Impact of freeze-drying shelf temperature on the bioactive compounds, physical properties and sensory evaluation of a product based on orange juice

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Summary Freeze-drying is a good alternative means of obtaining fruit products, with a significant amount of thermo-labile bioactive compounds. Despite the excellent benefits, this method provides the dehydrated product, and its main drawback is its high cost due to the duration of the process. Heat may be applied to the shelf so as to shorten the process time, as long as this does not affect the quality of the product. In this study, the impact of the freeze-drying shelf temperature, 30 and 50 °C, on the bioactive compounds of the product obtained from an orange juice formulated with gum Arabic and bamboo fibre has been considered, as has the temperature’s effect on the porosity, colour and mechanical properties of the cake and on the flowability and the rehydration capacity of the powder, together with the sensory evaluation of the rehydrated product. The results obtained point to 50 °C as being the recommended temperature for the freeze-drying of this product. This temperature shortens the process time by 64%, promotes the vitamin C content with no effect on the total phenols and carotenoids, leads to the cakes having better mechanical properties and does not affect the flowability and the rehydration behaviour of the powdered product.

Keywords Antioxidant capacity, crispness, powder flowability, total carotenoids, total phenols, vitamin C.

Introduction Fruits are foods of great importance in terms of human nutrition. In this group, citrus fruits are of particular importance, characterised by their distinctive flavour and as a good source of carbohydrates, dietary fibre, vitamins, minerals and biologically active phytochemicals, such as carotenoids and flavonoids, which provide antioxidant benefits (Hoffmann et al., 2003). The ingestion of these compounds is closely related to the prevention of chronic diseases, some types of cancer, cardiovascular problems and brain disorders, such as Alzheimer’s, so their consumption has a great and favourable impact on health (Boeing et al., 2012). However, the intake of these products is greatly limited by their availability and short shelf life. Dehydrated fruit, included fruit powder, may be a good alternative to lengthen the product’s shelf life, because its low water activity will prevent chemical and enzymatic reactions that will deteriorate it. Consumption in dehydrated form or the subsequent rehydration of the powder would permit fruit intake in a comfortable, safe and healthy way.

For many years, hot air has been the most commonly used food dehydration method. However, its main problem is related to the loss of thermolabile compounds and sensory properties as a consequence of the heat treatment. Freeze-drying is a method characterised by the fact that it offers high-quality products, based on sublimating a frozen sample at low pressures, without exposing the product to high temperatures (Que et al., 2008). Of the benefits of freeze-drying, the preservation of the taste, flavour and thermo-sensitive compounds with biological activity, together with the great rehydration capacity of the obtained products (Serna Cock et al., 2015), are of special importance in the case of fruit.

Despite the excellent benefits, this method provides the dehydrated product, and its main drawback is its high cost due to the long duration of the process, which
has limited its application being extended to the food industry. In recent years, nevertheless, the growing consumer interest in healthy nutrition has led to an emerging interest in studying the optimisation of the process variables trying to find alternatives to the reduction in time and, in turn, the energy cost, without sacrificing the quality of the product (Babić et al., 2009; Ceballos et al., 2012; Salazar et al., 2018). Of the process variables, the shelf temperature seems to be the one with the greatest impact on process time, although it may also be the one that most compromises the properties of the product (Shishegharha et al., 2002).

Based on the above, the objective of this study was to evaluate the effect of the freeze-drying shelf temperature on the properties of the cake and powder obtained from an orange juice formulated with gum Arabic and bamboo fibre. The analyses carried out were the retention of bioactive compounds, the porosity, colour and mechanical properties of the cake and powder, together with the flow properties and rehydration behaviour of the powder, included the sensory properties of the rehydrated formulated juice.

Materials and methods

Raw material and formulation

Orange (Citrus x sinensis var. Navelina), always acquired in the same chain of supermarkets in Valencia, was used for the study. To obtain the juice, a domestic juicer was used (Braun MPZ 6, Spain). To the juice (12 °Brix and 88 g of water/100 g juice), gum Arabic (GA, Scharlau, SL, Spain) and bamboo fibre (BF, Vitacel, Rosenberg, Germany) were added in a ratio of 100:5:1, respectively (Aguilera et al., 2017), and homogenised with a processor (Thermomix TM 21, Vorwerk, Spain) at 2500 rpm for 40 s and subsequently at 9200 rpm for 40 s. The formulated juice (FJ) presented 83 g of water/100 g and 16 °Brix.

Freeze-drying

The orange juice was placed on aluminium plates, of 25 cm in diameter and 1 cm thick, and frozen (Liebherr Medline, LCT 2325, Austria) at −45 °C for 48 h. The drying step was carried out using a Telstar Lyo Quest 55 (Spain) freeze-dryer operating at −50 °C in the condenser, P = 0.05 mbar and changing the shelf temperatures between 30 and 50 °C for 48 or 18 h. The selection of these times was based on previous experiments as to reach samples with around 0.04 g water/g sample (data not shown).

Analytical determinations

The formulated orange juice and all the freeze-dried samples were analysed as to the water content, vitamin C content (VC), antioxidant activity (AOA), total phenolics (TP) and carotenoids (TC). The freeze-dried cakes obtained from the orange juice were coded as C (30 °C) and C(50 °C), depending on the freeze-drying shelf temperature, and the mechanical properties, colour and bulk density of the freeze-dried cakes were also analysed. In addition, the freeze-dried cakes were crushed and sieved to particle size <800 μm as proposed by Uscanga et al. (2020). The powders obtained from C(30 °C) and C(50 °C) were called P(30 °C) and P(50 °C), respectively, and were characterised as to the particle size distribution, angle of repose, colour, porosity and Hausner and Carr indexes. The powder rehydration behaviour and the sensory properties of the rehydrated product were also determined.

Water content

The water content of the powders was analysed by coulometric Karl-Fischer titration (C10S Compact, Mettler Toledo, USA).

Vitamin C, total phenolics, total carotenoids and antioxidant activity

In all the cases, the methodology proposed by Uscanga et al. (2020) was applied.

To determine the VC, dehydroascorbic acid (DHAA) was reduced to ascorbic acid (AA) using DL-dithiothreitol (Sánchez-Mata et al., 2000; Xu et al., 2008). The analyses were performed by high-performance liquid chromatography (HPLC) (Jasco, Cremella, Italy).

The determination of TP was carried out spectrophotometrically (absorbance at 765 nm) following the Folin–Ciocalteu method (Benzie & Strain, 1999). For TC, absorbance at 446 nm was measured (AOAC, 1996) and β-carotene was considered the standard.

The AOA of the samples was evaluated using the DPPH and FRAP methods. The absorbance at 515 and 593 nm, respectively, was measured in an UV-visible spectrophotometer (V-1200, VWR, International Euro-Lab S.L., Spain). The results of DPPH were expressed according to eqn 1. In both cases, the results were referred to mmol of trolox equivalent (TE).

\[
\text{%DPPH} = \frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \times 100, \tag{1}
\]

where \(A_{\text{control}}\) = absorbance of the control (absorbance of the sample at time 0); \(A_{\text{sample}}\) = absorbance of the sample when the reaction has stabilised.

Mechanical properties

To evaluate the mechanical properties, a texturometer (TA-TXT2i, Stable Micro Systems, Ltd, Godalming, UK) was used. In the case of the freeze-dried cakes, a puncture-compression test was carried out according to Uscanga et al. (2020), and the powdered products
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were analysed following the methodology proposed by Telis & Martinez-Navarrete (2010). In both cases, a cylindrical punch of 10 mm in diameter was used to move forward in the sample. The force-distance curve was recorded, and the maximum force or the maximum force related to the sample mass was the parameter selected to characterise the mechanical properties of cakes and powders, respectively.

**Colour measurement**

A portable spectrophotometer (CM2600D, Konica Minolta Sesing, INC., Japan) was used, using the 5-mm-diameter measurement window, taking illuminant D65 and observer 10° as system reference. CIEL*a*b coordinates were obtained, and the angle hue (h°) and chroma (C°) were calculated by using eqns 2 and 3, respectively. The colour of the freeze-dried cakes was measured on samples previously cut into a circular shape by using a punch of 2.30 cm in diameter. To avoid the influence that the different degree of compaction may have on the colour of the powdered products, this was measured after being subjected to the mechanical compression test (2.3.3). The procedure proposed by Telis & Martinez-Navarrete (2010) was followed to this end. Briefly, after the compression test, the capsule with the sample was quickly inverted and forced against the optical glass placed on the spectrophotometer window. Measurements were taken in triplicate for each sample.

\[
h° = \arctg \left( \frac{b°}{a°} \right), \quad (2)
\]

\[
C° = \left( a°^2 + b°^2 \right)^{0.5}, \quad (3)
\]

**Cake apparent density**

To calculate the apparent density (eqn 4), a 2.30 cm in diameter punch was used to obtain, in triplicate, cylinders of the cakes which were precisely weighed (Mettler Toledo, XS204DR, Switzerland) and measured in diameter and height with a Vernier calliper.

\[
\rho_a = \frac{m}{V}, \quad (4)
\]

where \( \rho_a \) = apparent density (g cm\(^{-3}\)); \( m \) = mass (g); \( V \) = volume = \( \pi r^2 h \), \( r \) and \( h \) being the exact radius and height of the weighted piece (cm\(^3\)).

**Powder particle size distribution**

The sieving method was used to determine both, the relative sample mass of each size (eqn 5) and the mean particle size (eqn 6) (Uscanga et al., 2020).

\[
F_i = \frac{m_i \times 100}{m}, \quad (5)
\]

\[
\text{MPS} = \frac{\sum_i (m_i h_i)}{m}, \quad (6)
\]

\( F_i \) = relative frequency retained in each sieve (%), \( m_i \) = powder mass retained in each sieve (g); \( m \) = total sieved powder mass (g); \( \text{MPS} \) = mean particle size (mm); \( \Theta_i \) = each sieve mesh (mm).

**Powder flowability**

The angle of repose (\( \alpha^° \)), the porosity (\( \varepsilon \)) and the Hausner index (\( I_H \)) of the powders were determined. The methodology proposed by Uscanga et al. (2020) was followed for \( \alpha^° \) and \( \varepsilon \), and eqn 7 was used to obtain \( I_H \). The Carr index (\( I_C \)), related to the ability of powdered substances to be compacted, was also determined (eqn 8).

\[
I_H = \frac{\rho_t}{\rho_b}, \quad (7)
\]

\[
I_C = \frac{100 \rho_t - h}{\rho_i}, \quad (8)
\]

where \( I_H \) = Hausner index; \( I_C \) = Carr index (%); \( \rho_t \) = tapped density (g cm\(^{-3}\)); \( \rho_b \) = bulk density (g cm\(^{-3}\)) calculated before the sample is tapped.

**Powder rehydration behaviour**

On the one hand, the wettability was determined. On the other hand, \( P(30^°C) \) and \( P(50^°C) \) were rehydrated to obtain \( R(30^°C) \) and \( R(50^°C) \) samples, respectively, and the flow behaviour of the rehydrated powders was analysed. The procedure described by Uscanga et al. (2020) was followed in all the cases. The colour of the rehydrated juice was also measured (buckets 4 cm high and 1 cm thick). The physical colour was measured from the reflection spectra, which was obtained on a black background. The same spectrophotometer described in section 2.3.4 was used, and the \( h° \) and \( C° \) were also calculated (Eqs. 2 and 3).

**Sensory analysis**

A triangular discriminatory test was performed with a panel of 60 tasters in order to determine whether there are significant differences between the two samples of orange juice, \( R(30^°C) \) and \( R(50^°C) \). One trial was prepared per taster which had a duplicate and a different sample. Each sample was coded with three random digits and was presented an equal number of times in each of the possible positions: BAA, AAB, ABA, ABB, BBA, BAB in random order following a Williams design. Each taster evaluated the samples of the trial and marked the sample that they considered different.

**Statistical analysis**

Several analyses of variance (ANOVA) were carried out to evaluate the effect of temperature on the
properties of freeze-dried products. The method used to discriminate between means was Fisher’s least significant difference (LSD) procedure. When \( P < 0.05 \), significant differences were assumed between the samples.

**Results and Discussion**

The freeze-dried samples did not show a significant difference \( (P > 0.05) \) as regards the water content, the obtained values being \( 0.039 \pm 0.005 \) and \( 0.041 \pm 0.005 \text{ g water/g sample} \) when the drying was carried out at 30 and 50 °C, respectively. In both experiments, a freeze-dried product with approximately the expected 0.04 g water/g sample was obtained.

As reported in Table 1, both TP and TC decreased due to as a result of freeze-drying, with no observed effect of the drying temperature. The most sensitive appears to be carotenoids. Ice crystals and low pressure during freeze-drying cause the orange cell structure disruption and promote the action of polyphenol oxidase (Shofian et al., 2011; Gomes et al., 2018). As regards carotenoids, their high sensitivity to oxygen leads to their oxidation during the process even when the oxygen presence is minimum as when working at low pressure (Silva-Espinoza et al., 2020). In the case of VC (Table 1), an increase was observed after freeze-drying: the higher the drying temperature, the bigger the change \( (P < 0.05) \). This may be due both to the effect of temperature on the inactivation of the ascorbate peroxidase enzyme, which uses vitamin C as a cofactor in its metabolic processes (Cuastumal-Canaclan et al., 2016), and to the fact that the freeze-drying process was shorter at a higher temperature: 18 h at 50 °C vs. 48 h at 30 °C (Silva-Espinoza et al., 2020). These changes caused a decrease in the antioxidant activity \( (P < 0.05) \), without there being any observed effect of temperature in the case of FRAP \( (P > 0.05) \). Different studies have shown a close correlation of VC and TP but not of TC with the AOA determined by the FRAP assay on orange juice (Gardner et al., 2000), TC being more related to scavenging capacity of the DPPH-free radical (Liu et al., 2008). Due to the greater content of VC and TP than TC in orange samples and the lesser loss of the former as related to TC, the FRAP behaviour is highlighted.

**Characterisation of the cakes**

The cakes did not show any significant differences \( (P > 0.05) \) as regards their apparent density (Table 2), which would indicate no influence of the drying temperature in terms of the porosity achieved by the sample.

As to colour (Table 2), and as expected for this product, all the samples were found in the red-yellow quadrant of the colour wheel. The samples showed no significant differences \( (P > 0.05) \) in terms of \( L^* \), linked to their luminosity. Nevertheless, the cakes obtained at a higher temperature showed a slightly yellowish and less pure colour, related to the greater \( h^* \) and lower \( C^* \) values, respectively. The calculated colour difference between the two samples (eqn 9) was in the order of 16.66 units, indicating that they are differences perceptible to the human eye (Bodart et al., 2008).

\[
\Delta E = \sqrt{\Delta L^*^2 + \Delta a^*^2 + \Delta b^*^2}. \quad (9)
\]

An example of the graphs obtained from the mechanical test for each freeze-dried cake at both temperatures is shown in Figure 1. This profile gives us the evolution of the force as the punch of the equipment penetrates the sample. At the beginning of the test, the continuous increase in the force is caused by

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**Table 1** Mean values ± standard deviation of the total phenols (TP, mg GAE/100 g db), total carotenoids (TC, mg β-C /100 g db), Vitamin C (VC, mg/100 db) and antioxidant activity (mmolTE/100 g db) measured by the DPPH (2,2-diphenyl-1-picrylhydrazyl) and FRAP (ferric reducing/antioxidant power) methods for the different samples

| Property   | C(FJ) | P(30 °C) | P(50 °C) |
|------------|------|---------|---------|
| DPPH       | 1.05 ± 0.05* | 1.09 ± 0.08* | 0.93 ± 0.03* |
| FRAP       | 3.0 ± 0.9* | 2.3 ± 0.5* | 2.2 ± 0.1* |
| TP         | 705 ± 54* | 420 ± 5* | 378 ± 37* |
| TC         | 54.1 ± 6.1* | 8.1 ± 0.8* | 8.4 ± 0.4* |
| VC         | 236 ± 5* | 252 ± 8* | 276 ± 19* |

\*FJ, P(30 °C) and P(50 °C): formulated orange juice and the corresponding powders obtained by freeze-drying at 30 and 50 °C, respectively. db, dry basis; GAE, gallic acid equivalent; TE, Trolox equivalent. The same superscript letter (a-c) in rows indicates the homogeneous groups established by the ANOVA \( (P < 0.05) \).

**Table 2** Mean values (±) standard deviation of the different properties analysed for the cakes obtained from the orange juice freeze-dried at 30 °C (C 30 °C) and 50 °C (C 50 °C)

| Property* | C(30 °C) | C(50 °C) |
|-----------|---------|---------|
| \( \rho_a \) (g/mL) | 0.13 ± 0.01a | 0.13 ± 0.02a |
| \( L^* \) | 60.63 ± 3* | 60.93 ± 5* |
| \( a^* \) | 12 ± 2* | 8 ± 2* |
| \( b^* \) | 41 ± 6* | 34 ± 4* |
| \( C^* \) | 43 ± 6* | 35 ± 4* |
| \( h^* \) | 74.3 ± 2* | 77 ± 2* |
| \( F_{max} \) (N) | 11 ± 3a | 16 ± 5a |

\*\( \rho_a \): apparent density; \( L^* \), \( C^* \) and \( h^* \): luminosity, chroma and hue angle; \( a^* \), \( b^* \): colour coordinates; \( F_{max} \): maximum force recorded in the mechanical test. The same superscript letter (a-b) in rows indicates the homogeneous groups established by the ANOVA \( (P < 0.05) \).
the cutting effect caused by the perimeter of the punch, until the sample is completely broken off. The multiple force peaks observed correspond to small fractures in the samples related to their crispiness. The evolution of the curve thereafter corresponds to the progressive compaction of the cake that remains below the surface of the punch (10 mm in diameter). In both samples, the increase in $F$ was of the same order, until reaching the maximum $F$ value related to the significant break in the sample. This maximum force was higher ($P < 0.05$) in the samples freeze-dried at 50 °C (Table 2), indicating that these samples are less fragile, showing a higher mechanical resistance to breakage while handling (Silva-Espinoza et al., 2020).

Characterisation of the powdered products

To check whether the freeze-drying temperature affects the mechanical strength of the freeze-dried product, the particle size distribution of the powder obtained after the cake being crushed was characterised. In both cases, the mode of this distribution was found in the range of 0.3–0.5 mm and the median, corresponding to the particle size that leaves 50% of the powder above and below it, was in the range of 0.15–0.20 mm (Fig. 2).

As can be seen in Figure 2, the biggest differences between the two samples were observed for particle sizes of less than 0.2 mm, so that P(30 °C) had more particles of between 0.10 and 0.20 mm and fewer of <0.1 mm, linked to an increased resistance to crushing in this case. However, when the mean particle size was calculated (Table 3), the samples were observed to exhibit no significant differences ($P > 0.05$). The greater number of the smallest particles observed in P (50 °C) could be related to a greater ease of attrition during the sieving applied for this analysis when the samples were dried at a higher temperature. In this sense, the less fragile cakes obtained at 50 °C would lead to harder particles after being crushed, which generate more fine powder during processing.

The angle of repose is a measurement of the powder flowability that is related to the cohesion between molecules, the chemical composition of the material, the size and shape of particles and the water content. As a reference, the angle of repose of some flours is in the same order as our results (Table 3), with no significant differences observed between the samples ($P > 0.05$). According to the values obtained, the Spanish Royal

![Figure 1](image1.png)

**Figure 1** Example of force-distance curves obtained from the puncture-compression test performed on the freeze-dried sample at 30 °C (30 °C) and 50 °C (50 °C)

![Figure 2](image2.png)

**Figure 2** Particle size distribution based on the percentage of weight of the powder retained in each sieve relative to the total sieved sample (Weight, %). Particle size intervals: the largest mesh of the sieves that the sample did not pass through—the smallest mesh of the sieves that the sample passed through. Mean values and standard deviation. P(30 °C) and P(50 °C): powdered products coming from the cakes obtained after the freeze-drying of the orange juice at 30 and 50 °C, respectively

| Table 3 Mean values (±) standard deviation of the different properties analysed for the powdered products obtained from the orange juice freeze-dried at 30 °C (P 30 °C) and 50 °C (P 50 °C) |
| Property* | P(30 °C) | P(50 °C) |
|------------|----------|----------|
| MPS (mm) | $0.196 \pm 0.009^a$ | $0.189 \pm 0.015^b$ |
| Angle of repose | $30.9 \pm 0.3^a$ | $29 \pm 2^a$ |
| Porosity (%) | $77.5 \pm 0.2^a$ | $74 \pm 1^a$ |
| $I_h$ (%) | $1.25 \pm 0.02^a$ | $1.24 \pm 0.02^a$ |
| $I_c$ (%) | $20 \pm 3^a$ | $19 \pm 3^a$ |
| $F_{max}$ (N/g) | $404 \pm 140^a$ | $415 \pm 110^a$ |
| L* | $71.9 \pm 0.2^a$ | $71.8 \pm 0.3^a$ |
| a* | $14 \pm 0.2^a$ | $13 \pm 0.2^a$ |
| b* | $54.0 \pm 0.2^a$ | $54.1 \pm 0.3^a$ |
| C* | $55.7 \pm 0.3^a$ | $55.9 \pm 0.4^a$ |
| h* | $75.1 \pm 0.9^a$ | $75.6 \pm 0.6^a$ |

*MPS, mean particle size; $F_{max}$, maximum force recorded in the mechanical test; $I_h$, Hausner index; $I_c$, Carr index; L*, C* and h*, luminosity, chroma and hue angle; a* and b*, chromatic colour coordinates; the same superscript letter (a–b) in rows indicates the homogeneous groups established by the ANOVA ($P < 0.05$).
Pharmacopoeia allows us to classify our products with an excellent flowability (AEMPS, 2015), which is a favourable characteristic for a powdered product. The Hausner and Carr indexes, also related with the powder flowability, pointed to no significant differences between the samples \((P > 0.05)\). The drying temperature affected neither the colour nor the mechanical properties of the powdered products (Table 3). In the case of the powdered samples, always a continuous increase in the force was observed throughout the mechanical test.

The calculated real density of these samples was 1.4246 g cm\(^{-3}\). The powder bulk and tapped densities were 0.315 ± 0.003 and 0.394 ± 0.004 g cm\(^{-3}\) for P (30 °C) and 0.367 ± 0.016 and 0.455 ± 0.014 g cm\(^{-3}\) for P(50 °C), respectively. From these data, the samples showed significant differences as regards porosity \((P < 0.05)\), this being lower in sample P(50 °C) related to the fact that it is less fragile and when crushed, this sample generates more regular and rounded particles that would allow for a better packaging and, therefore, a lower volume of air. In addition, the greater presence in this sample of fine particles (Fig. 2), capable of occupying the gaps between the larger particles, contributes in the same way.

### Behaviour of the powder against rehydration

In addition to particle size, the wetting time is mainly associated with the composition of the sample, especially its water content, so that the wetting time lengthens when the water content decreases (Schubert, 1993). In Table 4, it can be observed that there were no significant differences between the two samples \((P > 0.05)\), which was to be expected since there were no compositional differences between the samples.

The flow curves of the rehydrated powdered products were obtained, following the procedure described in section 2.3.8. The data obtained were fitted to the Ostwald-de Waele model (Table 4), where we can confirm the pseudoplastic nature of both products \((n < 1)\). From the values of the model parameters \(K\) and \(n\), the viscosity at a shear rate of 100 s\(^{-1}\) was calculated (Table 4). There were no significant differences between the samples in any of the rheological parameters evaluated \((P > 0.05)\).

Rehydrated samples exhibited significant differences \((P < 0.05)\) in terms of luminosity and hue angle (Table 4), so that R(50 °C) was a more luminous and yellowish product with respect to R(30 °C). However, their overall calculated colour difference (eqn 9) gave a value of 1.2, which indicates that the colour difference was not perceptible to the human eye (Bodart et al., 2008).

### Sensory analysis

To analyse the data of the triangular test, the correct answers were considered and the result was compared with the data corresponding to the statistical table of triangular tests to evaluate whether there is a significant difference between the samples at 95% probability.

From the total of sixty triangles evaluated, twenty correct answers were obtained. As the tabulated value (27) is greater than the experimental (20), it can be concluded that no significant differences were found between the two samples of rehydrated powder, R (30 °C) and R(50 °C), since the tabulated value represents the minimum number of correct answers required to find significant differences between two samples.

### Conclusion

As regards the bioactive compounds, freeze-drying supposes an increase in VC and a decrease in both TP and TC. To apply 50 °C along drying does not affects TP nor TC and promotes an increase in VC content as compared to 30 °C. Despite the freeze-drying temperature did not affect the porosity, colour and crispiness of the cakes, the sample obtained at 50 °C was more resistant to breakage due to the application of small forces. This facilitates the handling of this product. On the other hand, when subjected to larger shear forces such as those needed to obtain a powder, more fine particles were obtained when the drying was done at a higher temperature, which decreases the interparticle porosity and allows for a better packaging. The average particle size and colour of the powder were also not affected by the drying temperature. Moreover,
neither did it affect the wetting time during rehydration nor the rheology and sensory perception of the rehydrated product. In view of the above, taking into account that freeze-drying at 50 °C means that the process time is 64% shorter compared with the process at 30 °C, with the consequent energy savings, and also the fact that there is a limited impact on the properties of the product, which are even somewhat better when the product is dried at 50 °C, this temperature could be recommended as a means of obtaining freeze-dried orange juice to be consumed either as a snack or as a powder to be rehydrated.

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Conflicts of interest

The authors have declared no conflicts of interest for this article.

Author Contributions

Mariana Uscanga: Conceptualization (equal); data curation (equal); formal analysis (equal); investigation (equal); methodology (equal); writing – original draft (supporting). Ana Salvador: Data curation (equal); formal analysis (equal); methodology (equal); writing – original draft (equal). María del Mar Camacho: Conceptualization (equal); data curation (equal); formal analysis (equal); investigation (equal); methodology (equal); supervision (equal). Nuria Martínez-Navarrete: Conceptualization (equal); data curation (equal); formal analysis (equal); funding acquisition (equal); investigation (equal); methodology (equal); resources (equal); supervision (equal); visualization (equal); writing – original draft (equal); writing – review and editing (equal).

Ethical Guidelines

Ethics approval was not required for this research.

Peer Review

The peer review history for this article is available at https://publons.com/publon/10.1111/jifs.15086.

Data Availability Statement

Research data are not share.

REFERENCES

This reference contains data of orange juice related to the different contribution of vitamin C phenolics and carotenoids to its antioxidants capacity.

In this reference a freeze-dried orange puree is studied and similar results to those found in this study are discussed.

This reference also provides details of the experimental methodology used in the current paper.

AEMPS. (2015). Agencia Española de Medicamentos y Productos Sanitarios. España: Real Farmacopea Española. Agudelo, C., Igual, M., Camacho, M. & Martínez-Navarrete, N. (2017). Effect of process technology on the nutritional, functional, and physical quality of grapefruit powder. Food Science and Technology International, 23, 61–74.

AOAC. (1996). Official Methods of Analysis of AOAC International, 16th ed. Maryland: Gaithersburg.

Babić, J., Cantalejo, M. & Arroqui, C. (2009). The effects of freeze-drying process parameters on Broiler chicken breast meat. LWT - Food Science and Technology, 42, 1325–1334.

Benzie, I.F.F. & Strain, J.J. (1999). Ferric reducing/antioxidant power assay: direct measure of total antioxidant activity of biological fluids and modified version for simultaneous measurement of total. Methods in Enzymology, 299, 15–27.

Bodart, M., Peñaranda, R.D., Denayer, A. & Flamant, G. (2008). Photometry and colorimetry characterisation of materials in daylighting evaluation tools. Building and Environment, 43, 2046–2058.

Boeing, H., Bechthold, A., Bub, A. et al. (2012). Critical review: vegetables and fruit in the prevention of chronic diseases. European Journal of Nutrition, 51, 637–663.

Ceballos, A., Giraldo, G. & Orrego, C. (2012). Effect of freezing rate on quality parameters of freeze dried sour soup pulp. Journal Food Engineering, 111, 360–365.

Cuautstumal-Canacuan, H.G., Valencia-Murillo, B.L. & Ordoñez-Santos, L.E. (2016). Efectos de los tratamientos térmicos en la concentración de vitamina C y color superficial en tres frutas tropicales. Revista Lasallista de Investigación, 13, 85–93.

Gardner, P.T., White, T.A.C., McPhail, D.B. & Duthie, G.G. (2000). The relative contributions of vitamin C, carotenoids and phenolics to the antioxidant potential of fruit juices. Food Chemistry, 68, 471–474.

Gomes, W.F., França, F.R.M., Denadai, M. et al. (2018). Effect of freeze- and spray-drying on physico-chemical characteristics, phenolic compounds and antioxidant activity of papaya pulp. Journal of Food Science and Technology, 55, 2095–2102.

Hoffmann, K., Boeing, H., Volatiere, J.-L. & Becker, W. (2003). Evaluating the potential health gain of the World Health Organization’s recommendation concerning vegetable and fruit consumption. Public Health Nutrition, 6, 765–772.

Liu, D., Shi, J., Colina Ibarra, A., Kakuda, Y. & Jun Xue, S. (2008). The scavenging capacity and synergistic effects of lycopene, vitamin E, vitamin C, and β-carotene mixtures on the DPPH free radical. LWT - Food Science and Technology, 41, 1344–1349.

Que, F., Mao, L., Fang, X. & Wu, T. (2008). Comparison of hot air-drying and freeze-drying on the physicochemical properties and antioxidant activities of pumpkin (Cucurbita moschata Duch.) slices. International Journal of Food Science & Technology, 43, 1195–1201.

Salazar, N.A., Alvarez, C. & Orrego, C.E. (2018). Optimization of freezing parameters for freeze-drying mango (Mangifera indica L.) slices. Drying Technology, 36, 192–204.

Sánchez-Mata, M.C., Cámara-Hurtado, M., Díez-Marqués, C. & Torijia-Isasa, M.E. (2000). Comparison of high-performance liquid chromatography and spectrofluorimetry for vitamin C analysis of green beans (Phaseolus vulgaris L.). European Food Research and Technology, 210, 220–225.
Schubert, H. (1993). Instantization of powdered food products. *International Chemical Engineering, 33*, 28–45.

Serna Cock, L., Vargas Muñoz, D.P. & Ayala Aponte, A. (2015). Structural, physical, functional and nutraceutical changes of freeze-dried fruit. *African Journal of Biotechnology, 14*, 442–450.

Shishegarha, F., Makhlof, J. & Ratti, C. (2002). Freeze-drying characteristics of strawberries. *Drying Technology, 20*, 131–145.

Shoifian, N.M., Hamid, A.A., Osman, A. et al. (2011). Effect of freeze-drying on the antioxidant compounds and antioxidant activity of selected tropical fruits. *International Journal of Molecular Science, 12*, 4678–4692.

Silva-Espinoza, M.A., Ayed, C., Foster, T., Camacho, M.M. & Martínez-Navarrete, N. (2020). The impact of freeze-drying conditions on the physico-chemical properties and bioactive compounds of a freeze-dried orange puree. *Foods, 9*(1), 32.

Swarbrick, J. (1997). *Encyclopedia of pharmaceutical Technology*. Nueva York: Marcel Dekker INC.

Telis, V. & Martínez-Navarrete, N. (2010). Application of compression test in analysis of mechanical and color changes in grapefruit juice powder as related to glass transition and water activity. *LWT-Food Science and Technology, 744–751*.

Uscanga, M.A., Camacho, M., Salgado, M.A. & Martinez-Navarrete, N. (2020). Influence of an orange product composition on the characteristics of the obtained freeze-dried cake and powder as related to their consumption pattern. *Foods and Bioprocess Technology, 13*, 1368–1379.

Xu, G., Liu, D., Chen, J., Ye, X., Ma, Y. & Shi, J. (2008). Juice components and antioxidant capacity of citrus varieties cultivated in China. *Food chemistry, 106*, 545–551.