The Effect of Selected Fruit (Apple, Bitter Orange and Grape) Juice Concentrates Used as Osmotic Agents on the Osmotic-Dehydration Kinetics and Physico-Chemical Properties of Pomegranate Seeds

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Abstract: This work aimed to study the osmotic-dehydration kinetics of pomegranate seeds (PSs) (Punica granatum L.) using three fruit (apple, bitter orange and grape) juice concentrates as osmotic solutions (OSs) (ratio (1/4) m/v, 55° Brix, at 50 °C). The osmodehydration time, pomegranate-seed water loss, solute gain and weight reduction were determined during the osmodehydration process. The equilibrium time periods of osmodehydration were 20, 60, 80 and 60 min for sucrose solution, and bitter-orange, apple and grape juice concentrates, respectively. The physico-chemical, biological and textural properties of PSs were significantly affected by the changes in OS. At the end of osmodehydration, the dry-matter and the Brix values were higher in all fruit-based OSs compared with sucrose OS. The color of PSs became darker in apple and grape OSs and brighter in bitter-orange OS compared with sucrose OS. On the other hand, all osmodehydrated PSs showed important antioxidant activity, with seeds osmodehydrated in grape-juice OS attaining an IC50 of 49.25 meq GA/100 g DM. All observed changes were attributed to the difference in the composition of the different OS matrices. Moreover, the hedonic analysis showed that consumers preferred osmodehydrated PSs obtained using bitter-orange juice as OS.

Keywords: pomegranate seeds; concentrated fruit juices; osmotic dehydration; physico-chemical properties

1. Introduction

Osmotic dehydration (OD) is a preservation technique that improves the quality of a product with the lowest energy consumption [1]. OD is defined as the partial removal of water from food. This is a process that occurs when a food is immersed in a hypertonic solution, and as a result, due to the osmotic pressure between the solution and the product, the phenomenon of osmosis takes place [2]. This technique is chosen to extend the shelf life of one of the widely consumed fruits to maintain good health, pomegranate. The pomegranate tree is a small tree, mostly cultivated in warm temperate zones and particularly in the Mediterranean basin. It can reach 6 m in height depending on the variety. Its bright-red flowers measure between 2.5 and 3 cm in diameter, appear in July and give fruits, pomegranates. This fruit is one of the first domesticated edible fruit crops along with fig, date palm, grape and olive. It was the symbol of health and fertility thanks to its important physico-chemical composition and biological activity. Pomegranate contains...
bioactive molecules, namely, phenolics, anthocyanins and others, that possess antimicrobial, antioxidant and antimutagenic activities, which reduce the risk of chronic illnesses [3].

Most scientists have used solutions of sucrose as osmotic solution. Indeed, Nono, et al. [4], Nono, et al. [5], Amami [1], Kowalska, et al. [6] and Bchir, et al. [7] have achieved the osmotic dehydration of papaya, mango, banana, carrots, pomegranate and yacon in sucrose solutions. However, the excess of sucrose in human food causes adverse health effects and promotes the development of several diseases, such as diabetes and obesity. Fruits containing bioactive molecules are characterized by significant antioxidant activity. Polyphenols are molecules well known for their activities of neutralizing free radicals and protection against oxidative stress. In addition, they can provide benefits to human health and prevent cardiovascular and carcinogenic diseases [8]. Several studies have established correlations between polyphenol content and antioxidant activity [9]. Therefore, scientific studies have focused on finding solutions to substitute the use of sucrose. One of the alternatives is the use of fruit juice concentrates as osmotic solution to prolong the storage life and to improve the nutritional, biological and sensory properties of fruits at the same time. For instance, Bchir, et al. [10] used concentrated date juice for OD; Sharif, et al. [11] performed OD with juice concentrate; and Samborska, et al. [12] used concentrated red-fruit juice. However, to our best knowledge, up-to-date information relating to this subject is still weakly developed in the literature.

Thus, the objectives of the present study were: (a) to study the kinetics of the OD of PSs in OS based on territory fruits (bitter orange, apple and grape); (b) to discern the effect of OD in different OSs on the physico-chemical, biological and textural properties of PSs as well as the viscosity of the used OS; and finally (c) to evaluate the acceptability of ODPSs by consumers through an hedonic test.

2. Materials and Methods
2.1. Raw Material
A batch of pomegranate fruits (Punica granatum L.) of the El Gabsi variety was used. Pomegranate fruits were harvested at the fully ripened stage from Gabes in autumn. Twenty kilograms of fruits of the same degree of ripeness, of the same weight and more or less of the same diameter was selected. Pomegranates were washed in tap water and then peeled by hand. Seeds were separated from the pericarp and the inter-carpellar membrane. The obtained pomegranate seeds (PSs) were stored in freezer bags and conditioned at −20 °C until analysis.

For the analyses, all used chemicals were of reagent grade and were purchased from Sigma-Aldrich, Co., St. Louis, MO, USA.

2.2. Preparation of Osmotic Solutions
A control (sucrose) and three test concentrated juices based on bitter orange, apple and grape were used. To all solutions, we added sucrose to reach a Brix level equal to 55° Brix [7] and kept them at 4 °C until use.

2.3. Osmodehydration Process
A ratio ((1/4) (m/v)) between fruit and osmotic solutions was applied for all the experiments [13]. Specifically, a quantity of 15 g of frozen PSs was immersed in a volume of 60 mL of osmotic solution, in a 100 mL bottle. The osmodehydration process was carried out in a water bath at a temperature of 50 °C with manual shaking every 3 min.

2.4. Mass Transfer Kinetics
At the end of each period (5, 10, 20, 40, 60, 80, 100 and 120 min), ODPSs were removed from the OS and were rinsed with distilled water, and the excess of the solution on the surface of the seeds was removed with absorbent paper. The mass and dry matter content were determined in order to be able to calculate the various mass transfer parameters, i.e.,
weight reduction (WR) (Equation (1)), gain in solute (GS) (Equation (2)) and water loss (WL) (Equation (3)), for which we applied the following formulas, respectively [14]:

\[
WL (\%) = \left(\frac{W_i - W_f}{W_i}\right) \times 100
\]

(1)

\[
GS (\%) = \left(\frac{W_{si} - W_{sf}}{W_i}\right) \times 100
\]

(2)

\[
WR (\%) = WL + GS
\]

(3)

where \(W_i\) is the initial weight of the sample (g), \(W_f\) is the final weight of the sample (g), \(W_{si}\) is the initial total solids content (g) and \(W_{sf}\) is the final total solids content (g).

2.5. Physico-Chemical Analyses

2.5.1. Dry Matter

A total of 5 g of sample was introduced into a previously tared crucible of mass and then placed in an oven at 105 °C until constant mass (AOAC (1995)).

2.5.2. pH and Water Activity

An 827 Lab Metrohm, Switzerland pH meter was used to measure the pH values of pomegranate seeds and osmotic solutions. The water activity was measured at 25 °C using a laboratory aw meter (swift aw; Novasina, Switzerland).

2.5.3. Brix Degree

According to AOAC (1995), a drop of the extract was placed on the glass of an ABBE refractometer (Kern Optics, Balingen, Germany). The Brix degree reflecting the total soluble solids value was read in the light using the eyepiece of the device.

2.5.4. Color

The color of PSs before and after OD was measured using a digital colorimeter (Konica Minolta chromameter CR-5). The determination of the color was based on the measurement of the CIE lab parameters of the color space, \(L^*, a^*\) and \(b^*\), defined by the International Commission on Illumination (CIE) [15]. \(L^*\) measures brightness ranging from 0 (black) to 100 (white), while \(a^*\) ranges from \(-100\) (green) to \(+100\) (red) and \(b^*\) ranges from \(-100\) (blue) to \(+100\) (yellow). The angles of hue (\(H^*\)) and of chroma or intensity (\(C^*\)) were calculated according to Equations (4) and (5):

\[
Chroma = \sqrt{(a^*)^2 + (b^*)^2}
\]

(4)

\[
hue = \arctan\left(\frac{b^*}{a^*}\right)
\]

(5)

2.5.5. Total Phenols

The preparation of the extracts from PSs before and after OD was carried out according to the method described by Kchaou, et al. [16]. A total of 1 g of sample was mixed with 20 mL of 70% acetone at room temperature (25 °C) under stirring (shaker) for 2 h. The mixture was centrifuged at 5000 rpm for 15 min, and the supernatant was collected. The process was repeated twice to improve the extraction. The supernatants were combined and used for the following experiments.

The determination of the content of phenols was carried out on the prepared extracts. The Folin–Ciocălcău method was used to determine the total phenolic content. The extract (500 µL) was transferred to a tube containing 2.5 mL of Folin–Ciocălcău solution diluted 10 times in water. A volume of 2 mL of 7.5% (w/v) sodium carbonate solution was added. The tubes were kept at room temperature for 15 min. Then, the absorbance was measured at 765 nm [17]. The total polyphenol content was expressed in milliequivalents of gallic
acid (GAE)/100 g. Gallic acid was used to obtain a standard curve with concentrations varying from 0 to 50 mg/L.

2.5.6. Antioxidant Activity

The free-radical-scavenging-activity method was used to assess the antioxidant activity of PSs and osmotic solutions. The test is based on the reduction of DPPH (2,2-diphenyl-1-picrylhydrazyl), which is a stable free radical possessing a dark-purple coloration that becomes yellow after reduction [18]. In the reaction tubes, 375 µL of ethanol and 125 µL of DPPH solution were added to 500 µL of crude extract and dilutions. For each concentration, a blank was prepared by mixing 500 µL of sample and 500 µL of ethanol without adding the DPPH solution. A control tube was also prepared by mixing 500 µL of the solvent (without sample), 375 µL of ethanol and 125 µL of the DPPH solution. After shaking, tubes were placed in the dark at 25 °C for 1 h. Then, the absorbance value was measured at 517 nm using a spectrophotometer (UV-VIS Spectrophotometer-UV mini 1240, SHIMANDZU), and the radical scavenging activity was determined using the following formula (Equation (6)):

\[
\text{Scavenging activity (\%)} = \left( \frac{A_{\text{Control}} + A_{\text{Sample}} - A_{\text{Blank}}}{A_{\text{Control}}} \right) \times 100
\]

where \( A_{\text{control}} \), \( A_{\text{sample}} \), and \( A_{\text{blanc}} \) are the absorbance values at 517 nm of control, sample and blank tubes, respectively.

2.6. Hardness Analysis

The hardness of PSs was determined with a TPA (texture profile analysis) test. A curve (force–time) of two successive compression cycles was obtained following the use of a texturometer (LLOYD instruments, Fareham, UK). For PSs, the TPA test was performed using a 2 mm diameter probe. The seeds were compressed by 50% with a speed of 5 mm/s over two compression cycles of 120 s. All operations were automatically controlled with “Exponent” texture software (Ametek NEXYGENPlus; LLOYD instruments, Fareham, UK).

2.7. Viscosity Analysis

The viscosity analyses were determined using a “Myr” type viscometer (Viscotech Hispania, Tarragona, Spain). The viscosity values of the osmotic solutions before and after OD were measured [13].

2.8. Hedonic Evaluation

A hedonic test was applied. The tasters were 60 people who could be qualified as “naive”. Participants had no specific information about the product. The ages of the tasters were between 15 and 65, of which 68% were women and 32% were men. The tasting session lasted between 20 and 30 min for each person in order to avoid the phenomena of desaturation. We did not set a time limit for the tasters, so they took all the time they needed to fully appreciate the products. To taste well and maintain a clear and constant sensitivity, participants drank water between tastings. Mineral water and goblets were available to them. Before all these steps, we started with an explanatory session concerning the test, so that we could help the tasters to evaluate the different samples and to appropriately fill out the form [19]. Participants responded to the test and questions on a scale of 1 to 7 by assigning a mark.

2.9. Statistical Analysis

The results obtained in the current study were analyzed using SPSS20 software (IBM, SPSS, statistics 20). Means with a significant difference were determined by applying Duncan’s multiple range test at a significance level (\( p < 0.05 \)).
3. Results and Discussion

3.1. Kinetics of Osmotic Dehydration

From the values of WL, GS and WR recorded during the OD process (Figure 1), we noticed that WL varied from 16.93 to 35.55%, 42.33 to 65.31%, 48.04 to 58.93% and from 52.81 to 74.73% for sucrose solution, bitter-orange juice, apple juice and grape juice, respectively. Likewise, GS ranged from 3.27 to 20.43%, 6.04 to 22.20%, 8.24 to 21.19% and from 9.13 to 24.00% for sucrose solution, bitter-orange juice, apple juice and grape juice, respectively. On the other hand, the values of WR varied remarkably during the 120 min of OD. They ranged from 6.25 to 19.35% for sucrose solution, from 36.29 to 43.34% in the case of bitter-orange juice, from 36.42 to 38.92% for apple juice and 43.67 to 50.73% in the case of grape juice. For sucrose solution, the changes were more significant during the first 20 min and then tended to stabilize. Indeed, WL did increase to have a value of ≈27% and then tended to be stable until the end of pretreatment. The same observation was described in the study by Bchir, Besbes, Attia and Blecker [7], in which the duration of 20 min was chosen as the optimal time for OD in sucrose solution. This choice was justified by the importance of the molecular mass of sucrose (342.29 g/mol) compared with Xylose (150.12 g/mol), Erythritol (122.11 g/mol) and Sorbitol (182.17 g/mol). This characteristic made the penetration of sucrose into plant cells difficult. A similar conclusion was obtained in [20], which showed that lemon by-products needed more time to reach the final Brix in sucrose solution than in glucose solution. The authors attributed this result to the fact that glucose had a lower molecular weight, allowing it to diffuse easily. In fact, the speed of the diffusion of sugar molecules in the fruit membrane cells is negatively correlated with their molecular size [21]. As a result, we could confirm that 20 min was the optimal time to have good OD performance in sucrose solution. For concentrated bitter-orange juice, the most significant changes occurred during the first 60 min of pretreatment. In this case, WL reached 60% and then tended to stabilize at a value of ≈65%. The same trend was observed for WR. Using the same conditions, GS significantly increased over the first 60 min, reaching 16%, and then tended to be stable. So, the optimal OD time using concentrated bitter-orange juice was 60 min. In comparison with sucrose solution, the increase in optimum time was due to the osmotic pressure (amount of sucrose added) and the initial composition of the juices (fiber, sugar, proteins, etc.). For concentrated apple juice, the most significant changes were observed after 80 min of OD. In fact, WL reached its maximum (59%) and then tended to stabilize. GS reached its maximum (21%) after 80 min and remained stable until the end of OD. So, we could conclude that the optimal OD time in concentrated apple juice was 80 min. However, the optimal time was longer than that of sucrose, which can be explained by the initial composition of the juice. In fact, apple initially contains fibers, which prevents the entry and the exit of water and solutes. For concentrated grape juice, the most significant changes were noticed during the first 60 min. Indeed, WL increased to 72%; then, it tended to be stable at a value of ≈75%. At the same time, GS increased and reached a value of 20% after 60 min and then tended to stabilize during the remainder time of the OD process.

The optimal OD time using concentrated grape juice was longer than that using sucrose. This can be explained by the initial composition of the juice. The optimal OD time using concentrated grape juice was 60 min. Of the four osmotic solutions, sucrose solution had the shortest optimal time. In fact, this solution only contains sucrose, whereas juices initially contain natural sugars; then, sucrose is added. Therefore, sucrose solution maintains a high osmotic pressure, which serves to remove water in a short time without gaining much solute [13].
Figure 1. Mass transfer kinetics of osmodehydration procedure of pomegranate seeds in osmotic solutions. (A) Sucrose solution; (B) bitter-orange juice; (C) apple juice; (D) grape juice.

3.2. Evolution of Physico-Chemical Properties of PSs after OD

The determination of the physico-chemical composition of PSs before and after OD showed that this pretreatment had a direct impact on the various parameters studied. Indeed, according to the results presented in Table 1, OD contributed to the increase in the dry-matter values from 0.19 (before OD) to 0.30 for sucrose solution, to 0.34 for bitter-orange juice and to 0.40 for apple and grape juices. Likewise, regarding the Brix degree, a significant increase was recorded after OD, reaching almost 40°B in the case of grape juice from 15°B before OD. These facts can be explained by the exchanges of material that PSs underwent in the various osmotic solutions used, in particular WL and GS. The same observation was described in the study by Sharif, Pirouzifard, Alizadeh and Esmaiili [11], who investigated the effect of OD on the production of candied apples. Sharif, Pirouzifard, Alizadeh and Esmaiili [11] showed that OD ensured changes in the physico-chemical characteristics of fruits, such as the reduction in humidity and the increase in total sugar content.

Regarding the color parameters, variations were noted in the level of brightness, L*, the red index, a*, and the yellow index, b*. The L* index slightly increased after OD in sucrose solution and bitter-orange juice, remained constant in grape juice and decreased in apple juice. a* reached its maximum after OD in bitter-orange juice, whereas b* decreased in the case of bitter-orange and grape juices and slightly increased in sucrose solution and apple juice. All the observed changes could be attributed to the difference in the initial coloration of the osmotic solutions and the migration of pigments between the seed pulp...
and the osmotic medium. It should be noted that bitter-orange juice mainly influenced the $a^*$ index because of its orange-yellow color. On the other hand, apple juice, having undergone enzymatic browning, had a greater effect on the brightness of seeds, making them darker after OD. The browning index is used to determine the formation of colored compounds following enzymatic and non-enzymatic browning reactions (Maillard reaction, caramelization). The values obtained in Table 1 showed an increase in the browning index before and after OD (sucrose solution and fruit juice). It reached a maximum of 0.09 in sucrrose solution, followed by bitter-orange juice with an index of 0.062 and apple juice having a browning index equal to 0.054; finally, in grape juice, it maintained a browning index equal to that of the seeds before OD. These observations could be explained by the difference in the polyphenol and flavonoid composition of the used osmotic solutions [22] and by the amount of sugar added to the osmotic solutions during concentration (55 °B). Indeed, these molecules, in particular polyphenols, have the capacity to protect seeds against enzymatic and non-enzymatic browning [23]. On the other hand, a large amount of added sugar promotes the initiation of browning reactions (Maillard reaction).

Table 1. Evolution of physico-chemical properties of pomegranate seeds after osmodehydration process.

| Parameter               | PSs Before OD | PSs After OD |
|-------------------------|---------------|--------------|
|                         | Succre Solution | Bitter-Orange Juice | Apple Juice | Grape Juice |
| Dry matter              | 0.19 ± 0.01 $^a$ | 0.32 ± 0.01 $^b$ | 0.34 ± 0.04 $^c$ | 0.40 ± 0.73 $^d$ | 0.40 ± 1.39 $^d$ |
| Brix                    | 15.16 ± 0.28 $^a$ | 29.05 ± 0.90 $^b$ | 33.16 ± 1.06 $^c$ | 36.23 ± 1.55 $^d$ | 39.13 ± 0.92 $^e$ |
| $a_w$                   | 0.948 ± 0.001 $^d$ | 0.911 ± 0.002 $^b$ | 0.906 ± 0.008 $^a$ | 0.932 ± 0.005 $^c$ | 0.956 ± 0.001 $^e$ |
| CIE lab parameters      |               |               |               |
|                         |               |               |               |
| $L^*$                   | 29.12 ± 0.58 $^b$ | 31.92 ± 0.91 $^d$ | 32.30 ± 1.19 $^e$ | 27.82 ± 0.79 $^a$ | 30.06 ± 0.10 $^c$ |
| $a^*$                   | 10.39 ± 0.79 $^c$ | 8.69 ± 0.42 $^a$ | 11.07 ± 1.86 $^d$ | 9.68 ± 0.17 $^b$ | 8.76 ± 0.14 $^a$ |
| $b^*$                   | 11.54 ± 0.21 $^c$ | 12.69 ± 0.24 $^d$ | 8.53 ± 0.48 $^a$ | 12.00 ± 0.14 $^d$ | 9.40 ± 0.34 $^b$ |
| $C^*$                   | 15.34 ± 0.18 $^c$ | 15.77 ± 0.14 $^c$ | 13.99 ± 1.78 $^e$ | 15.05 ± 0.27 $^c$ | 12.85 ± 0.34 $^a$ |
| Browning index          | 0.050 ± 0.002 $^a$ | 0.090 ± 0.007 $^d$ | 0.062 ± 0.004 $^c$ | 0.054 ± 0.002 $^b$ | 0.049 ± 0.009 $^a$ |
| Total phenols (meqGA/100 g) | 384.22 ± 25.65 $^a$ | 341.31 ± 21.13 $^c$ | 376.49 ± 17.61 $^d$ | 256.03 ± 17.69 $^a$ | 314.10 ± 21.48 $^b$ |
| IC$_{50}$ (mg/mL)       | 57.43 ± 1.23 $^d$ | 59.55 ± 0.45 $^e$ | 52.78 ± 0.98 $^c$ | 49.80 ± 1.09 $^b$ | 49.25 ± 0.78 $^a$ |
| Hardness (N)            | 0.66 ± 0.09 $^e$ | 0.48 ± 0.08 $^b$ | 0.36 ± 0.06 $^a$ | 0.55 ± 0.12 $^d$ | 0.53 ± 0.09 $^c$ |

All the data are expressed as means ± SD and are the mean of three replicates. Values with different letters in the same line differed significantly ($p < 0.05$). PS, pomegranate seed; OD, osmodehydration.

The result of the study of bioactive molecules and antioxidant activity of PSs is shown in Table 1. From the obtained values, it was found that PSs are rich in polyphenols. Indeed, pomegranates are classified among the fruits that are the richest in polyphenols compared with other fruits. After OD, a slight loss of polyphenols was recorded, going from 384.22 meq GA/100 g DM before OD to 341.31, 376.49, 256.03 and 314.10 meq GA/100 g DM for the sucrose solution, and bitter-orange, apple and grape juices, respectively. The difference was mainly due to the initial composition of the juices and to the exchanges that took place during the OD phenomenon.

The results of the DPPH neutralization test in terms of 50% radical inhibition concentration (IC$_{50}$) are shown in Table 1. Prior to OD, the IC$_{50}$ value was 57 meq GA/100 g DM, while after OD, the value slightly varied depending on the osmotic solution used. It increased in the case of OD in sucrrose solution and decreased after OD using concentrated fruit juices as immersion solutions. Noting that a low IC$_{50}$ corresponds to better antioxidant activity, the PSs dehydrated in grape juice had the greatest antioxidant activity (IC$_{50}$ equal to 49.25 meq GA/100 g DM). Indeed, the initial composition of grape of polyphenols and flavonoids allows it to prevent oxidation, hence having significant antioxidant activity.
3.3. Hardness of PSs  
To better understand the effect of OD on the structure of PSs, a TPA (texture profile analysis) test was performed. The texture analysis primarily measured the mechanical properties of PSs. Through the TPA test, we were able to characterize the modifications caused by dehydration on the structural parameters of PSs. Among the parameters studied during the analysis of the texture profile, hardness, expressed in (N), was the resistance of the sample to deformation caused by compressive forces.

As shown in Table 1, before OD, PSs were characterized by a hardness of 0.66 N, while after OD, the recorded values decreased and reached 0.48, 0.36, 0.53, and 0.55 for sucrose solution, bitter-orange juice, apple juice, and grape juice, respectively. This modification was the result of the weakening of the cell tissue of the seed pulp of grenade. In fact, during OD, the difference in solute concentration between the osmotic medium and PSs creates an osmotic force that promotes the transfer of solutes through the cell wall. In addition, one must take into consideration the effect of ice crystals formed during freezing, which can damage the cell membrane during storage, inducing the loss of binding capacity between the walls. Bchir, Besbes, Karoui, Attia, Paquot and Blecker [13] also showed that, under the effect of freezing, PSs lost their firmness and reduced their resistance.

3.4. Evolution of Physico-Chemical Properties of Osmotic Solutions after OD

Table 2, relating to the physico-chemical characterization of the osmotic solutions before and after OD, shows that sucrose solution as well as the various juices used underwent changes in their parameters. The dry-matter values indicated that after OD, a decrease of 15 to 25% was recorded for all solutions used. Along with dry matter, the Brix degree dropped by 10% after OD. These findings were a consequence of the material exchanges described above in the characterization of large seeds before and after OD. In fact, the reductions in dry matter and Brix degree proved the passage of a fraction of solutes from the osmotic medium to PSs. The decrease in the pH of osmotic solutions could be attributed to the diffusion of certain organic solutes and acids from PSs to the aqueous solution. Many authors found similar trends during the osmotic dehydration of various fruits [20,24–26]. After OD, each osmotic solution also underwent changes in the color parameter levels. The saturation or colorimetric purity, C*, and the hue, h°, changed differently depending on the solution used for OD. These changes were attributed to the initial color of the juices and the exchange of pigments between immersion solutions and PSs.

| Parameters | Sucrose Solution | Bitter-Orange Juice | Apple Juice | Grape Juice |
|------------|------------------|---------------------|-------------|-------------|
| **Dry matter (%)** | Before OD | After OD | Before OD | After OD | Before OD | After OD | Before OD | After OD |
| 0.55 ± 0.09 b | 0.45 ± 0.07 a | 0.51 ± 0.08 b | 0.43 ± 0.06 a | 0.57 ± 0.10 b | 0.45 ± 0.04 a | 0.53 ± 0.09 b | 0.46 ± 0.03 a |
| **Brix** | 55.00 ± 0.00 b | 50.69 ± 0.41 a | 55.00 ± 0.03 b | 49.66 ± 0.76 a | 55.00 ± 0.00 b | 49.23 ± 0.05 a | 55.00 ± 0.00 b | 49.63 ± 0.11 a |
| **pH** | 6.41 ± 0.23 b | 5.56 ± 0.21 a | 2.67 ± 0.01 a | 2.71 ± 0.02 a | 4.42 ± 0.13 a | 4.37 ± 0.05 a | 3.90 ± 0.04 b | 3.81 ± 0.02 a |
| **CIE lab parameters** | | | | | | | | |
| L° | 13.84 ± 1.40 a | 14.55 ± 1.45 b | 22.02 ± 0.88 b | 22.25 ± 0.62 a | 21.27 ± 0.13 a | 21.74 ± 0.07 a | 29.30 ± 1.01 a | 28.90 ± 0.97 a |
| a° | 0.15 ± 0.01 b | -0.06 ± 0.04 b | -1.07 ± 0.27 a | 1.77 ± 0.77 b | 5.54 ± 0.34 a | 6.13 ± 0.06 b | 4.22 ± 0.24 a | 4.47 ± 0.28 b |
| b° | 0.12 ± 0.01 b | 0.44 ± 0.02 b | 9.36 ± 0.62 b | 8.39 ± 0.52 a | 11.32 ± 0.24 a | 12.11 ± 0.39 b | 12.29 ± 1.22 b | 11.67 ± 1.25 b |
| C* | 0.39 ± 0.01 a | 0.44 ± 0.04 b | 9.65 ± 0.97 b | 8.59 ± 0.65 a | 12.69 ± 0.04 a | 13.10 ± 0.24 b | 12.99 ± 0.65 a | 12.50 ± 0.68 a |
| h° | 266.26 ± 2.48 b | 149.41 ± 6.60 b | 96.47 ± 2.54 b | 78.29 ± 4.53 b | 62.47 ± 2.89 a | 67.93 ± 4.97 b | 71.09 ± 0.26 a | 69.08 ± 0.38 a |
| **Total phenols (meqGA/100 g)** | 0.88 ± 0.07 a | 6.19 ± 0.56 b | 31.03 ± 1.32 a | 60.17 ± 2.33 b | 14.31 ± 0.54 a | 32.06 ± 0.92 b | 54.13 ± 2.12 a | 79.82 ± 3.16 b |
| **IC50 (mg/mL)** | ND | 2.41 ± 0.45 b | 0.26 ± 0.09 a | 0.50 ± 0.07 b | 0.18 ± 0.01 a | 0.40 ± 0.01 b | 0.28 ± 0.01 a | 0.35 ± 0.01 a |
| **Viscosity (mPa·s)** | 72 ± 2 | 57 ± 4 | 80 ± 5 | 59 ± 3 | 80 ± 2 | 59 ± 1 | 80 ± 2 | 62 ± 3 |

All the data are expressed as means ± SD and are the mean of three replicates. Values with different letters in the same line differ significantly (p < 0.05). OD, osm dehydration.

From Table 2, a significant increase in polyphenols in osmotic solutions after OD was observed, which may be related to the above-discussed decrease in polyphenol content in
On the other hand, the IC$_{50}$ of the different osmotic solutions remarkably increased, which is the cause of the exchange of phenolic compounds between seeds and the juices used. Thus, the penetration of these molecules contributed to the improvement of the antioxidant activity of PSs, which was reflected in the decrease in the IC$_{50}$ and the lowering of the activity of osmotic solutions after OD.

### 3.5. Apparent Viscosity of Apple, Bitter-Orange and Grape Juices

When preparing osmotic solutions, the viscosity of the juices increased with the addition of sucrose (55 °B). This was due to the high molecular weight of sucrose (342.29 g/mol) [20]. From Table 2, it can be seen that after OD, the viscosity of the osmotic solutions decreased for all juices. By observing the differences between the viscosity values before OD and after OD, we could notice that the decrease in viscosity was greater the longer the duration of OD was. In fact, for sucrose solution, which showed the shortest OD duration (20 min), the viscosity only decreased by 15 mPa·s, whereas for bitter-orange and grape juices, which had the same optimum OD time (60 min), the viscosity decreased by almost 20 mPa·s. In the case of apple juice, which showed the longest optimum OD time (80 min), the viscosity decreased by 25 mPa·s. This fact was due to the migration of water from seeds to the osmotic solution and the levels of carbohydrates and proteins present in the juices [13].

### 3.6. Hedonic Evaluation of PSs and OSs

#### 3.6.1. Pomegranate Seeds

Figure 2 presents the hedonic profiles of the four samples of dehydrated and dried PSs obtained on the basis of the means of the scores assigned by the panel of tasters for the six criteria of each sample. It was noticed that the four samples of dehydrated PSs had quite different profiles. By observing the taste axis, we found that the tasters appreciated the PSs dehydrated in concentrated bitter-orange juice, whose average scores were the highest (6.3), followed by the seeds of pomegranate dehydrated in concentrated apple juice, with a score of 5.2. Although PSs dehydrated in grape juice had a low taste rating (5), they were liked more than the control. This difference can be explained by the influence of the taste of the fruit juices used with PSs. This is due to the harmony of the two tastes (bitter orange and pomegranate) and to the variation in the ages of the participating population. For “visual aspect” and “color”, PSs dehydrated in bitter-orange juice ranked the highest, with hedonic scores of 6.3 and 6.2, respectively, for the two descriptors. The other samples had almost the same visual appearance as well as the same color. This difference can be explained by the effect of the color of bitter-orange juice on PSs. For “The smell”, the hedonic test of the tasters showed that PSs dehydrated in concentrated bitter-orange juice were the most preferred by the participants, with a score of 6.3, followed by seeds dehydrated in sucrose solution and in grape juice (5.5). The smell of seeds dehydrated in apple juice was the least appreciated by tasters, with a score of 5. So, after tasting the different samples, taking into consideration the hedonic score of each descriptor, we could say that PSs dehydrated in bitter-orange juice were the most appreciated by the tasters.

#### 3.6.2. Osmotic Solutions

Figure 2 presents the hedonic profiles of the four osmotic solutions obtained on the basis of the averages of the scores assigned by the tasters for the six criteria of each sample. From the results, we noticed that the osmotic solutions had quite different hedonic profiles. Looking at the taste axis, we found that tasters liked bitter-orange juice, with the highest score of 6.1, followed by apple juice and grape juice, with a hedonic score reaching 5.2. Sucrose solution was the least acceptable among all osmotic solutions. This difference can be explained by the combination of tastes (pomegranate and fruit juice). For the appreciation of color, the means of the scores of the samples were different, with the best (6.1) being that of bitter-orange juice, followed by the samples of grape juice, apple juice and sucrose solution in order, with mean scores of 5.3, 5 and 4.7, respectively. These scores showed that the tasters appreciated the color of bitter-orange juice more than that of the
control. For the descriptor “odor”, the hedonic test of the tasters showed that bitter-orange juice was the most assimilable by the participants, with a score of 6, followed by grape juice and apple juice, which had scores of 5.1 and 4.8, respectively. The smell of sucrose solution was the least accepted by tasters, with a score of 4.5. So, after tasting the different samples, taking into consideration the hedonic score of each descriptor, we could say that bitter-orange juice was the most appreciated by tasters.

Figure 2. Hedonic evaluation of pomegranate seeds and osmotic solutions after the osmodehydration process. (A) pomegranate seeds; (B) osmotic solutions.

4. Conclusions

In this work, concentrated fruit juices (bitter orange, apple and grape) were shown to be suitable for the OD of PSs. The choice of such OSs could be a promising solution to reduce the use of sucrose, which is highly used in the food industry. Based on the studied mass parameters, WL, GS and WR, the equilibrium time periods were 20, 60, 80
and 60 min for sucrose solution, and bitter-orange, apple and grape juices, respectively. Changes in the physico-chemical parameters of PSs and OSs were highlighted after OD, especially in terms of the Brix and dry-matter values, which increased due to the passage of solutes from the osmotic medium to PSs. The texture of PSs, the viscosity of OSs and the color parameters of both showed the important effect of the use of concentrated fruit juices in the OD process. In fact, the hardness of PSs decreased due to the weakening of the cell tissue of the seed pulp of grenade. After OD, the viscosity of the osmotic solutions decreased for all juices, and we noticed that the decrease in viscosity was greater the longer the duration of the OD was. Indeed, the color of PSs became darker with apple and grape OSs and brighter with bitter-orange OS than with sucrose OS. The observed differences were attributed to the difference in the composition of the OSs used. In fact, with sucrose OS, dry-matter diffusion concerned only sucrose, whereas with fruit OSs, there was the diffusion of sugars (sucrose and inverted sugars, essentially fructose), enzymes such as phenol oxidase, phenolic compounds, vitamins, pigments, etc. Finally, the hedonic test showed that ODPSs in bitter-orange juice were highly appreciated by consumers. In light of these results, we succeeded in extending the consumption period of a territory fruit (Punica granatum L.) and in giving a new taste to PSs using concentrated fruit juices as OSs. The studied process promotes the use of fruit OSs to reduce sucrose in osmodehydrated pomegranate seeds while improving their nutritional and sensory properties.

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