Dried orange juice waste as a source of bioactive compounds.

Galindo, R.G.[1], Chis, M.S. [2], Martínez-Navarrete, N. [1], Camacho, M.M. [1*]

[1]*Food Technology Department, Food Investigation and Innovation Group, Universitat Politècnica de València, Camino de Vera s/n, 46022, Valencia, Spain.*

[2]*Department of Food Engineering, University of Agriculture Science and Medicine Veterinary, Cluj-Napoca, Romania*

*Corresponding author e-mail: mdmcamvi@tal.upv.es

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Abstract

Purpose: The waste generated in the process of obtaining orange juice may be used as a natural source of bioactive compounds, thus contributing to the profitability and sustainability of the process. To offer it as a dried matter would contribute to the integral valorisation of the juice waste and also may expand its field of application.

Design/methodology/approach: To find out whether the juice waste matrix protects the bioactive compounds, this study compares the behaviour of the extracts of these compounds against drying with that resulting from drying the juice waste for further extraction. Dehydration was carried out at 25 or 50 ºC and gum Arabic and bamboo fibre were used as stabilising biopolymers. Vitamin C (L-ascorbic and L-dehydroascorbic acids) and hesperidin were analysed before and after the drying.

Findings: The results suggest that to dry the juice waste gives a higher yield of bioactive compounds, which are also more stable, than when the extract is dried. Furthermore, both the higher temperature and the presence of the biopolymers favour the extraction of both vitamin C and hesperidin. In this way, all the waste from the orange juice processing industries is converted into a high-value product to be used for cosmeceutical or nutraceutical purposes and also as an ingredient for human food.

Originality: Both for their potential as a natural source of vitamin C and flavonoids and for the profitability and sustainability of the processes, to dehydrate the waste of the orange juice industry, results in a value-added product to be used as a natural source of these compounds, fulfilling part of

the circular economy. This is particularly true for the innovative use of waste from these industries as raw material for sectors such as cosmetics, pharmaceuticals and animal or human food itself.

Key words: ascorbic acid; dehydroascorbic acid; hesperidin; orange juice waste; vitamin C extract; gum Arabic; bamboo fibre; drying temperature.

1. Introduction

Citrus (*Citrus* spp.) is one of the most important fruit crops in the world, with a great increase in the last year till a production of 98 million tons in 2020/21 (USDA, 2021). Spain is the largest producer of citrus fruits in the European Union (EU) and the fifth in the world, with a production of 6,9 million tons in the same period, which represents 61% of the whole production of the EU. Of all this citrus production in the world, approximately 51% corresponds to oranges with China, Brazil and EU being the main producers. Of the total 6.5 million tons of oranges produced in the EU in 2020/21, 3.6 million tons were produced in Spain (USDA, 2021). Approximately 20% of this production was destined for juice extraction purposes, leaving approximately half of the fruit weight, in the form of albedo, flavedo, essential oils, juice, segments and membranes, as waste. The elimination of all this rind is a major problem for many companies, as it causes high environmental pollution due to its high water content and microbial load (O'Shea et al. 2012).

The sustainable conversion of agri-food waste into a value-added product is called valorisation technology and supports the concepts of "zero waste" and "waste to wealth" (Hussain et al., 2020). As it has been demonstrated that agri-food waste can be a good source of bioactive compounds and can serve as an ingredient to improve the nutritional content and potential functional value of different products, to propose novel or emerging methods of valorisation of the orange juice waste, is a challenging field of study that would contribute to ensure environmental protection, promote economic development and, at the same time, contribute to the sustainability of the planet. Emerging valorisation

methods are proposed against conventional or traditional waste management. While the latter leads to its underutilisation as biocompost or biofuel, the former suppose a more efficient approach with the objective of reducing the amount of waste finally disposed of by recovering valuable components and reincorporating them into the food chain or for other end uses as pharmaceuticals, cosmetics, etc. (Esparza et al., 2020).

As described by Marin *et al*. (2007), the major components of dry citrus peel residues are dietary fibre (cellulose up to 35%, pectin up to 23%, hemicellulose up to 11% and lignin up to 8%) and sugars (up to 23%, including glucose, fructose, sucrose and xylose). Currently, to a greater or lesser extent, the citrus processing industry has implemented processes for the recovery of cells or pulp from the waste, which they reincorporate into the juice. Research is also continuing into the recovery of waste for the extraction of health-promoting dietary fibres (Hussain *et al*., 2020). Nevertheless, citrus peel also contains other components which, although minor, appear to contribute to a healthy diet. This is the case of flavonoids and vitamins (mainly vitamin C), among other phytochemicals (Garau *et al.* 2007). These minor bioactive compounds, with a potential role as natural antioxidants, could be extracted to be used as food additives or included in pharmaceutical formulations or food matrices to produce nutraceuticals and functional foods, respectively (Jiménez-Moreno *et al.* 2020). Moreover, both dietary fibre and phenolic compounds can stimulate the growth of beneficial microorganisms, especially lactic acid bacteria and other probiotics. Thus, bioactive compounds present in fruit waste emerge as potential prebiotic ingredients (Albuquerque *et al.*, 2021).

Vitamin C in its reduced (L-ascorbic acid, AA) and oxidized (dehydroascorbic acid, DHAA) forms prevents oxidative damage to lipids, DNA, and proteins, which are associated with the development of chronic degenerative diseases, such as cardiovascular disease, cancer, and cataracts (Escobedo-Avellaneda *et al*. 2014). Both AA and DHAA have biological functions (Oro and Donnamaria, 2006) and, although most physiological effects are attributed to AA, in recent years DHAA has been receiving greater attention from the scientific community because it can be transported from the blood to the cells by glucose and so cross the blood-brain barrier, improving cerebral ischemia, something that AA cannot do (Koepsell, 2020; Malik *et al*. 2021). On the other hand, although AA is the most reduced of the two species, DHAA appears to have antioxidant properties beyond those of AA. DHAA has been described to protect low-density lipoproteins from the oxidation that causes the development of human atherosclerosis (Santosh and David, 2017), has a potent anti-cancer effect (Toohey, 2008), and has a vasodilatory effect (Akolkar *et al*. 2017; Morelli *et al*. 2020). In addition, it is absorbed from the small intestine in humans and reduced to AA intracellularly. This reduction is important for maintaining adequate cellular levels of AA and probably explains the anti-scorbutic effect of DHAA (Deutsch, 2000).

On the other hand, flavonoids are natural compounds that belong to a class of secondary metabolites of plants with a polyphenolic structure that have favourable biochemical and antioxidant effects associated with the prevention of several diseases, such as cancer, Alzheimer's and atherosclerosis, among others (Panche *et al*. 2016). The majority flavonoid, in the case of orange, is

hesperidin, which represents 50% of the total phenolic compounds (O'Shea *et al.* 2012) and is mainly located in the peel (Sánchez-Moreno *et al.* 2003; Roussos, 2011; Escobedo-Avellaneda *et al.* 2014; M'hiri *et al*. 2014; Lahmer *et al*. 2015; Rafiq *et al*. 2019). It improves microcirculation, has high antioxidant, anti-inflammatory and analgesic effects, lowers blood lipids, aids in the healing of venous ulcers and has marked anti-cancer activity, among other health benefits (Kanaze *et al*. 2004; Adham, 2015).

While it is true that there is a large market for antioxidant substances of synthetic origin, it is also true that there is currently a growing concern about the possible toxicity of this type of antioxidants, which is promoting their substitution by compounds of plant origin as healthy natural antioxidants (Dugmore *et al.* 2017; Panwar *et al*. 2021). In this sense, taking into account the potential as a natural source not only of fibre but also of vitamin C and flavonoids from the waste generated in the citrus processing industry, and with the profitability and sustainability of the processes in mind, this waste, with no initial value and high environmental cost, could become a value-added product, fulfilling part of the circular economy strategies in the European agri-food sector (COM, 2020). This new economic model aims to do away with the concept of waste as we know it, focusing on a new paradigm in which every resource is a nutrient for nature, industry or society. The circular economy offers multiple strategies for value creation applicable to the agri-food sector. The utilisation of organic waste for use in human food but also in other sectors are part of these strategies. To this end, it is essential to know how best to stabilize and recover the compounds of interest. In this sense, there are many

studies related to the best way to extract them, but no information has been found comparing the yield of these compounds when extracted from fresh or dehydrated waste. Since the orange juice waste, in addition to the large volume it occupies, may be limited in its subsequent exploitation as it is prone to microbial deterioration (O'Shea *et al*. 2012), its drying would contribute both to stabilising it and to facilitating its handling. From this point of view, the initial hypothesis for this study was to find out which is the best raw material to be dried: the juice waste as such, so that later, when required, the bioactive compounds of interest can be extracted, or directly the interest compound previously extracted. The most appropriate dried product would be the one that provides a higher content of bioactive compounds.

As many bioactive are heat-sensitive compounds (Berk, 2018), in order to retain their nutritional value and potential functionality, it is necessary to select a drying method that does not reach high temperatures, which is favoured if low pressures are applied during the process. Despite other drying methods could be used, freeze-drying can be proposed as the best drying method in the case of the juice waste as to obtain a matter rich in bioactive compounds. Although it is true that freeze-drying is a long and therefore cost-intensive process, its use can be justified if the aim is to provide a source of bioactive compounds with high added value. In this sense, one aspect to be considered could be the use of moderate temperatures during drying to shorten the process without affecting the content of bioactive compounds. For drying purposes, it is also important to note the high content of low molecular weight sugars in orange peel (Pacheco *et al.* 2019). These, when subjected to the

rapid elimination of water, result in an amorphous matrix that can undergo changes related to the glass transition temperature (Tg) that include the development of stickiness, caking and gumming phenomena, which leads to the instability of the dried product (Telis and Martínez-Navarrete, 2012). One of the control strategies to manage this problem is the incorporation of high molecular weight biopolymers before drying to increase its Tg. Gum Arabic and bamboo fibre represent some of these biopolymers, which are also widely known for their encapsulating function (Riguetto and Netto, 2006). Gum Arabic is an exudate of the Acacia tree, widely used in pharmaceutical preparations and in food; it is a good emulsifier and prevents water adsorption, oxidation and the volatilization of compounds (García-Martínez *et al.* 2018). The structure of bamboo fibre, on the other hand, allows it to play a steric role also avoiding stickiness phenomena (Agudelo *et al*. 2017).

As to offer a potential source of bioactive compounds for different purposes, the aim of this study was to know the stability of vitamin C and hesperidin in the dehydrated orange juice waste. Three different factors were considered to this end: i) the matrix to be dehydrated, ii) the use of gum Arabic and bamboo fibre as biopolymers with which to physically stabilise the dehydrated juice waste and which, in addition, can play a certain encapsulating role, and iii) the dehydration temperature.

2. Materials and Methods

2.1 Raw material

The oranges (*Citrus sinensis* (L.) var. Valencia-Late) were bought in a local supermarket (Valencia, Spain). They were selected for their appearance, size, colour, absence of physical damage and firmness. The biopolymers (Bp) used as anti-sticking, anti-wetting and encapsulating agents in the process were gum Arabic (GA, Scharlau, Spain) and bamboo fibre (BF, VITACEL® Rosenberg, Germany).

2.2 Sample preparation

The samples were prepared according to the scheme shown in Fig. 1. The juice (J) and the juice waste formed by the flavedo, albedo, central axis and septa, obtained after the extraction of juice from the oranges using a juice extractor (Orange Juicer w/arm 120W, Lacor, Spain), were studied.

The juice waste was diluted with water, in a 1:1 ratio, for ease of handling, crushed and homogenized at 1400 rpm for 1 min in an emulsifying grinder (Eurofred, Spain), and at 2500 rpm for 40 s and at 9200 rpm for 40 s in a laboratory robot (Thermomix Vorwek TM-21, Spain).

A part of this diluted juice waste (JW) was formulated with 5 g GA and 1 g BF / 100 g sample, obtaining the JWBp sample. The amount of Bp was selected based on the results of previous studies (Agudelo *et al.* 2017). Part of the JW and JWBp samples obtained was reserved for the extraction of the bioactive compounds. This was done with 0.1% oxalic acid in a ratio of 1:9 (JW: oxalic acid) with magnetic agitation (MS-51M, Jeio Tech, Korea) for 10 min. The extract obtained was filtered through a nylon membrane filter 90 μm (VWR, Radnor, PA, USA) to obtain the EJW and EJWBp samples.

All of the prepared and homogenized samples, except J, were placed on 10.2 cm x 7.6 cm aluminium trays with a thickness of 1 cm and frozen at -45ºC (Liebherr LGT 2325, Germany) for 24 h for subsequent freeze-drying. The frozen JW and JWBp samples were separated into two batches and freeze-dried (Teslar Lyo Quest-55, Spain) at -55ºC in the condenser and at a pressure of 0.050 mbar to obtain FDJW and FDJWBp samples. One of the batches was freeze-dried without heating the shelves, at a room temperature of 25ºC, for 26 h. The other batch was freeze-dried with the shelves heated up to 50ºC for 18 h. The EJW and EJWBp extracts were freeze-dried in only one of the conditions used (25ºC, 26 h), which provided the samples FDEJW and FDEJWBp. The times were selected based on previous experiments, trying to obtain products with a water content of under 5 % (data not shown).

2.3 Analytical determinations

The J, JW and JWBp samples were analysed for total soluble solids (ºBrix), at 20 ºC (30PX Refractometer, Mettler Toledo, Japan). The water content of the samples, before being freeze-dried, was analysed according to the AOAC 934.06.2000 method, in a vacuum oven at 60ºC and pressure <100 mmHg (Vacioterm, J.P., Selecta, Spain), until reaching a constant weight. After the freeze-drying process, an automatic Karl-Fisher coulometric titrator was used (C10s Mettler Toledo Compact Coulometric KF Titrator, USA). This parameter was expressed in g water/ 100 g sample. All determinations were carried out in triplicate.

Vitamin C (VC) was analysed considering its two active forms: AA and its oxidized form, DHAA (Sánchez-Mata *et al.* 2000). The analysis was carried out by using high-performance liquid chromatography (HPLC) equipment connected to a DAD detector (Jasco equipment, Italy), a KromaPhase 100 C18 5µm column (250x4.6mm) (Scharlaw, Spain) and the following chromatographic conditions: 20 µL injection volume, 1mL/min flow rate and 0.1% oxalic acid as mobile phase. The identification was made at a wavelength of 243 nm. A set of AA calibration curves were prepared from a battery of AA standard solutions (Dr. Ehrenstorfer, Germany) in 0.1% oxalic acid as a solvent, with five different concentrations, and analysed in quadruplicate. The curves were constructed as a function of the peak area obtained with respect to the known concentration of the analyte.

For the extraction of AA, 1 g of each sample was dissolved in 9 mL of oxalic acid solution (0.1% w/v) (Xu *et al.* 2008). After manual homogenization and resting for 3 min in the dark, the solution was filtered through a 0.45 µm membrane filter before injecting the analyte into the HPLC equipment. The AA determinations were performed in quadruplicate. The procedure used for the determination of the total content of VC (AA+DHAA) was the reduction of DHAA to AA (Sánchez-Moreno *et al.* 2003) using DL-dithiotreitol (DTT) (Scharlau, Spain) as a reducing agent. 0.5 g of fresh sample or 0.075 g of freeze-dried sample were taken to react with 2 mL of DTT solution (20 g/L) for 2 h in the dark and at room temperature. Subsequently, the same procedure as that used for the AA method was performed.

The analysis of hesperidin (HES) was carried out by HPLC, at a wavelength of 284 nm, using a gradient of water and methanol grade HPLC (VWR, Spain) for the mobile phase. HES was extracted with dimethylsulfoxide (DMSO) (Scharlau, Spain) grade HPLC (Manthey and Grohmann, 1996; Gómez-Mejía *et al.* 2019), taking 1 g of fresh sample or 0.075 g of freeze-dried sample to react with 1 mL of double-distilled water and 2 mL of DMSO, under magnetic agitation for 10 min (MS-51M, Jeio Tech, Korea). Subsequently, the solution was centrifuged at 2031 xg (GYROZEN 123GR, Korea) for 10 min, at 4ºC. The supernatant was filtered through a 0.45 µm membrane filter for injection into the HPLC. The measurements were taken in triplicate.

To make the results comparable, the values of VC, AA, DHAA and HES of the samples, both before and after being freeze-dried, were expressed in mg compound/100 g sample before freeze-drying, without added biopolymers, and, in the case of juice waste samples, before being diluted.

2.4 Statistical analysis

In order to determine the influence of the matrix (the JW or the VC and HES extracted from the JW), the incorporation or not of biopolymers to these matrices (the addition or not of GA + FB) and the freeze-drying temperature (25 or 50 ºC) on the stability of the bioactive compounds in the freeze-dried JW, the experimental design followed with the samples processed as described in section 2.2 is shown in Table 2. Experiments were conducted in triplicate. All the data were expressed as mean ± standard deviation. In order to determine the differences between the samples, an analysis of variance

(ANOVA), using the Fisher's least significant difference (LSD) procedure with a 95% confidence level (p-value < 0.05), was carried out. Statistical analyses were performed by Statgraphics Centurion versión XVIII (Statgraphics Technologies, Inc. The Plains, Virginia).

3. Results and Discussion

3.1 Characterization of orange juice and juice waste before freeze-drying

Table 1 shows the soluble solid, water, VC, AA and HES content of the orange juice and its waste before being freeze-dried and, in the case of juice wastes, referred to the sample before being diluted. The soluble solid and water content were similar to that reported by other authors (Escobedo-Abellaneda *et al.* 2014; Pacheco *et al.* 2019; Sánchez-Moreno *et al.* 2003). The JW, with the presence of a higher soluble fibre content, presented a significantly lower water content and greater soluble solids than J. On the other hand, and as expected when incorporating GA and BF, there was a significant decrease in the water content and an increase in the soluble solids, especially related to the presence of GA, as BF is mainly insoluble (Contreras *et al.* 2014).

The JW presented more VC than the juice, although the former had lower values of AA ($p<0.05$). Taking into account that the VC content is the sum of the AA and DHAA contents, we can conclude that the higher concentration of the VC of the JW is due to the high concentration of DHAA. Escobedo-Avellaneda *et al.* (2014) showed similar results. They found that orange peel contains more DHAA than AA, contrary to what happens in the edible part of

the fruit, which is justified by the barrier that the peel represents against the oxidation of AA in the edible part. The HES content was also higher in the JW than in the J (Table 1). The low solubility in water of this compound could partially justify these differences (Manthey and Grohmann, 1996).

The added Bp favours the extraction of bioactive compounds because it decreases the viscosity of the samples, which in turn favours encapsulation and protects the product from its loss (Mishosieini *et al.* 2010; Shamsara *et al.* 2017). This can be observed for each of the studied compounds, although this increase was not significant ($p>0.05$) in the case of AA. This compound's increased sensitivity to oxidation during juice waste handling, when incorporating the biopolymers, could justify this result.

3.2 Impact of freeze-drying on the stability of VC, AA, DHAA and HES

The FDJW and FDJWBp, at 25 and 50 ºC, presented a similar water content, with no significant differences ($p>0.05$), reaching a mean value of 0.045 ± 0.006 g water/g sample. The water content of the freeze-dried extracts was not affected by biopolymers addition ($p>0.05$), the mean value being 0.073 ± 0.003 g water/g sample. In the first case, the value was in the expected order, but in the case of the extracts, the water content was higher, which may be related to their higher hygroscopicity. The freeze-dried extract obtained turned out to be a dry solid foam that, from a thermodynamic point of view, behaved in a very unstable way. This structure collapsed quickly due to its large interfacial area, which proved to be highly hygroscopic, exhibiting significant

stickiness and suffering structural contraction in the presence of environmental humidity.

As to study the best way to stabilize the bioactive compounds of the orange juice waste, VC was selected as it has been considered a nutritional quality indicator frequently used in different industrial processes due to its easy degradation (Klimczak *et al.* 2007). Thus, we compared the VC, AA and DHAA content of the freeze-dried juice waste, with and without biopolymers added, with that of the corresponding freeze-dried extract. For this study, all the samples were freeze-dried at 25 ºC (Fig. 2). FDEJW and FDEJWBp presented a lower VC, AA and DHAA content than FDJW and FDJWBp. These results coincide with the results obtained by Riguetto and Netto (2006) when comparing the stability of synthetic AA with the VC present in green West Indian cherry juice. The difference could be due to the greater exposure of the VC in the case of the extracts, which facilitates its degradation. In this sense, a protection of the VC from the whole juice waste matrix in general and from some specific compounds, such as phenolics in particular, has been described (Miller and Rice-Evans, 1997; Riguetto and Netto, 2006). For both these reasons and also due to the marked instability of the freeze-dried extract mentioned above, it was decided to study the whole freeze-dried waste of the orange juice directly in order to meet the rest of the objectives of the study.

Fig. 2 shows the content of VC, AA and DHAA of the freeze-dried juice waste at both temperatures, 25 and 50 ºC. All the freeze-dried juice waste presented a higher concentration of VC compared to the juice waste before being freeze dried ($p<0.05$, Table 1). As it is not easy to explain that a product can gain VC

during its freeze-drying, it seems reasonable to think that the result is a consequence of the greater or lesser ease with which the compound is extracted from the matrix. The better accessibility of the VC in the freeze-dried juice waste favours its extraction (Spigno *et al.* 2007).

As regards the impact of the added biopolymers and the drying temperature on the stability of VC along FD, a different behaviour was observed. At 25 ºC it seems that the incorporation of Bp, although it protects AA along vacuum drying, promotes the degradation of DHAA so that a decrease in the VC was finally quantified. This could be due to the GA composition, one of the added Bp, which is formed by polysaccharide chains and proteins (Atgié *et al.* 2019). It has been described that DHAA, in low water content systems, reacts with proteins to form other degradation products, such as scorbamic acid (Hayashi *et al.* 1985; Larisch *et al.* 1996;). When the drying was carried out at 50 ºC, the AA content of the sample with no added Bp was not affected but a decrease in the DHAA was observed ($p<0.05$), demonstrating the great instability of this compound described by other authors (Wechtersbach *et al.* 2011, Bradshaw *et al*. 2011). In the sample with Bp dried at 50ºC, despite AA decreases the DHAA content increases with respect to the sample obtained at 25ºC ($p<0.05$). On the one hand, a degradation of AA to DHAA has been described with temperature (Bradshaw *et al.* 2011). On the other hand, Riguetto and Netto (2006), observed a greater degree of protection by several DHAA encapsulants at temperatures of between 35 and 45ºC than at temperatures of between 15 and 25ºC. All these contributions lead to a significant ($p<0.05$)

decrease or increase of VC in the absence or presence of Bp, respectively, as the drying temperature increases.

Fig. 3 shows the HES content of the dried samples obtained at the two temperatures considered. As compared to data shown in Table 1, an increase of this compound was observed after freeze-drying ($p<0.05$), as occurred with VC, probably due to the high porosity of the freeze-dried sample, favouring the extraction of bioactive compounds. The behaviour of the HES of the dried samples was similar and neither the temperature nor the incorporation of Bp significantly affected its content ($p>0.05$). However, although it was unlikely that there were any significant differences, a greater HES content was observed in FDJWBp samples. A much smaller effect was observed in the case of temperature and, in any case, it can be confirmed that increasing the temperature during freeze-drying up to 50 °C does not have a negative effect on HES.

From this point of view, in addition to the benefit of adding GA to favour the extraction of VC and HES from the natural juice waste, its presence seems to be particularly advisable when drying is to be carried out at a higher temperature, in this case at 50 ºC.

Taking into account the results of this study, it seems appropriate to propose the dehydration of orange juice waste as an emerging method for its integral valorisation. Its final use for the extraction of bioactive compounds for different purposes could be an application. But it also seems interesting to study the possibility of using the dehydrated juice waste in a powdered form as an ingredient for human food. Given the growing awareness of consumers about

the food-health relationship, but also about environmental issues, the food production sector should be particularly sensitive to any initiative that leads to a green production as to improve sustainability and reduce the negative environmental and ecological impacts of this key socio-economic sector. Considering integrated valorization approaches to convert a waste into a human food ingredient will require, on the one hand, consumer education for acceptance of the novel food and, on the other, close attention to the economics associated with the process to ensure cost-effectiveness. In this case, the scale up of this laboratory based trial seems simple in terms of the equipment needed, although an economic study would obviously be necessary to support the proposal. In this sense, this research team advocates the cost-effectiveness of the approach. This is so for two fundamental reasons. On the one hand, the fact that we have seen that freeze-drying of the juice waste with Bp at 50 ºC does not entail losses of the bioactive compounds studied and allows the process time, and therefore its economic cost, to be reduced by 30 %. On the other hand, an economic study comparing the obtaining of grapefruit powder points out that it is much more profitable to use freeze-drying rather than spray-drying, another dehydration technique known for being cheaper and leading to products of similar quality, the latter costing 2.3 times more than the former (Camacho *et al.*, 2019). Despite the greater electrical cost of freeze-drying, this is a consequence of the much higher costs of the raw material, caused due to the loss of powder that remains adhered to the walls and nozzle of the spray-dryer in the case of fruit processing.

4. Conclusions

Orange juice waste has a higher content of VC and HES than the edible part of the fruit, which makes it very interesting for use as a natural source of these compounds. The VC dried extract was physically very unstable and difficult to handle, in addition to the fact that it showed a very low content of VC associated with its greater exposure to external factors when deprived of the matrix that provides all the juice waste. For this reason, to dry the juice waste for the subsequent extraction of the bioactive compounds may be proposed. In order to extract the highest amounts of VC and HES from the juice waste, it is recommended both to incorporate GA and BF into the formulation and also to increase the drying temperature to 50ºC which, in addition, contributes to the shortening of the process time. In this way, this dried orange juice waste may be proposed as a value-added product, fulfilling part of the circular economy strategies in the agri-food sector. This integral valorisation proposal contributes to reduce the amount of juice waste finally disposed into the environment. The dehydrated juice waste could be used as a nutraceutical or as an ingredient for human food.

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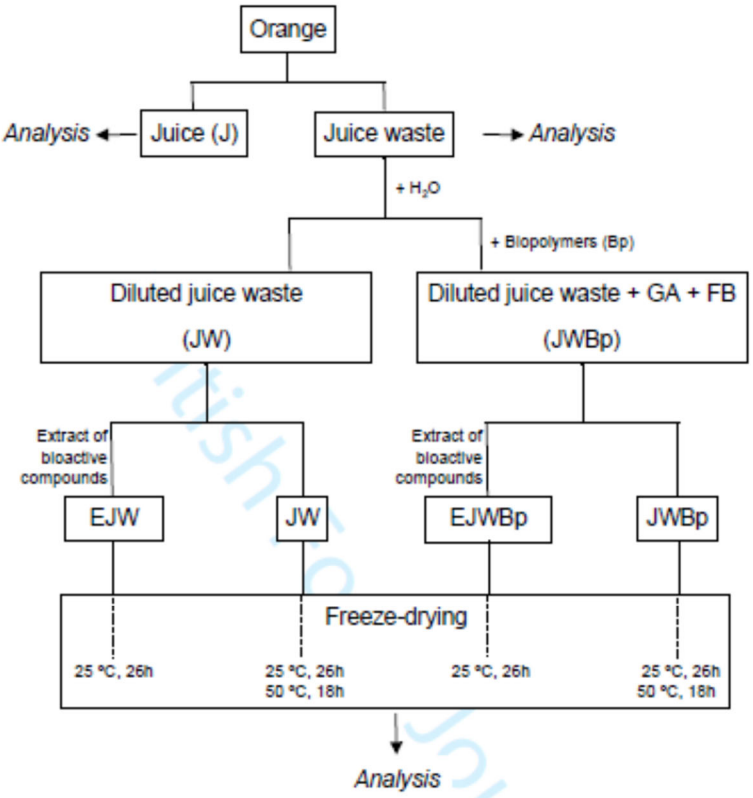

Figure 1. Scheme of sample preparation for subsequent analysis.

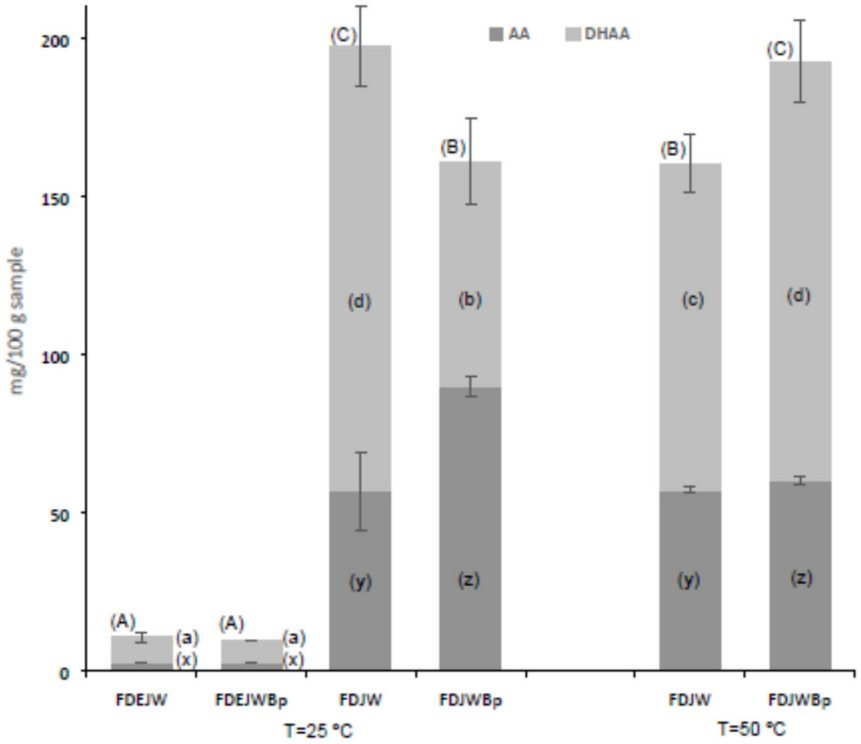

Figure 2. Vitamin C (VC), ascorbic acid (AA) and dehydroascorbic acid (DHAA) content of the juice waste, without (FDJW) and with (FDJWBp) added biopolymers freeze dried at 25 and 50 ºC, and values of the VC extracts (FDEJW and FDEJWBp) freeze dried at 25 ºC. Mean values referred to the sample before freeze-drying and the addition of biopolymers (mg/100g). Different letters indicate significant differences ($p<0.05$) for the ANOVA performed between the samples: A-C for VC; x-z for DHAA and a-c for AA. The method currently being used to discriminate among the means is Fisher's least significant difference (LSD) procedure.

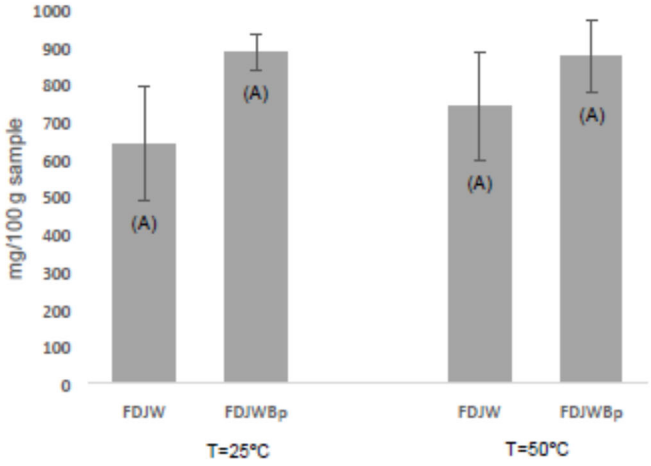

Figure 3. Hesperidin (HES) content of the juice waste without (JW) and with added biopolymers (JWBp) freeze dried at 25 and 50 °C. Average values referring to the sample before freeze-drying and the addition of the biopolymers (mg/100g). The same letters indicate no significant differences ($p>0.05$) for the ANOVA performed between the samples. The method currently being used to discriminate among the means is Fisher's least significant difference (LSD) procedure.

Table 1. Chemical and physicochemical composition of the juice (J) and its waste, without and with added biopolymers (JW and JWBp, respectively).

Sample	ºBrix[1]	X_w[2]	VC[3]	AA[3]	HES[3]
J	12.10 ± 0,15 [a]	0.8930±0.0005 [c]	49 ± 4 [a]	53 ± 3 [b]	38 ± 6 [a]
JW	30.96 ± 0,19 [b]	0.7320±0.0007 [b]	133 ± 3 [b]	37 ± 3 [a]	507 ± 21 [b]
JWBp	36.20 ± 0,30 [c]	0.7260±0.0018 [a]	144± 4 [c]	38 ± 5 [a]	615 ± 14 [c]

[1] g soluble solids/100 g liquid fraction of the sample before dilution

[2] g water/g sample before dilution

[3] VC: Vitamin C; AA: Ascorbic acid; HES: Hesperidin, all expressed in mg /100 g sample before dilution and without Bp

a-c: Superscripts of different letters in the same column indicate significant differences between the samples ($p<0.05$) for the ANOVA

Table 2. Experimental design followed to determine the influence of the matrix, use of biopolymers and drying temperature on the bioactive compounds studied (VC: vitamin C; HES: hesperidin). The codes used correspond to the samples shown in Fig. 1.

	Sample			
T (°C)	FDJW	FDEJW	FDJWBp	FDEJWBp
25	VC, HES	VC	VC, HES	VC
50	VC, HES	-	VC, HES	-