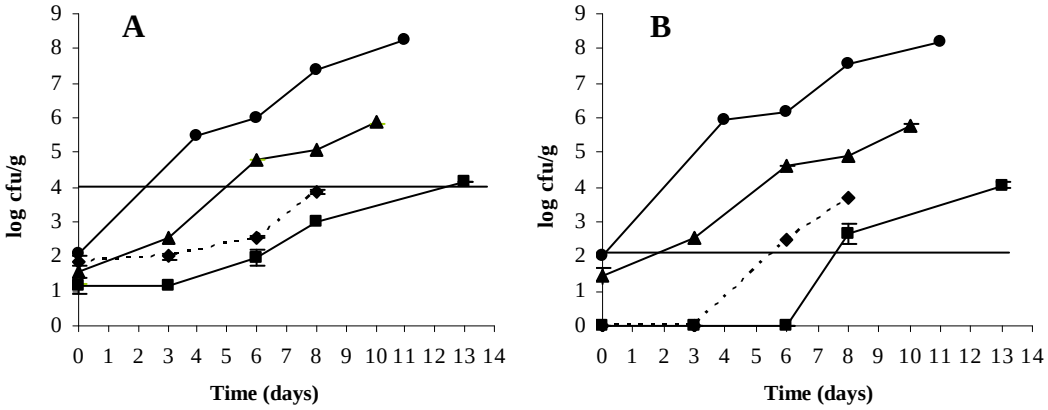


Figure 2

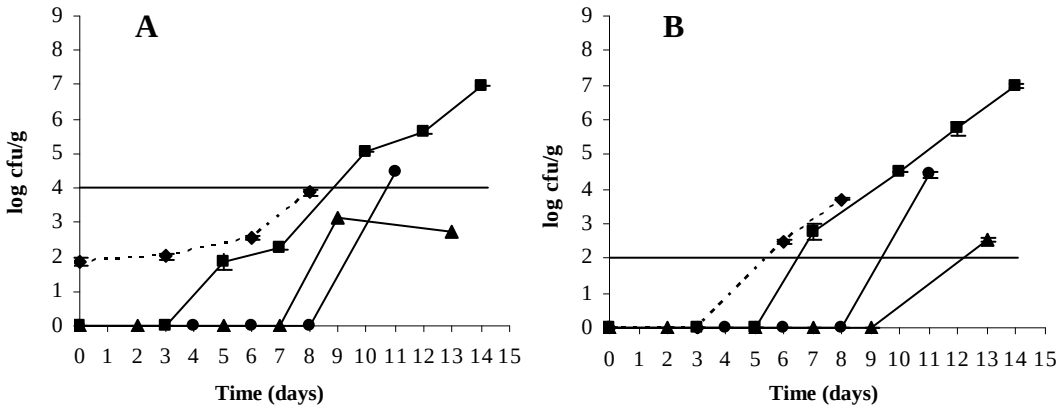
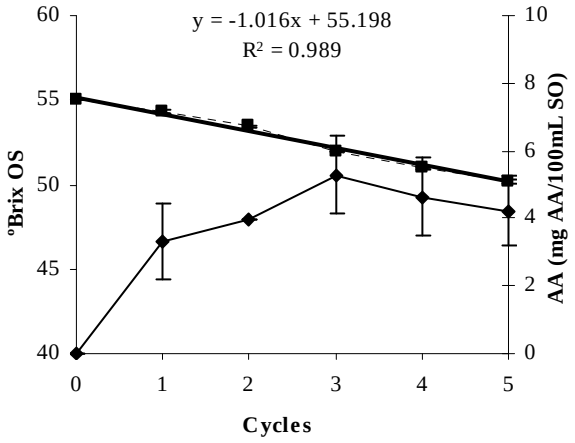


Figure 3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R² = 0.989
°Brix OS
AA (mg AA/100mL SO)
Cycles
60
55
50
45
40
10
8
6
4
2
0
0
1
2
3
4
5

Figure 4