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Effect of freezing, osmodehydro-freezing, freezedrying and osmodehydro-freezedrying on the physicochemical and nutritional properties of arazá (*Eugenia stipitata* McVaugh)

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ABSTRACT

Arazá, a berry rich in heat-sensitive bioactives (phenolics/carotenoids/flavonoids/ascorbic acid), may be suitable for preparing functional foods, however, its high perishability hinders its industrial application. Although freezing (Fr) and freezedrying (LIO) are popular methods for preserving fruits, they may cause undesirable quality changes. Adding an osmo-dehydration pretreatment (OD) before freezing (osmodehydro-freezing/OD-Fr) or freezedrying (osmodehydro-freezedrying/OD-LIO) diminishes processing time which may improve the final product quality This study analyzed the effect of Fr/LIO/OD/OD-Fr/OD-LIO on color, antioxidant content/activity, polyphenol composition and bioaccessibility (BAC) of arazá.

OD pretreatment increased freezing rate (58 %) and reduced osmodehydro-frozen arazá drip-loss (40%) Osmodehydrated samples presented the highest discoloration levels (15.7), freezing/freezedrying pretreated arazá improved them 16–48 %.

Fr/LIO gave the best results regarding polyphenol content (99–48 %), and activity (97–88 %) retention; whereas OD/LIO produced the highest losses (59–84 %).

Results showed that in comparison with untreated fruit, freezing arazá did not affect the bioaccessible antioxidants content/activity, conversely, freezedrying reduced antioxidant activity and total-flavonoids BAC levels (14–21 %) without modifying polyphenols/carotenoids bioaccessibilities.

Although osmodehydro-freezing increased total-polyphenol bioaccessibility (22 %) without affecting that corresponding to total-flavonoids, it reduced antioxidant activity retention (16–42 %). Osmodehydro-freezdrying impact was negative on all properties (13–55 %) except Ferric-Reducing-Antioxidant-Power that increased (10 %).

1. Introduction

Arazá (*Eugenia stipitata* Mc Vaugh) is a tropical berry highly appreciated because of its flavor and nutritional properties. The fruit is rich in heat-sensitive bioactives including vitamin C, cinnamic, gallic acids, kaempferol, quercetin, myricetin, as well as carotenoids like β-chryptoxanthin, zeinoxanthin and lutein, with proven antigenotoxic and antimutagenic properties. (Neri-Numa et al., 2013). However, the fruit`s fragility and high perishability requires a careful selection of the processing technique and the operating conditions to improve its shelf life

Abbreviations: AA, ascorbic acid; ARA, antiradical activity; BAC, bioaccessibility; BI, browning index; CHLO, chlorogenic acid; CIN, cinnamic acid; ΔE, degree of discoloration; EGlu, eriodictyol-7-O-glucoside; FA, untreated arazá; Fr, freezing; FRAP, ferric reducing antioxidant power; GA, gallic acid; LIO, freezedrying; NID, unidentified compound; OD, osmotic dehydration; OD-Fr, osmodehydro-freezing; OD-LIO, osmodehydro-freezedrying; QRut, quercetin 3-O-rutinoside; RH_2O, rutin hydrate form; RET, retention; TP, total polyphenols; TF, total flavonoids; TC, total carotenoids; tFER, trans-ferulic acid; Wc, water content.

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without impairing its sensory and nutritional properties.

Although freezing (Fr) is one of the most used techniques for fruit preservation, its application in fruits with a high water content may produce undesirable alterations in color and texture as well as in its physicochemical and nutritional properties (Giannakourou et al., 2020). Osmodehydrating (OD) fruit before freezing (osmodehydro-freezing, OD-Fr) lowers the amount of freezable water and increases the soluble solids concentration, leading to a reduction of the freezing temperature and an enhancement of the freezing rate. These changes diminish the process duration and its consumption of energy as well as the packaging, distribution and storage costs (Ramallo & Mascheroni, 2010). In addition, the water loss and solids gain can increase the products glass transition temperature extending the range of storage temperatures within which the product remains in the glassy state (Giannakourou et al., 2020).

Several studies reported that an osmodehydrating pretreatment reduced freezing duration in osmodehydro-frozen pineapple (Ramallo & Mascheroni, 2010), preserved the texture of cucumbers, and vitamin C retention in tomatoes (Giannakourou et al., 2020) and improved color, texture, drip loss, as well as polyphenol and aromatic compounds contents in mango (Zhao et al., 2016). Freezedrying (LIO) is considered one of the best methods for drying heat-sensitive products; however, since this process includes a freezing step before ice sublimation, the physicochemical and nutritional quality of the freezedried product will be strongly influenced by the freezing rate; therefore, an OD pretreatment (osmodehydro-freezedrying, OD-LIO) may also affect the quality of the final product. Osmodehydro-freezedrying fruit reduced the drying time and operational costs, improved color, texture and structure retention in strawberries, apples and chinese yam. (Dziki, 2020).

The efficacy of a product health benefits depends on its bioactives composition and activity combined with their bioaccessibility (BAC) and stability under gastrointestinal digestion conditions (Ribas-Agusti et al., 2018). Only polyphenols released from the food matrix by the action of digestive enzymes and microbiota are bioaccessible in the gut and therefore potentially bioavailable. The disruption of the natural matrix along with the microstructure created during processing may influence the release, transformation and subsequent absorption of nutrients in the digestive tract. Freezing and freezedrying may affect the initial physicochemical properties as well as the cellular structure of food products, which can result in changes in nutrients release, bioaccessibility and bioavailability (Ribas-Agusti et al., 2018). These changes are strongly dependent on the nature of the polyphenolic compound, the characteristics of the food matrix and the operating conditions. Dalmau et al. (2017) showed that freezing or freezedrying apple disrupted the cellular structure and diminished the total polyphenols (TP) bioaccessibility and antioxidant activity retention. However, in a later study using beetroot, Dalmau et al. (2019) reported an increment of TP bioaccessible levels and antioxidant activity retention. Kamiloglu (2019) demonstrated that freezing strawberries increased total polyphenols, total flavonoids (TF) and anthocyanins bioaccessibility levels following simulated gastrointestinal digestion.

There are very few publications analyzing freezing influence on the quality of arazá and in all of them, the fruit is frozen as a paste (García-Reyes et al., 2010; Silva-Bustos et al., 2011; Narváez-Cuenca et al., 2015). Cutting, grinding, and pressing the fruit reduces particle size, increasing the dried material surface area. These changes accelerate the drying rate and, as a result, may modify the characteristics of the dried product (Dziki, 2020); therefore, those results are not applicable to whole pieces.

Iturri et al. (2021) and de Araujo et al. (2021) determined the effect of gastrointestinal digestion on the polyphenol composition of microencapsulated powders or ground microencapsulated freezedried arazá. To our knowledge, this is the first study evaluating the influence of freezing, osmodehydro-freezing and osmodehydro-freezedrying on the physicochemical properties and bioaccessibility of arazá whole cuts.

Specific objectives were to determine the effects of:

(a) Freezing, freezedrying, osmotic dehydration, osmodehydro-freezing and osmodehydro-freezedrying on the color, antioxidant content, activity and polyphenol composition.
(b) Osmotic dehydration pretreatment on the soluble solids concentration, freezing time and drip loss from frozen arazá.
(c) Process influence on antioxidant content/activity/bioaccessibility of gastro-intestinally digested arazá.

2. Materials and methods

2.1. Fruit selection and processing

Fresh arazá (*Eugenia stipitata,* McVaugh; FA), purchased at a local market in Ibagué (Colombia), was selected according to its maturity stage (intermediate, yellow-green pulp), size (10–14 cm diameter) and stored until processing (7 days, 4ºC). After storage, samples water content (H;%), total soluble solids (TSS) and titratable acidity (TA) were 94.52±2.39 % w.b. (wet basis), 6.41±1.32°Bx and 2785±221 mg malic acid (100 g w.b)$^{-1}$ respectively.

2.1.1. Osmotic dehydration

After washing, and peeling, fruit samples were cut in pieces (6 × 1 cm) and osmo-dried (OD) for 60 min with a commercial sucrose (Ledesma, Ledesma, Argentina) solution (60ºBx), in a shaker (TT 400, FERCA, Buenos Aires, Argentina) at 100 cycles min^{-1}, 40ºC and a sample/solution ratio =1:10 (w/w). The operating conditions (60 min/40ºC/60ºBx) were selected according to Reyes-Alvarez et al. (2022) recommendations for obtaining osmodried arazá with high levels of antioxidant content/activity and overall acceptability.

2.1.2. Freeze-drying

Untreated (FA) and osmo-dehydrated (6 × 1 cm) arazá cuts, were placed forming a single layer in aluminum trays (≈300 g), and frozen at −35 °C (48 h (h)) followed by a 24 h period at −80 °C. The frozen fruits were freeze-dried (48 h) with a FIC L1–1-E300-CRT (Buenos Aires, Argentina) equipment (vacuum pressure<13.32 Pa; −35 °C (condenser temperature); 22 °C (heating plate)). The freeze-dried samples, wrapped in polyethylene/polyamide film (70 μm), were stored at −18ºC until analysis (< 7 days).

2.1.3. Freezing and thawing

Untreated and osmodehydrated samples (6 × 1 cm) were precooled at 4 °C for 12 h and then frozen with a conventional air-blast tunnel (Friotecnología S.R.L, Argentina) at an air temperature of −30 °C. Fruit temperature was determined with copper- constantan thermocuples at the samples core. Freezing time was defined as the time necessary for diminishing the temperature in the center of the samples from 0ºC to −18ºC (Ramallo & Mascheroni, 2010). Frozen (Fr) and osmodehydro-frozen (OD-Fr) samples were thawed inside a refrigerator at 4ºC for 10 h.

2.2. Water content, total soluble solids, titratable acidity and color

Samples water content (Wc,%) was analyzed according to AOAC 934-06 method (AOAC, 1998) while the total soluble solids (°Bx) was measured with a refractometer Atago N2 (Tokyo, Japan). Titratable acidity (TA) was determined by titrating fruit samples (5 g) in a 50 ml dilution with 0.1 N NaOH to pH 8.2 (Nielsen, 2010).

Color, expressed with the CIE L*a*b* coordinates, was measured with a Minolta CR-400 Chroma Meter (Minolta, Osaka, Japan), each value was the average of 9 measurements on triplicate samples. The total color changes relative to the untreated samples (ΔE; Eq. (1) were calculated as:

Drying and freezing may increase browning therefore color variations were also monitored using the browning index (BI; Eqs. (2) and (3); Maskan, 2001).

$$BI = \frac{[100(x - 0.31)]}{0.17} \quad (2)$$

where

$$x = \frac{(a + 1.75L)}{(5.645L + a - 3.012b)} \quad (3)$$

All determinations were done in triplicate samples.

2.3. Drip loss

Drip losses (DL) during thawing of frozen (Fr) or osmodehydro-frozen arazá (OD-Fr) were quantified on triplicate samples following Ramallo and Mascheroni (2010) protocol. The frozen samples were placed on blotting paper and thawed for 2 h at 20 °C inside tightly closed jars to minimize evaporation. DL values (g liquid*(g FA d.b (dry basis))$^{-1}$) were calculated with Eqs. (4) and (5); M_0 and Mi represent the frozen sample weight (g) of the untreated and osmo-dried samples respectively whereas W_0 and Wt correspond to the weight of the dry and wet paper before thawing and at time t.

$$DL(Fr) = \frac{W_t - W_0}{M_0} \quad (4)$$

$$DL(OD - Fr) = \frac{W_t - W_0}{M_0} \quad (5)$$

2.4. Antioxidant content/activity and polyphenol composition

Antioxidants were extracted with a methanol/H2O (50:50 v/v, pH=2.0) mixture as described by Reyes-Alvarez and Lanari (2020).

The total phenolic content of the extracts (TP; mg GAE (gallic acid equivalents)*g^{-1} w.b) was assessed according to Schlesier et al. (2002).

Total flavonoids (TF; mg CAT (catechin equivalents)*g w.b^{-1}) and carotenoids levels (TC; mg β-carotene equivalents*g w.b^{-1}) were determined according to Chang et al. (2002) and Ordoñez-Santos et al. (2014) respectively. Ascorbic-acid (AA; mg AA*(100 g w.b)$^{-1}$) concentration was analyzed by high-performance liquid chromatography (Waters, model R-414, Milford, USA) as described by Reyes-Alvarez and Lanari (2020).

Antiradical activities (ARA; Eqn. (5)) were analyzed with the DPPH scavenging assay (Brand-Williams et al., 1995) using ethanol as solvent. 100 µl of extract were mixed with 3.9 ml of a DPPH• ethanolic solution (25 mg/L). The absorbance reduction was measured with a Shimadzu UV–VIS (Seisakusho Ltd., Kyoto, Japón) spectrophotometer at 517 nm during 120 min when the reaction reached a plateau.

$$ARA(\%) = \frac{Absb_0 - Abss_t}{Absb_0} * 100 \quad (6)$$

$Absb_0$ and $Abss_t$ correspond to the blank (b) and the sample (s) absorbancies at 0 and 120 min.

To analyze the Ferric Reduction Antioxidant Power (FRAP), 900 µl of the FRAP reagent (TPTZ, $FeCl_3$, acetate buffer) were mixed with 30 ul of the extract and 90 µl distilled water. Ferric Reducing Antioxidant Power (FRAP; µM Fe^{+2}*g w.b^{-1}) was evaluated after 30 min reaction time (Pulido et al., 2000) at 593 nm with a calibration curve ranging from 100 to 1200 µM Fe^{+2}.

To study the influence of the different processing methods (OD, LIO, Fr, OD, OD-Fr, OD-LIO) on the nutritional properties, the antioxidants concentrations and FRAP values of the treated samples were expressed per weight of dry untreated fruit (g FA d.b) while ARA was calculated as mg GAE*(g FA d.b)$^{-1}$.

Polyphenol composition was analyzed following Soares et al. (2019) protocol with a Waters Model 6000A (Milford, USA) chromatograph equipped with a diode array detector (DAD) and a C-18 Altex-Ultrasphere™-ODS column (250 mm×4.6 mm i.d, 5 µm part size). Compound identification was done comparing their DAD spectra and retention times (Rt (min)) with those from commercial standards (Sigma-Aldrich, Buenos Aires, Argentina) and from Soares et al. (2019) publication. Polyphenols concentrations were expressed per weight of dry untreated fruit (g FA d.b). All determinations were done in triplicate samples.

2.5. Simulated in vitro gastrointestinal digestion

The effect of gastrointestinal digestion on the antioxidants concentrations and the antioxidant activity of the samples was assessed following Chiang et al. (2013) protocol. The method comprised 2 sequential steps: a gastric digestion (G) (pepsin/HCl pH= 2.5 Sigma-Aldrich, Buenos Aires, Argentina) followed by intestinal digestion (GI) (pancreatin/bile salts pH= 8). Before digestion, the samples were homogenized in milli-Q water (1:10) for 5 min with a vortex (Precytec, Argentina).

To determine the effect of each digestion step (gastric or intestinal) on TP, TC, TF, ARA and FRAP values, fractions of the gastric or intestinal digesta were collected, centrifuged (10,000 g/10 min/25 °C; Rolco CM 2036, Buenos Aires, Argentina) and the supernatant was filtered with a membrane (0.45 µm diameter pore).

Antioxidants bioaccessibility (BAC) and activity retention (RET) were calculated with Eqs. 7 and 8.

$$BAC = \left(\frac{c}{d}\right) \quad (7)$$

$$RET = \left(\frac{AOAdig}{AOAnondig}\right) \quad (8)$$

"c" and "d" represent the antioxidant content before and after the simulated digestion respectively and AOA the antioxidant activity values. All results were expressed per weight of dry untreated fruit (FA).

2.6. Statistical analysis

All results of the physicochemical analysis were reported as mean ±standard deviation (SD) of at least triplicate samples. Treatment effects were determined by analysis of variance followed by pairwise comparisons with the Tuckey test ($P<0.05$) (Infostat v. 2013 Universidad Nacional de Cordoba, Argentina).

The correlation among the different physicochemical properties (color, antioxidant content/activity, polyphenol composition, bioaccessible polyphenol level) and their association with the treatments (osmotic dehydration, freezing, freezedrying, osmoticdehydro-freezing, osmoticdehydro-freezedrying) was analysed using Principal Component Analysis (PCA; Infostat v. 2013). The input data set was automatically scaled with the standard scaler function; the principal components number was selected considering the lowest quantity necessary to explain at least 80 % of the variability.

3. Results and discussion

3.1. Effect of freezing and freeze-drying on the physicochemical properties of untreated and osmodried arazá

Table 1 shows the effect of freezing (Fr), freezedrying (LIO), osmotic dehydration (OD), osmodehydro-freezing (OD-Fr), and osmodehydro-freezedrying (OD-LIO) on the water content (H%), total soluble solids level (TSS), freezing time, drip loss (DL), degree of discoloration (ΔE) and browning index (BI) along with the antioxidants contents and activities of arazá pulp.

Osmotic dehydration reduced FA water content by 32.7 % ($P<0.05$) and increased TSS 433 %; results showed that although freezing/thawing osmodried arazá only diminished its humidity an additional 3 %, due to drip loss, the effect was still significant ($P<0.05$). On the other hand,

Table 1
Effect of freezing (Fr), freeze-drying (LIO), osmotic drying (OD), osmodehydro-freezing (OD-Fr) and osmofreeze-drying (OD-LIO) on the physicochemical properties of arazá.

Treatments	H (%)	TSS	Fr time	DL	ΔE	BI
FA	94.19 ±0.42^{a}	6.41 ± 1.32^{b}			0	54.36 ±2.53^{d}
Fr	93.52 ±0.17^{a}		9.53 ±2.01^{a}	0.14 ±0.04^{a}	13.12 ±0.53^{b}	83.41 ±1.67^{c}
LIO	28.76 ±0.10^{d}				8.28 ±0.42^{d}	55.53 ±1.42^{d}
OD	59.76 ±0.04^{b}	34.20 ±2.65^{a}			15.70 ±0.19^{d}	94.42 ±3.40^{a}
OD-Fr	56.85 ±1.53^{c}		4 ± 1.01^{b}	0.10 ±0.01^{b}	13.34 ±0.72^{a}	80.42 ±2.61^{c}
OD-LIO	17.33 ±0.05^{e}				10.42 ±0.60^{c}	89.39 ±1.10^{b}
Treatments	TP	TF	TC	AA	ARA	FRAP`
FA	383.01 ±2.28^{a}	53.66 ±0.26^{a}	60.92 ±1.75^{a}	195.01 ±6.96^{e} ^{a}	507.77 ±4.80^{a}	2656.55 ±19.92^{a}
Fr	379.21 ±4.94^{a}	48.23 ±0.50^{b}	60.71 ±0.72^{a}	118.61 ±3.47^{b}	503.61 ±2.74^{a}	2605.45 ±17.28^{a}
LIO	357.33 ±2.61^{b}	47.73 ±0.08^{b}	50.42 ±3.06^{b}	159.95 ±4.56^{c}	494.40 ±2.42^{a}	2343.92 ±5.08^{b}
OD	117.08 ±0.61^{c}	12.84 ±1.11^{c}	42.82 ±0.41^{c}	102.67 ±3.39^{d}	218.38 ±2.48^{b}	1456.23 ±16.89^{c}
OD-Fr	100.23 ±4.38^{d}	12.60 ±0.14^{c}	42.63 ±0.16^{c}	103.10 ±3.70^{dc}	216.77 ±3.91^{b}	1391.15 ±5.47^{d}
OD-LIO	69.81 ±2.74^{e}	8.51 ±0.45^{d}	25.14 ±0.19^{d}	50.37 ±1.34^{e}	143.83 ±1.96^{b}	874.87 ±6.53^{e}

H%: water content; TSS (°Brix): total soluble solids; Fr time (min): freezing time; DL: drip loss; ΔE: discoloration; BI: browning index; TP: Total polyphenols (mg GAE(g FA d.b)$^{-1}$); TF: Total Flavonoids (mg CAT(g FA d.b)$^{-1}$); TC: Total Carotenoids (mg β-carotene eq(g FA d.b)$^{-1}$); ARA: Antiradical activity (mg GAE(g FA d.b)$^{-1}$); FRAP: Ferric Reducing Antioxidant Power (μM Fe^{+2} (g FA d.b)$^{-1}$). Superscripts with different letters within the same column indicate significant differences ($P< 0.05$; Tukey).

osmotic dehydration did not influence the freeze-drying dehydrating capacity ($P>0.05$) as H(%) levels in LIO and OD-LIO products were similar ($P>0.05$).

Experimental results from the freezing profiles (data not shown) indicated that the partial dehydration and total soluble solids (TSS) increments produced by OD enhanced the freezing rate, reducing the freezing times from 9.53 min (Fr) to 4 min (OD-Fr). Ben Haj Said et al. (2016) and Ramallo and Mascheroni (2010) reached similar conclusions working with apple and pineapple.

In accordance with previous publications with apples, kiwi, pears, strawberries (Marani et al., 2007) or melons (Maestrelli et al., 2001), osmodehydro-freezing reduced arazá's drip loss by 29 % (Table 1). Schudel et al. (2021) reported that the freezing rate increment and the freezing temperature drop generated by the OD pretreatment induced the formation of smaller ice crystals resulting in a reduction of cellular damage and consequently a lower drip loss.

Treatments impact on the total color difference and the browning index in increasing order were:

$$\Delta E(LIO) < \Delta E(OD\text{-}LIO) < \Delta E(Fr) \approx \Delta E(OD\text{-}Fr) < \Delta E\ (OD)$$
$$BI(FA) \approx BI(LIO) < BI(Fr) \approx BI(OD\text{-}Fr) < BI\ (OD\text{-}LIO) < BI(OD)$$

ΔE values from the freezedried samples (LIO; OD-LIO) were 16 %−47 % lower ($P<0.05$) than those from the frozen (Fr; OD-Fr) and the osmodried (OD) products. These results were expected since lyophilization is considered one of the best methods for preserving color from thermosensitive material. Dziki (2020) informed that, in comparison with fruits dried with traditional methods, the freezedried products had better color, flavor and appearance.

Although the osmodried arazá had ΔE highest values, freezing the pretreated fruit reduced them to levels similar to those of the frozen fruit. Marani et al. (2007) and Zhao et al. (2014) working with osmodehydrofrozen strawberries and mangoes reported similar results. In accordance with Assis et al. (2018) using osmo-freezedried apples, freezedrying reduced by 34 % the ΔE level from OD pretreated arazá.

Freezedrying untreated arazá did not change BI value ($P>0.05$), however, freezing or osmotic dehydration increased it by 53 % and 74 % respectively ($P<0.05$). Bhat et al. (2021) reached the same conclusion analyzing osmodehydrated kiwi.

No significant interaction was detected between freezing and osmotic dehydration ($P>0.05$) in the browning index from osmodehydrofrozen samples, in contrast, BI(OD-LIO) was 61 % higher than BI(LIO).

The influence of the different treatments on antioxidant contents and activities was:

$$TP(FA) \approx TP(Fr) > TP(LIO) > TP(OD) > TP(OD\text{-}Fr) > TP(OD\text{-}LIO)$$
$$TF(FA) > TF(Fr) \approx TF(LIO) > TF(OD) \approx TF(OD\text{-}Fr) > TF(OD\text{-}LIO)$$
$$TC(FA) \approx TC(Fr) > TC(LIO) > TC(OD) \approx TC(OD\text{-}Fr) > TC(OD\text{-}LIO)$$
$$AA(FA) > AA(LIO) > AA(Fr) > AA(OD) \approx AA(OD\text{-}Fr) > AA(OD\text{-}LIO)$$
$$FRAP(FA) \approx FRAP(Fr) > FRAP(LIO) > FRAP(OD) > FRAP(OD\text{-}Fr) > FRAP(OD\text{-}LIO)$$
$$ARA(FA) \approx ARA(Fr) \approx ARA(LIO) > ARAODA) \approx ARA(OD\text{-}Fr) \approx ARA(OD\text{-}LIO)$$

Freezing and freezedrying the non-pretreated samples gave the best results; although freezing did not modify ($P>0.05$) TP/TC contents, it reduced TF(10 %) and AA(39 %). TP/TC/TF losses in the freezedried samples ranged between 7 %−17 % while AA dropped 18 %. Marques et al. (2011), working with freezedried guava, mango, papaya and pineapple detected ascorbic acid losses between 3 and 37 %.

Osmotic dehydration reduced total polyphenols, total flavonoids and ascorbic acid, concentrations as well as the ARA and FRAP values by 30–76 %. Nowicka et al. (2015) reported similar results working with sour cherries; Devic et al. (2010) suggested that this behavior might be attributed to chemical degradation and diffusion from the fruit to the solution.

Osmodehydro-freezing arazá diminished TP/FRAP levels by 4–6 % ($P<0.05$), however, no effect was detected in TC/TF/AA/ARA ($P>0.05$); in contrast, osmodehydro-freezedrying produced additional losses in TP (13 %), TF(8 %), TC(28 %), AA(27 %) and FRAP(40 %).

3.2. Treatments effects on the polyphenol composition of arazá

Table 2 and Fig. 1App show the effect of freezing, freezedrying, osmotic dehydration, osmodehydro-freezing, and osmodehydro-freezedrying on the samples polyphenolic profile. In accordance with Soares et al. (2019) and de Araujo et al. (2021), HPLC analysis identified 4 phenolic acids: chlorogenic (CHLO), cinnamic (CIN), gallic (GA) and trans-ferulic (tFER) and 3 flavonoids: eriodictyol-7-O-glucoside (EGlu), Quercetin-3-O-rutinoside (QRut) and rutin mono-hydrate (RH_2O).

Although freezing did not modify ($P>0.05$) CHLO, CIN, tFER, and QRut concentrations, it diminished ($P<0.05$) GA(7 %), EGlu(9 %) and RH_2O(64 %). In the case of lyophylization, the process did not affect ($P>0.05$) CHLO, GA, tFER, QRut and RH_2O levels but reduced ($P<0.05$) CIN (65 %) and EGlu (19 %).

Osmotic dehydration diminished CHLO, GA, tFER, QRut and RH_2O contents 86–96 % ($P<0.05$) while CIN and EGlu were not detected. On the other hand, the process generated a non-identified compound (NID) with a retention time of 5.6 min that may be associated to browning reactions (Koulani et al., 2016).

Although osmodehydro-freezing arazá did not modify ($P>0.05$) CHLO, GA, tFER, QRut or NID ($P>0.05$), the peak corresponding to RH_2O (Fig. 2App) did not appear indicating that, in this case, both treatments interacted negatively. Freezedrying osmodried arazá did not affect CHLO, GA and RH_2O, however, tFER and QRut were not detected and NID increased 3 % ($P<0.05$).

Table 2
Effect of freezing (Fr), freezedrying (LIO), osmotic drying (OD), osmotic dehydro-freezing (OD-Fr) and osmotic dehydro-freezedrying (OD-LIO) on polyphenol composition of arazá.

Treatment	CHLO	CIN	GA	t-FER	EGlu	QRut	RH_2O	NID
FA	45.22±0.52^{a}	110.24±0.54^{a}	160.81±0.46^{a}	73.76±0.49^{a}	155.69±0.28^{a}	83.51±1.18^{a}	119.43±1.65^{a}	–
Fr	42.85±0.37^{a}	107.63±0.84^{a}	149.79±0.67^{b}	69.27±0.54^{a}	128.45±0.48^{c}	81.87±0.83^{a}	42.75±0.69^{b}	–
LIO	48.39±0.82^{a}	38.85±0.22^{b}	156.61±0.38^{ab}	76.59±0.19^{a}	136.24±0.62^{b}	82.64±0.96^{a}	115.22±0.48^{a}	–
OD	4.15±0.14^{b}	–	5.77±0.13^{c}	7.36±0.41^{b}	–	11.74±0.14^{b}	14.41±0.37^{c}	12.96±0.17^{b}
OD-Fr	4.53±0.21^{b}	–	4.23±0.26^{c}	5.17±0.68^{b}	–	10.98±0.74^{b}	–	12.85±0.58^{b}
OD-LIO	4.88±0.17^{b}	–	6.49±0.16^{c}	–	–	–	17.24±0.41^{c}	16.65±0.41^{a}
Rt (min)	9.6	27.5	4.4	17.4	25	3.1	3.5	5.6

FA: untreated; CHLO: Chlorogenic acid (mg (100 g (d.m)$^{-1}$);; CIN: Cinnamic acid (mg (100 g (d.m)$^{-1}$); GA: Galic acid (mg (100 g (d.m)$^{-1}$); tFER: Trans-ferulic acid; mg (100 g (d.m)$^{-1}$); EGlu: Eriodictyol-7-O-glucoside (mg GAE (100 g d.m)$^{-1}$); QRut: Quercetin 3-O-rutinoside (mg R_3H_2O eq (100 g d.m)$^{-1}$); RH_2O: Rutin hydrate form. (mg R_3H_2O eq (100 g d.m)$^{-1}$); NID: unidentified compound ((mg $R(H_2O)_3$ eq(100 g d.m)$^{-1}$); Rt: Retention time. *Superscripts with different letters within the same column indicate significant differences ($P< 0.05$; Tukey).

3.3. Principal component analysis (PCA)

The results from the Principal Component analysis indicated that all responses were included in 2 principal components (PC) explaining 94.0 % of the total variance (PC1(84.8 %)/PC2(9.2 %)). .

The vector distribution in the biplot (Fig. 1) showed that the polyphenolic antioxidants concentrations and the color indicators (ΔE, BI) vectors location in the 1st, 2nd and 4th quadrants was consistent with their opposed behaviors since high antioxidant concentrations and activities were strongly associated with low ΔE, BI and NID values. In addition, the small separation angles detected between TP/TF/TC/AA/ARA/FRAP/CHLO/CIN/GA/ tFER/EGlu/QRut/RH_2O loading vectors (Fig. 1) also implied collinearity. The strength of the relationship between the different properties was quantified using correlation analysis (Table 1App.) considering that only coefficients (R) ≥ 0.6 indicated a good degree of collinearity (Ribeiro et al., 2010). The results showed that the browning index was strongly associated to all 13 antioxidant indicators with R values between −0.6 and −0.96 (Table 1App); on the other hand, ΔE was mainly correlated to GA(−0.62), tFER(−0.81), EGlu (−0.69), R(H_2O) (−0.82), TF(−0.63), AA(−0.72) and ARA(−0.62).

Principal components analysis separated the treatments scores in 3 groups (Fig. 1):

OD/OD-Fr/OD-LIO (G1), Fr (G2) and FA/LIO (G3); their positions in the 1st-3rd, 2nd and 4th quadrants were consistent with the results from Tables 1 and 2. OD/OD-Fr/OD-LIO were characterized by low antioxidant concentrations/activity along with high discoloration and NID levels. On the other hand, groups 2 and 3 positions implied that both treatments had high antioxidant concentration and activity values. However, Fr location pointed out that, in comparison with the untreated and lyophilized samples, frozen arazá samples were more discolored as shown in Table 1.

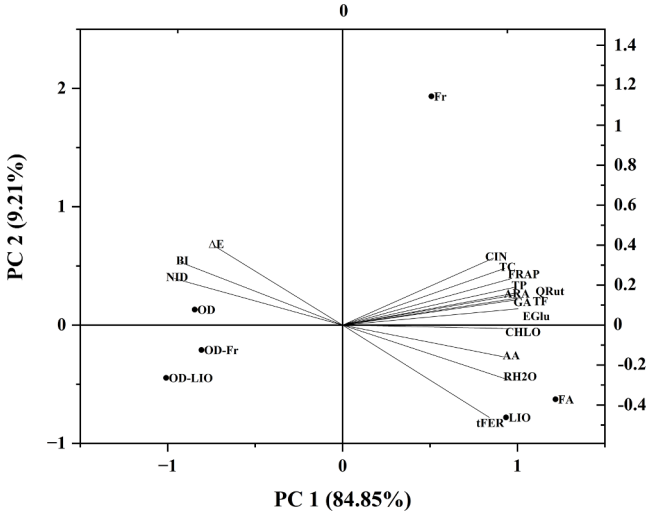

Fig. 1. Loading and score biplots describing the relationship between antioxidant content/ activity, polyphenol composition, degree of discoloration (ΔE), browning index (BI) and their association with the processing treatments. TP; total polyphenols; TF: total flavonoids; TC: total carotenoids; AA: Ascorbic acid; ARA: antiradical activity; FRAP: ferric reducing antioxidant power; CHLO: Chlorogenic acid; CIN: Cinnamic acid; GA: Galic acid; tFER: Trans-ferulic acid; EGlu: Eriodictyol-7-O-glucoside; QRut: Quercetin 3-O-rutinoside; RH_2O: Rutin hydrate form; NID: unidentified compound. Fa: untreated; Fr: freezing; LIO: freezedrying: OD: osmotic drying: OD-Fr: osmodehydro-freezing; OD-LIO: osmodehydro-freezedrying .

3.4. Processing effect on antioxidants bioaccessibilities and activity retention

Table 3 shows the impact of gastric and gastrointestinal digestion on the antioxidant content, activity as well as bioactives bioaccessibility and activity retention of untreated and processed arazá. Comparison with the undigested samples showed that gastric digestion significantly diminished ($P<0.05$) total polyphenols (65–80 %), total flavonoids (36–59 %) and total carotenoids (50–76 %) contents along with the ARA (42–93 %) and FRAP (46–72 %) values. However, those losses were partially compensated during the intestinal step since TP(GI), TF(GI) and TC(GI) recovery levels ranged from 22 to 81 % whereas those corresponding to ARA(GI) and FRAP(GI) were 42–93 %. de Paulo Farias et al. (2021) informed that total flavonoids content and antioxidant activity (DPPH, ABTS, ORAC) values from *Eugenia pyriformis* untreated edible fraction followed a similar behavior during gastrointestinal digestion. In contrast, de Araujo et al. (2021) showed that although the gastric digestion of untreated arazá increased TP/TF without modifying ARA, the gastrointestinal step only improved TF and reduced TP/ARA. The differences between these results and those from the present study may be due to variations in the substrate state since we used whole pieces of peeled pulp while de Araujo et al. (2021) worked with a freezedried ground mix of skin and pulp.

The effects of the different processing treatments on the antioxidant content and activity after gastrointestinal digestion in decreasing order were:

TP(GI)
FA ≈ Fr > LIO > OD ≈ OD-Fr > OD-LIO
TF(GI)
FA≈ Fr > LIO > OD ≈ OD-Fr > OD-LIO
TC(GI)
FA ≥ Fr ≥ LIO > OD ≈ OD-Fr > OD-LIO
ARA(GI)
FA ≈ Fr > LIO > OD ≈ OD-Fr > LIO
FRAP(GI)
FA > Fr > LIO > OD ≥ OD-Fr > OD-LIO

In accordance with our results, Kamiloglu (2019) reported that

Table 3
Effect of freezing (Fr), freezedrying (LIO), osmotic drying (OD) osmotic dehydro-freezing (OD-Fr) and osmotic dehydro-freezedrying (OD-LIO) on the antioxidant content, activity and bioaccessibility (BAC) of arazá following simulated gastric (G) and gastrointestinal digestion (GI).

TRT	TP	TF	TC	ARA	FRAP
FA	383.02 ±2.28^{a}	53.66 ±0.26^{a}	60.92 ±1.75^{a}	507.77 ±4.80^{a}	2656.55 ±19.92^{a}
Fr	379.21 ±4.95^{a}	48.23 ±0.50^{b}	60.71 ±0.72^{a}	503.61 ±2.74^{b}	2605.45 ±17.29^{a}
LIO	357.33 ±2.62^{b}	47.73 ±0.09^{b}	50.42 ±3.07^{b}	494.40 ±2.42^{b}	2343.92 ±5.08^{b}
OD	117.08 ±0.62^{c}	12.84 ±0.92^{c}	35.21 ±0.34^{c}	218.38 ±2.49^{d}	1456.23 ±16.90^{c}
OD-Fr	100.23 ±4.39^{d}	12.60 ±0.12^{c}	35.06 ±0.14^{c}	216.77 ±3.94^{d}	1391.15 ±5.48^{d}
OD-LIO	69.81 ±2.74^{e}	8.51 ±0.37^{d}	20.67 ±0.16^{d}	260.83 ±0.50^{c}	874.87 ±6.53^{e}
TRT	TPG	TFG	TCG	ARAG	FRAPG
FA	113.68 ±0.46^{b}	27.31 ±0.52^{a}	27.59 ±0.90^{a}	294.42 ±4.06^{a}	1406.31 ±7.97^{a}
Fr	121.08 ±0.33^{a}	28.63 ±0.76^{a}	26.93 ±0.40^{a}	286.93 ±6.60^{a}	1395.22 ±23.05^{a}
LIO	71.46 ±0.52^{c}	19.65 ±0.27^{b}	25.28 ±1.48^{a}	226.65 ±1.61^{b}	1040.35 ±3.05^{b}
OD	39.94 ±1.39^{d}	7.80 ±0.18^{c}	8.75 ±0.08^{b}	68.58 ±1.30^{c}	582.49 ±6.90^{c}
OD-Fr	33.92 ±0.19^{e}	8.08 ±0.05^{c}	8.62 ±0.33^{b}	66.91 ±0.22^{c}	544.06 ±4.38^{c}
OD-LIO	24.32 ±0.10^{f}	3.73 ±0.08^{d}	8.32 ±0.14^{b}	18.74 ±0.24^{d}	248.63 ±1.31^{d}
TRT	TPGI	TFGI	TCGI	ARAGI	FRAPGI
FA	266.13 ±2.74^{a}	44.67 ±0.78^{a}	51.87 ±2.10^{a}	428.18 ±7.78^{a}	2087.15 ±11.16^{a}
Fr	267.41 ±5.94^{a}	42.16 ±1.76^{a}	49.81 ±1.20^{ab}	423.40 ±6.57^{a}	2010.36 ±16.14^{b}
LIO	249.10 ±1.05^{b}	32.54 ±0.20^{b}	45.41 ±1.24^{b}	326.29 ±1.63^{b}	1575.23 ±15.65^{c}
OD	88.65 ±1.86^{c}	11.32 ±0.09^{c}	26.26 ±0.25^{c}	180.50 ±3.71^{c}	815.49 ±9.60^{d}
OD-Fr	85.41 ±0.75^{c}	10.34 ±0.29^{c}	25.63 ±0.32^{c}	170.76 ±1.27^{c}	801.81 ±7.67^{ed}
OD-LIO	41.51 ±0.07^{d}	6.89 ±0.11^{d}	15.15 ±0.03^{d}	99.29 ±0.58^{d}	762.40 ±9.76^{e}
TRT	BAC(TPG)	BAC(TFG)	BAC(TCG)	RET (ARAG)	RET(FRAPG)
FA	0.29 ±0.01^{c}	0.51 ±0.01^{c}	0.45 ±0.02^{b}	0.58±0.01^{a}	0.53±0.01^{a}
Fr	0.32 ±0.01^{b}	0.59 ±0.01^{b}	0.44 ±0.01^{b}	0.57±0.01^{a}	0.54±0.01^{a}
LIO	0.20 ±0.01^{d}	0.41 ±0.01^{d}	0.50 ±0.03^{a}	0.46±0.01^{b}	0.44±0.01^{b}
OD	0.34 ±0.01^{a}	0.61 ±0.04^{ab}	0.25 ±0.01^{d}	0.31±0.01^{c}	0.40±0.01^{c}
OD-Fr	0.34 ±0.01^{a}	0.64 ±0.01^{a}	0.25 ±0.01^{d}	0.31±0.01^{c}	0.39±0.01^{c}
OD-LIO	0.35 ±0.01^{a}	0.44 ±0.02^{d}	0.40 ±0.01^{c}	0.07±0.01^{d}	0.28±0.01^{d}
TRT	BAC(TPGI)	BAC (TFGI)	BAC(TCGI)	RET (ARAGI)	RET-(FRAPGI)
FA	0.69 ±0.01^{c}	0.83 ±0.01^{ab}	0.85 ±0.03^{ab}	0.84±0.01^{a}	0.79±0.01^{b}
Fr	0.71 ±0.01^{c}	0.87 ±0.03^{a}	0.82 ±0.02^{b}	0.84±0.01^{a}	0.77±0.01^{b}
LIO	0.70 ±0.01^{c}	0.68 ±0.01^{c}	0.90 ±0.05^{a}	0.66±0.01^{c}	0.67±0.01^{c}
OD	0.76 ±0.01^{b}	0.81 ±0.05^{ab}	0.75 ±0.01^{c}	0.83±0.02^{a}	0.56±0.01^{d}
OD-Fr	0.85 ±0.03^{a}	0.82 ±0.02^{ab}	0.73 ±0.01^{c}	0.79±0.01^{b}	0.58±0.01^{d}
OD-LIO	0.60 ±0.02^{d}	0.81 ±0.03^{ab}	0.73 ±0.01^{c}	0.38±0.01^{d}	0.87±0.01^{a}

TP: Total polyphenols (mg GAE(g FA d.b)$^{-1}$); TF: Total Flavonoids (mg CAT(g FA d.b)$^{-1}$); TC: Total Carotenoids (mg β-carotene eq(g FA d.b)$^{-1}$); ARA: Anti-radical activity (mg GAE(g FA d.b)$^{-1}$); FRAP: Ferric Reducing Antioxidant Power (μM Fe^{+2} (g FA d.b)$^{-1}$). Superscripts with different letters within the same column indicate significant differences ($P< 0.05$; Tukey).

freezing strawberries in a blast freezer did not affect TP/TF or ARA levels after gastrointestinal digestion.

Osmotic dehydration alone or as a pretreatment had a negative impact in the antioxidants contents/activity following gastrointestinal digestion; in those cases, the properties reached their lowest levels. Muñoz-Fariña et al. (2023) identified similar results in osmodried blueberries.

Results also showed that freezing effect did not modify the bioactives concentration or the antioxidant activity of the pretreated samples ($P>0.05$) however, freezedrying the osmodehydrated fruit reduced them between 5 and 55 %. The differences detected in TP/TF/ARA/FRAP values between frozen and freezedried untreated or osmodehydrated arazá may be explained by considering that during the drying step there is a loss of cellular wall integrity combined with an increase of porosity that enhances the incidence of the oxidation and degradation reactions (Betoret et al., 2015).

Principal component analysis showed that all responses were included in 2 principal components (PC) explaining 99.27 % of the total variance (PC1(97.54 %)/PC2(1.73 %)). Although all responses had a high positive relationship with PC1, TC/FRAP/TCGI/ARAGI were negatively correlated to PC2. All loading vectors were closely located (Fig.2) implying a high degree of colinearity (0.93–0.99; Table 2App).

Treatment scores were separated into 4 groups: OD-LIO (G1), LIO (G2), OD/OD-Fr (G3) and FA/Fr (G4), G1and G3 positions in the 1st and 3rd quadrants indicated that these treatments were characterized by low concentration of bioaccessible antioxidants and activity levels. On the other hand, LIO and FA/Fr scores location in the 2nd and 4th quadrants had the opposite behavior reflecting the results from Table 3.

Contrasting OD-LIO vs OD/OD-Fr and LIO vs FA/Fr scores locations in the biplot (Fig. 2) pointed out that freezedrying and osmodehydro-freezedrying reduced the antioxidant content and activity bio-accessible values confirming the results from Table 3.

The effect of the processing methods on the antioxidants bio-accessibilities and activity retention after the gastrointestinal step (Table 3) in decreasing order were:

BACGI(TP)
OD-Fr > OD > Fr ≈ LIO ≈ FA > OD-LIO
BACGI(TF)

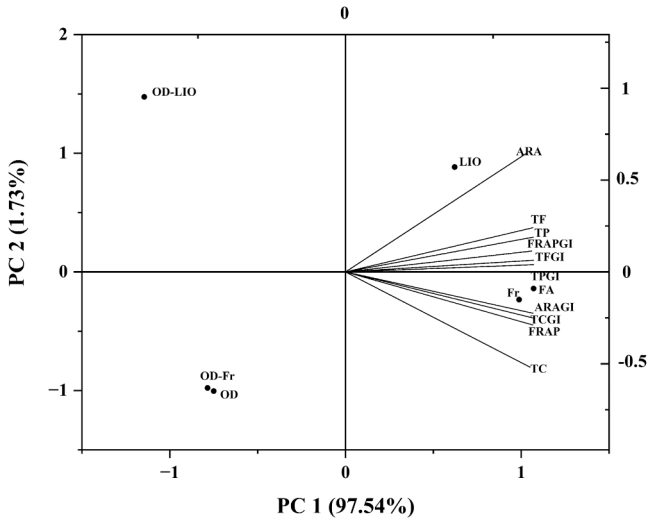

Fig. 2. Loading and score biplots describing the relationship between antioxidant content/ activity and their association with the processing treatments following simulated gastrointestinal digestion. TP; total polyphenols; TF: total flavonoids; TC: total carotenoids; ARA: antiradical activity; FRAP: ferric reducing antioxidant power. Fa: untreated; Fr: freezing; LIO: freezedrying: OD: osmotic drying: OD-Fr: osmodehydro-freezing; OD-LIO: osmodehydro-freezedrying .

Fr ≥ FA ≈ OD-Fr ≈ OD-LIO ≈ OD > LIO
BACGI(TC)
LIO ≥ FA ≈ Fr > OD ≈ OD-Fr ≈ OD-LIO
RETGI(ARA)
FA ≈ Fr ≈ OD > OD-Fr > LIO > OD-LIO
RETGI(FRAP)
OD-LIO > FA ≈ Fr > LIO > OD-Fr ≈ OD

Statistical analysis showed that freezing was the only processing method that did not have a negative effect ($P>0.05$) on BACGI or RETGI values since they were not affected ($P>0.05$) by the treatment. In contrast, freezedrying diminished BACGI(TF) and RETGI(ARA/FRAP) 23 %, 21 % and 15 % ($P<0.05$) respectively.

Although freezing osmodehydrated arazá incremented BACGI(TP) by 12 %, it diminished RETGI(ARA) 5 % ($P<0.05$) without altering BACGI(TF/TC) and RETGI(FRAP) levels ($P>0.05$).

Freezedrying osmodehydrated arazá enhanced RETGI(FRAP) by 55 %, however, the treatment diminished BACGI(TP) and RETGI(ARA) 21 % and 55 % ($P<0.05$) whereas no effects were detected in BACGI(TF/TC) ($P>0.05$).

Comparing the bioaccessibility and activity retention values from the OD-Fr products with those from the untreated arazá showed that osmodehydro-freezing:

(a) improved BACGI(TP) 23 %
(b) did not affect BACGI(TF) and
(c) reduced BACGI(TC), RETGI(ARA) and RETGI(FRAP) 13 %, 6 % and 27 % respectively.

In contrast, osmotic dehydrofreezedrying impact was negative as all properties dropped 3 %−55 % except BACGI(FRAP) that improved 26 %.

4. Conclusions

Freezing followed by freezedrying gave the best results regarding the retention of antioxidant content, activity and bioaccessibility of arazá.

Although the osmotic drying pretreatment increased the freezing rate and reduced the drip loss of osmodehydro-frozen arazá; it also enhanced discoloration and antioxidants concentration/activity losses. However, freezing or freezedrying the OD pretreated arazá partially compensated these effects.

Osmodehydro-freezing arazá improved total polyphenol bioaccessibility and FRAP retention without modifying BACGI(TF), but it also diminished BACGI(TC)/RETGI(ARA). In contrast, osmotic dehydrofreezedrying impact was negative on all the analyzed properties except FRAP that increased significantly.

Results from this work indicate that future studies must determine:

(a) Storage conditions (time, temperature, relative humidity) to obtain products with optimum quality and extended shelf life
(b) A thorough analysis regarding the inclusion of antibrowning agents (ascorbic acid, citric acid, ethylene diamintetracetic acid) and alternative carbohydrates (Glucose. Maltose, Oligofructose, high density Maltodextrin) as well as $CaCl_2$ or Ca lactate to the osmotic solution.

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CRediT authorship contribution statement

Camilo Andrés Reyes-Alvarez: Data curation, Formal analysis, Investigation, Methodology, Visualization, Writing – review & editing, Conceptualization. **María Cecilia Lanari:** Data curation, Formal analysis, Investigation, Methodology, Visualization, Writing – review & editing, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.focha.2023.100496.

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