Research Article

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Upcycling orange by-product: phenolic bioaccessibility and technological features of jelly candies enriched with hydroalcoholic and enzymatic extracts

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Abstract

BACKGROUND: Phenolic extracts are recognized for their health-promoting properties, although some physicochemical characteristics limit their food applications. The present study aimed to valorize orange juice by-products through enzymatic and hydroalcoholic extraction to obtain flavonoid ingredients and to evaluate their application in pectin-based jelly candies as a functional delivery system.

RESULTS: Hydroalcoholic extraction yielded higher total phenolic content and antioxidant capacity, with flavonoids, particularly hesperidin, as the predominant compound. Enzymatic extraction favored the recovery of aglycone forms, especially hesperetin. The incorporation of both extracts (0.1 and 0.2 g kg^{-1}) into jelly candies preserved key techno-functional properties, including moisture (15.34–16.91%) and water activity (0.7285–0.7375), with extract-specific effects on texture (hardness, adhesiveness and stickiness) and color attributes. HPLC analysis confirmed the successful incorporation of characteristic flavonoids into the candy matrix. Despite differences in phenolic content among extracts, the extract type did not significantly affect phenolic bioaccessibility after *in vitro* simulated digestion. All formulations exhibited controlled release and high phenolic bioaccessibility (> 90%), indicating effective protection and delivery of bioactive compounds within the pectin matrix.

CONCLUSION: Orange by-products can be upcycled into flavonoid extracts suitable for incorporation into pectin-based jelly candies without compromising technological quality. The high phenolic bioaccessibility highlights the potential of these candies as functional confectionery products. It supports the use of pectin gels as effective bioactive delivery systems within a circular economy framework.

Supporting information may be found in the online version of this article.

Keywords: antioxidant compounds; bioprocessed extract; flavanones; functional foods; *in vitro* simulated digestion

INTRODUCTION

The orange juice industry generates substantial amounts of by-products during fruit processing. These by-products often contain valuable compounds of nutritional and technological interest, such as polyphenols, dietary fibers and natural pigments. As a result, researchers and industry have intensified efforts to maximize the reuse of by-products, aiming to extract and utilize the beneficial properties still present in these materials.[1] These efforts align with the growing interest in sustainability and the circular economy, particularly in light of the urgent requirements outlined in the Sustainable Development Goals

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(i.e. SDGs), which emphasize the need to repurpose agro-industrial by-products.[2]

Among the compounds found in agro-industrial by-products, phenolic compounds stand out as a group of phytochemicals, known for their antioxidant activity, produced by plants as a defense mechanism against external factors.[1] Flavonoids are the main phenolic compounds in citrus fruits and their by-products, such as peel, albedo and pulp. Although glycosylated flavonoids (e.g. hesperidin, naringin and narirutin) are naturally found in orange fruit and their by-products, aglycone forms appear much less frequently in nature.[3,4] One method to obtain them is through enzymatic hydrolysis, which breaks the sugar bonds of glycosylated compounds.[5,6] Enzymatic processes represent a sustainable alternative for enhancing extraction yields, as they facilitate the targeted degradation of plant cell walls, thereby promoting the release of bioactive compounds.[1] Additionally, they reduce the use of organic solvents, aligning with the principles of green chemistry and thereby minimizing environmental impact.[7]

Because of the well-known antioxidant properties of flavonoids, the functional properties of orange extracts have been extensively reported in the literature. These extracts exhibit a wide range of health effects, including anti-glycation activity, inhibition of digestive enzymes[8] and anti-lipogenic properties,[9,10] as well as antimicrobial and anti-inflammatory effects,[11,12] amongst many others.[13] Given the well-known health benefits of flavonoids, there is growing interest in identifying sustainable sources of these bioactive compounds. Furthermore, incorporating these extracts into food products could simultaneously address nutritional demands at the same time as enhancing the value of orange by-products.

Despite their benefits, polyphenols face challenges in food incorporation because of their low solubility, oxidative degradation and instability in the gastrointestinal tract. Their inherent bitterness also limits the sensory acceptability of consumer-friendly products. Thus, strategies to enhance bioaccessibility and mitigate undesirable flavors are critical for broader food applications.[14] A possible approach to delivering polyphenols is their incorporation into biopolymer-based colloidal delivery systems, particularly pectin gels, one of the most widely used gelling agents in the food industry.[15]

As consumer demand for health-oriented products grows, the food industry has intensified its efforts to develop foods that enhance health. Within this context, phenolic-enriched gummy and jelly candies have emerged as a promising delivery format given their widespread appeal and consumption across all age groups.[15] Phenolic extracts from different sources have been tested in gummy and jelly candies, including pomegranate peel,[16] mango,[17] sage,[18] chickpea leaves[19] and hibiscus.[20] Citrus peel extracts obtained via ultrasound-assisted hydroalcoholic extraction were conjugated with ascorbic acid and incorporated into gelatin-based gummies, confirming the retention of key functional properties of candies.[21] However, most available studies focus on gelatin- or starch-based matrices and emphasize either technological performance or functional properties, rarely addressing both aspects simultaneously, particularly when pectin-based systems and different extraction strategies are considered. Additionally, the effect of digestion on the biological activity (e.g. phenolic release and antioxidant capacity) of candies containing extracts remains underexplored, leaving a critical gap in understanding their potential benefits upon consumption.

Orange by-products represent a valuable, but underutilized, source for the recovery of bioactive compounds with established health benefits. When obtained through efficient and scalable processes, the by-products of the orange juice agroindustry can be transformed into high-value products for the pharmaceutical and food sectors. Incorporating orange by-product extracts into jelly candies could thus enhance functional properties and appeal at the same time as supporting a circular economy. Although several studies have explored the extraction of phenolics or their incorporation into confectionery products, most investigations focus on isolated aspects, such as extraction efficiency, technological performance or functional properties alone.

In this context, the originality of the present study lies in its integrated approach, which combines (i) the recovery of phenolic compounds from orange by-products using two distinct extraction strategies (enzymatic and hydroalcoholic); (ii) their application in pectin-based gummy candies, an alternative matrix that remains scarcely explored compared to other gelling systems; and (iii) the comprehensive characterization of both extracts and final products by HPLC. In addition, key technological properties and the bioaccessibility of phenolic compounds were simultaneously evaluated through *in vitro* simulated digestion, providing a holistic assessment that links processing, product quality, and potential biological relevance.

MATERIALS AND METHODS

Obtaining the orange by-product

The orange by-product was donated by Cutrale® (Araraquara, São Paulo, Brazil). The material was obtained in the form of pellets, which are produced after treating the solid and liquid residues left over from the extraction of orange juice (*Citrus sinensis*). These residues include peels, seeds and pulp, and the varieties used in this process are Pera Rio, Lima, Baianinha/Baia and Valencia. Once acquired, the pellet was maintained in a frozen state. For particle size reduction, a commercial blender (Model BL.2.202, 1500 W, 220 V; MC Marchesoni, Santana de Parnaíba, São Paulo, Brazil) was used. The content was sieved (1.68 mm, 10 mesh; Bertel Metallurgical Industries, Caieiras, São Paulo, Brazil) and the orange by-product was stored at freezing temperature for future applications.

Polyphenol extraction from orange by-product

The extraction of polyphenols was carried out using either enzymatic aqueous extraction or traditional solvent extraction methods. For the enzymatic method extraction, a combined system of cellulase, pectinase and tannase enzymes containing 10 U of each was employed in the orange by-product.[5] Cellulase (Celluclast 1.5 L; Novozymes, Copenhagen, Denmark) and pectinase (Pectinex Ultra SP; Novozymes) were obtained commercially. The tannase was produced through solid-state fermentation using *Paecilomyces variotii* (CBMAI 1157).[22] Enzyme activities were assessed spectrophotometrically before application.[23-25] For the enzymatic extraction, 2 g of orange by-product was added to 25 mL of sodium acetate buffer (20 mM, pH 5.0) and incubated under shaking (300 rpm) for 6 h at 40 °C.[26]

The reaction was stopped in an ice bath for 30 min, then the extract was centrifuged (Allegra X-30R Centrifuge; Beckman Coulter, Brea, CA, USA) for 30 min at 4255 × *g* and 4 °C. The supernatant was collected, and the orange enzymatic extract (OEE) was stored frozen for up to 72 h before being freeze-dried. It was then

maintained at a freezing temperature for up to 2 weeks before further analysis and use.

The hydroalcoholic extraction method was also applied.[9,27] Initially, 10 g of the orange by-product were mixed with 250 mL of a water-ethanol solution (1:1, v/v). The content was sonicated (15 min at 30 °C) in a bath-type ultrasound (UNIQUE UltraSonic Cleaner, model USC-1800A, 40 kHz, 135 W; Indaiatuba, São Paulo, Brazil) and stirred in an orbital shaker (model 496; Thermo Fisher Scientific, Waltham, MA, USA) for 15 min at 25 °C and 200 rpm. Subsequently, the extract was filtered on Whatman filter paper (No. 1) (Cytiva, Wilmington, DE, USA), and the solution was concentrated on a rotary evaporator (50 °C) to remove ethanol. The orange hydroalcoholic extract (OHE) was stored frozen for up to 72 h before freeze-drying and kept at freezing temperature for up to 2 weeks before further analysis and use.

The extraction yield was calculated in triplicate by dividing the mass of the freeze-dried extract (g) by the initial mass of the orange byproduct (g). The results were expressed as a percentage. Both extracts were evaluated for total phenolic compounds content, phenolic profile and antioxidant capacity.

Preparation of jelly candies enriched with phenolic extracts

The ingredient quantities for producing jelly candies were determined based on standard recommendations for candy production,[28] ensuring the final product achieved the desired texture and consistency. The complete formulation of jelly candies, along with the raw material specifications, is provided in the Supporting information (Table S1).

First, a mixture of high-methoxyl (HM) pectin and 18.4% of the total sucrose specified in the formulation was hydrated in hot water under continuous stirring in a digital mixer (713D model; Fisatom, Perdizes, São Paulo, Brazil) at 550 rpm for 15 min. Next, the remaining sucrose and glucose syrup were added, manually homogenized and cooked on a conventional stove until the mixture reached approximately 80 °Brix, measured with a digital refractometer (Q767BD Abbe; Quimis, Diadema, São Paulo, Brazil). Based on preliminary tests, the cooking time was 10 min and 30 s. After this period, the heating stopped, and OHE or OEE was added at 0.1 and 0.2 g kg^{-1}. Subsequently, the mixture was manually homogenized, and the citric acid solution was incorporated. Then, the content was spread into a square silicone mold (length × width × height: 20 × 20 × 4 cm) and stored at 25 °C for 24 h. After gelation, the mass was cut into square pieces (2 × 2 cm) using a mold, and the candies were evaluated as described below. Jelly candies (JC) containing OHE extract at 0.1 and 0.2 g kg^{-1} were coded as JC-OHE1 and JC-OHE2, respectively, whereas those containing OEE were coded as JC-OEE1 and JC-OEE2. Candies without extracts were prepared as a control.

Physicochemical characterization of the jelly candies

The moisture content was assessed using the Karl Fischer volumetric method with a titrator (901 Titrando model; Metrohm, Herisau, Switzerland), using a methanol: formamide solution (2:1 v/v) as solvent.[29] The pH (Ultra Basic UB-10 pH/mV meter; Denver Instrument, Denver, CO, USA) and water activity (AquaLab Series 4TEV; Meter Group, Pullman, WA, USA) were also measured.[30]

The color was assessed using the CIEL*a*b* system with a colorimeter (Model CR-400; Konica Minolta Sensing Inc., Osaka, Japan). The hue angle (hue°) and chromaticity (C*) were calculated.[31] The instrumental texture was measured on a texturometer (TA. XTplusC Texture Analyser; Stable Micro Systems, Godalming, UK) from 10 replicates.[32] The parameters of the candies' hardness, adhesiveness and stickiness were determined.[33]

In vitro simulated digestion of jelly candies

This step followed the INFOGEST protocol.[34] Enzyme activities were assessed based on the supplementary information provided in the guidelines. For simulated digestion of jelly candies, 5 g of each sample was weighed into a light-protected Falcon tube and mixed with 4 mL of simulated salivary fluid (pH 7.0). The mixture was stirred magnetically at 37 °C until it was completely dissolved. The solution was then supplemented with 1 mL of 0.3 M calcium chloride, amylase (75 U mL^{-1} final concentration) and water, to reach a final volume of 5 mL of simulated salivary fluid, followed by a 2-min incubation at 37 °C in a shaking water bath (150 rpm). To initiate the gastric phase, simulated gastric fluid, 0.3 M calcium chloride and pepsin solution (2000 U mL^{-1} final concentration) were added, and the pH was adjusted to 3.0. Samples underwent 2 h of shaking incubation under these conditions. For the intestinal phase, simulated intestinal fluid, pancreatin (100 U mL^{-1} final concentration), bile (5 mg mL^{-1}) and additional calcium chloride were introduced, followed by a final 2 h incubation under identical conditions. Post-digestion aliquots were collected for subsequent determination of the phenolic profile and antioxidant capacity, as described in the analytical methods below.

Identification of phenolic compounds using HPLC

Sample preparation for injection

The freeze-dried orange extracts and the pectin used in the formulation of candies were dissolved in 70% HPLC-grade methanol using vortex-assisted dissolution (2 min) followed by centrifugation (2716 × g for 15 min at 25 °C). The supernatant was filtered through a hydrophilized polytetrafluoroethylene membrane filter (H-PTFE, 0.45 µm). For the extraction of phenolic compounds from the candy matrix before digestion, samples were first dissolved in ultrapure water (1:1 w/v) under magnetic stirring, then extracted with 7 mL of HPLC-grade methanol (vortexed for 2 min). After centrifugation (2716 × g for 15 min at 25 °C), the supernatants were membrane-filtered (0.45 µm hydrophilic PTFE) for analysis. The methodology proposed by Oliveira Júnior and Cunha[35] was applied to the digested pectin candy. The intestinal digesta was centrifuged (10 864 × g for 15 min at 4 °C) and the supernatant was then mixed 1:1 (v/v) with HPLC-grade methanol, yielding a final 50% (v/v) intestinal digesta solution for analysis. This solution was vortexed (2 min) and recentrifuged (2716 × g for 15 min at 25 °C), followed by membrane filtration (0.45 µm hydrophilic PTFE) for subsequent analysis of the main phenolic compounds.

Analysis of major phenolics and bioaccessibility calculation

The profile of phenolic compounds in the samples was determined using HPLC, with a diode array detector (DAD; Dionex UltiMate DAD-3000; Thermo Fisher Scientific) and a C18 column (Acclaim 120 column, Dionex, 3 µm, 4.6 × 150 mm) at 30 °C. Analyte separation was achieved using the mobile phases: A (99.9:0.1 v/v water/formic acid) and B (99.9:0.1 v/v methanol/formic acid) in a linear gradient at a flow rate of 0.6 mL min^{-1}: 90% A (0–5 min), 20% A (5–80 min), 90% A (80–85 min) and 90% A (85–95 min).[36] The phenolic compounds were identified using standards (Extrasynthese, Lyon, France) and compared with their UV-visible spectra (Model UV-M51; BEL Engineering, Monza, Italy) and retention time. Target compounds were detected in triplicate, with quantification based on standard curves.

The bioaccessibility of each compound was calculated as the percentage of phenolic compounds released from the food matrix into the soluble fraction after simulated digestion, relative to the total content in the original, undigested sample, according to:

$$\text{Bioaccessibility index}\,(\%) = \frac{\text{Soluble fraction}}{\text{Initial content in the food matrix}} \times 100$$

Total polyphenols content determination

Total phenolic compounds (TPC) of freeze-dried orange extracts were measured using the Folin–Ciocalteu method,[37] with a gallic acid standard curve. The freeze-dried orange extracts were diluted in water at different concentrations for testing. Absorbance was measured in a 96-well plate at 725 nm in a plate reader (ST-360; KHB, Shanghai, China).

In vitro antioxidant capacity assay

The 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical-scavenging activity of the samples was measured. Extracts were diluted to 70% methanol and centrifuged (2716 × *g* for 5 min at 25 °C). Undigested candies were homogenized in an aqueous solution and then adjusted to 70% methanol for identical centrifugation. For digested candy samples, 300 µL of intestinal digesta-centrifuged (10 864 × *g* for 5 min at 4 °C) supernatant was mixed with 700 µL of absolute methanol, vortexed, and then analyzed. The decolorizing process was measured,[38] in a 96-well plate at 520 nm (FLUOstar Optima; BMG Labtech, Ortenberg, Germany).

For the oxygen radical absorbance capacity (ORAC) assay,[39,40] the extracts and undigested candies were diluted in sodium phosphate buffer (pH 7.4) at varying concentrations. The digested candies were centrifuged (10 864 × *g* for 5 min at 4 °C) and then tested without further dilution. Fluorescence measurements were recorded in a 96-well dark plate at 37 °C, in a fluorimeter (FLUOstar Optima; BMG Labtech).

DPPH and ORAC assays were measured using a Trolox standard curve.

Statistical analysis

All measurements were carried out as independent experiments in triplicate. Data are expressed as the mean ± SD. Analysis of variance (ANOVA) was conducted, with one-way ANOVA used to characterize the extract and the physicochemical properties of the candies, and two-way ANOVA applied to the digested samples. Tukey's test was employed to identify significant differences among multiple means, whereas a *t*-test was used for comparisons between two samples. $P < 0.05$ was considered statistically significant. All analyses were carried out using Prism, version 8.0.1 (GraphPad Software Inc., San Diego, CA, USA).

RESULTS AND DISCUSSION

Phenolic profile and *in vitro* bioactivity of the orange extracts

HPLC identified the phenolic compounds based on their retention times and respective spectra. Figure 1 illustrates the chromatographic profiles of each extract, highlighting the main compounds and their corresponding spectra. The chromatographic profiles of the OHE and OEE revealed flavonoids as the major class of phenolic compounds in both extracts. In the OHE, two main peaks were identified, corresponding to narirutin and hesperidin. Minor chromatographic peaks were also detected, indicating the presence of hesperetin and tangeretin at lower concentrations. In the OEE, one major peak was identified as hesperetin, whereas two minor peaks were attributed to hesperidin and naringenin.

The content of each compound was also quantified using its corresponding standard calibration curves (Table 1).

In the present study, the hesperidin levels in OHE were higher than those reported in extracts obtained by supercritical carbon dioxide from orange pomace[41] and by ultrasound-assisted aqueous extraction of orange peels.[42] Conversely, higher concentrations of hesperidin and narirutin have already been reported in navel orange peel, depending on the extraction process applied. For hesperidin, ethanolic extraction performed better than aqueous extraction, whereas ethyl acetate extraction yielded higher narirutin recovery.[43] Flavonoids, specifically the glycosylated ones such as hesperidin and narirutin, are the main group of phenolic compounds found in citrus fruits, such as oranges, lemons and tangerines. The amount can vary depending on the fruit type, the part used for extraction and the fruit's ripeness.[44] Furthermore, the extraction method employed directly influences the recovery rate of these compounds. Regarding aglycones, this class of polyphenols is less common in nature. One way to obtain them is by using enzymes during the extraction process. This occurs because the enzymatic application acts on plant tissues, hydrolyzing the sugar moieties bound to the phenolic structure.[1,5,45]

Our results indicate that the applied enzymatic treatment bioconverted the glycosylated compounds into their aglycone forms because the OEE showed only traces of hesperidin and no narirutin was detected. Furthermore, enzymatic treatment increased the extraction yield compared to the hydroalcoholic process; however, the total phenolic content of the OHE was three times higher ($P < 0.05$) than that of the OEE (Table 2). Similarly, the ability of the samples to neutralize free radicals, as evaluated by the DPPH method, was found to be three times greater in the OHE compared to the OEE. Additionally, the ORAC method revealed an eightfold higher antioxidant activity for the OHE (Table 2). It is essential to note that the ORAC method evaluates the capacity to protect against oxidation mediated by peroxyl radicals, making it more sensitive to antioxidant bioactivity in biological systems.[40]

It is well established that polyphenols are strongly associated with antioxidant capacity.[46,47] In the present study, the TPC content of OHE was higher than that reported for orange by-product extract,[27] comparable to red orange extract, but lower than that of blonde[21] and sour orange[48] extracts. Additionally, as expected, the OEE contains aglycone compounds, such as naringenin and hesperetin, which are similar to those found in other enzymatically bio-transformed citrus extracts.[5,45,49,50] These aglycone flavanones have exhibited higher biological potential compared to their glycosylated forms in the literature.[6,26,49] However, in the present study, their concentration does not appear to be sufficient to demonstrate a higher antioxidant potential of this extract compared to the OHE. An increase in extraction yield did not lead to a corresponding increase in the total phenolic content and antioxidant capacity of the OEE. This may suggest the recovery of other non-phenolic components in the extract. Ethanol is a more effective solvent for extracting a wider range of phenolic compounds, particularly flavonoids.[7,48] Although no alcoholic extraction method was applied after the aqueous-enzyme extraction, this could have resulted in a lower recovery of phenolic compounds from the by-product matrix, as observed previously for white grape pomace[51] and navel orange peel.[43]

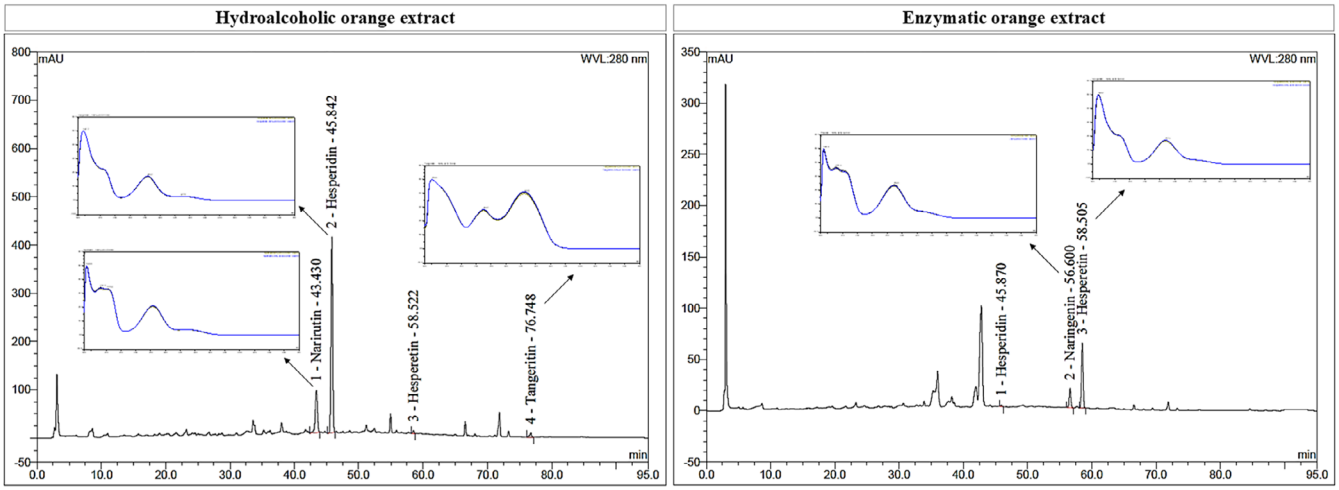

Figure 1. HPLC-DAD chromatograms and the respective UV spectra of each compound in orange extracts.

Table 1. Profile of major phenolic compounds identified by liquid chromatography in extracts derived from orange by-product

Compound	Class	Limit of detection (ug of compound)	OHE (g kg^{-1})	OEE (g kg^{-1})
Narirutin	Glycosylated flavanone	0.031–20.0	2.999 ± 0.155	nd.
Hesperidin	Glycosylated flavanone	0.031–4.0	12.250 ± 0.605	tr.
Naringenin	Aglycone flavanone	0.031–2.0	nd.	0.086 ± 0.002
Hesperetin	Aglycone flavanone	0.016–2.0	0.087 ± 0.005[b]	0.287 ± 0.005[a]
Tangeretin	Methoxyflavone	0.031–2.0	0.236 ± 0.012	nd.

Means and standard deviations with different letters in the same row differ via a *t*-test ($P < 0.05$). OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract; nd., not detected; tr., traces. Narirutin, 280 nm; hesperidin, 280 nm; naringenin, 280 nm; hesperetin, 280 nm; tangeretin, 260 nm.

Table 2. Yield of extraction, total phenolic compounds and antioxidant capacity *in vitro* of the extract obtained from the orange by-product

Parameter	OHE	OEE	*P*-value
Yield (%)	21.98 ± 0.86 b	52.05 ± 1.45 a	< 0.0001
TPC (g GAE kg^{-1})	23.04 ± 0.84 a	6.28 ± 0.36 b	< 0.0001
DPPH (mmol Trolox kg^{-1})	81.39 ± 6.83 a	28.56 ± 2.09 b	0.0002
ORAC (mmol Trolox kg^{-1})	925.22 ± 106.46 a	111.98 ± 46.30 b	< 0.0001

All values are expressed on a lyophilized extract basis. Different lowercase letters in the same row indicate a difference via a *t*-test ($P < 0.05$). GAE, gallic acid equivalent; TPC, total phenolic compounds; OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract.

These results reinforce the influence of the solvent type and the technique used on the efficiency of citrus flavonoid extraction. Still, further research is needed to explore and optimize combined water, enzyme and ethanol extraction methods for scaling up, aiming to produce high-yield, high-bioactivity extracts. This also involves applying emerging technologies to improve efficiency and sustainability. Among emerging technologies, hydrodynamic cavitation has recently gained attention as a scalable, water-based technology for citrus by-products. Beyond extraction efficiency, this technique can induce unique structural modifications, such as the formation of stable conjugated phytocomplexes of pectin, flavonoids and volatile compounds, which have been linked to improved bioaccessibility and enhanced pharmacological activity.[52,53] Additionally, techniques such as ultrasound and microwave-assisted extraction, supercritical fluid extraction and pressurized liquid extraction have also demonstrated promising results in enhancing phenolic recovery at the same time as reducing solvent consumption and processing time.[54,55] Their application, whether alone or in combination with enzymatic strategies, may represent viable options for industrial-scale processes, producing bioactive-rich extracts for incorporation into food products, thereby further strengthening the circular economy perspective.

At the same time, the phenolic profile and bioactivity of by-product extracts depend not only on the extraction method, but also on the sample's characteristics, such as form, variety, plant part used, origin, growing conditions and other factors.[47,56] The orange by-product used in this study was in pellet form, produced by mechanical compression to reduce volume and facilitate commercialization as an energy source or animal feed.[57,58] The lower

phenolic content observed in both extracts, compared to previous studies from our group,[3,5,45] may be related to the nature of the by-product, as the pelletizing and drying processes can reduce the availability of phenolic compounds.[48]

Nevertheless, the results demonstrate that, even from an underutilized by-product, it is possible to obtain, through appropriate processes, an economically viable source of flavonoids, such as hesperidin, a compound widely used in the pharmaceutical industry and of high added commercial value.[59] Furthermore, the results provide insights to guide the agroindustry in adopting more efficient waste treatment practices, aiming to harness its potential as a source of bioactive compounds or other value-added raw materials.

Physicochemical characterization of jelly candies

For the physicochemical characterization of the candies, pH, moisture, water activity and colorimetric profile were analyzed (Table 3). Regarding pH, only JC-OEE2 exhibited a noticeable increase (11.70%) compared to the control candy. This sample differed from others, except in comparison to JC-OHE1. However, incorporating orange extracts did not alter the moisture and water activity of the samples ($P > 0.05$), which presented results within a range suitable for gummy and jelly candies.[60] For HM pectin, pH plays a crucial role in gel formation and texture. Research shows that the optimal pH range for effective pectin gelation is between 2.8 and 3.6, depending on the degree of esterification.[28]

However, the effects of incorporating extracts into candies are influenced by multiple factors, including the type of extract, its concentration, and the candy formulation. For example, the pH level of the pectin-gelatin candies decreased as the pectin content increased after the addition of *Garcinia atroviridis* extract.[61] On the other hand, similar to the present study, the addition of chickpea leaf extract increased the pH of pectin candies due to the extract's alkaline profile. Conversely, the moisture content of the candies added with this extract increased slightly compared to the control.[19] Nevertheless, pectin candies added with hibiscus extract (*Hibiscus sabdariffa* L.) showed no differences in moisture content,[20] demonstrating how different extracts particularly affect specific physicochemical properties.

Another key factor to consider when formulating food is color, which is linked to consumer acceptance. The color coordinates of the candies were measured, and the inclusion of 0.1 g kg^{-1} OEE did not alter the samples' luminosity (L^*) compared to those without extracts. However, in the remaining samples, this parameter decreased by an average of 8.44% compared to the control candy. The candies added with the extracts exhibited positive results (> 0) for the a^* and b^* coordinates, which are associated with the color of the components. In terms of hue°, the addition of extracts at all concentrations increased this parameter. However, OHE exhibited a more than 90% increase in hue°, compared to the control candy, which had the lowest value. Similarly, the chromaticity of the samples, also referred to as color intensity or saturation, showed positive effects with the addition of all extracts (Table 3).

The increase in hue° and chromaticity gradually shifted the sample's color toward more positive values, enhancing its orange tonality. By contrast, luminosity is associated with the brightness/opaqueness of samples and can naturally decrease when extract is added, which contains suspended insoluble particles.[62] Similar results were observed in previous studies upon the addition of extracts to the candies' formulation.[18,20,61]

The visual appearance of the control and orange extract-added candies is shown in Fig. 2. The control sample was translucent and uniform, with no visible particles. The samples containing the OHE showed a progressively more intense coloration as the extract concentration increased, indicating good incorporation and homogeneity, thereby eliminating the need for colorants in the formulation. On the other hand, in the candies containing the OEE, suspended particles are visible in both concentrations, suggesting that the extract was not homogeneously incorporated into the candy matrix. Some hypotheses can be proposed based on these observations. Although OEE showed a higher extraction yield, its lower total phenolic content and antioxidant capacity (Phenolic profile and *in vitro* bioactivity of the orange extracts section) suggest that the appearance of the candies may be due to a higher load of suspended solids. This effect could be associated with the presence of hydrophobic and less soluble compounds in OEE, not necessarily phenolics because hesperetin is more soluble than hesperidin,[43] but likely fragments derived from the enzymatic extraction medium. Enzymatic treatments target specific polysaccharides in the cell wall, breaking down complex polymers such as cellulose, hemicellulose and pectin. However, they often do not achieve complete solubilization, which can result in partial hydrolysis of other components, including proteins and lipids.[63,64] Additionally, the particles observed in the candies containing OEE may be associated with the flocculation behavior of the HM pectin used in the formulation. At acidic pH and low concentrations, HM pectin can form larger complexes through electrostatic bridging, resulting in increased turbidity and reduced system stability. This phenomenon has been reported in protein–pectin model systems, where induced flocculation led to a more turbid medium.[65] Thus, the interaction between pectin and constituents of the OEE may have promoted aggregate formation, which could explain the less homogeneous appearance of the candies compared to those produced with

Table 3. Physicochemical and colorimetric properties of pectin-based candies enriched with orange by-product-derived extracts

Sample	pH	Moisture (%)	Water activity (w_a)	L^*	a^*	b^*	*Hue*°	Chroma
Control JC	3.47 ± 0.11 bc	16.05 ± 0.38 a	0.7330 ± 0.0001 a	40.33 ± 1.47 a	0.10 ± 0.11 c	3.68 ± 0.62 c	−1.40 ± 1.70 e	3.69 ± 0.62 c
JC-OHE1	3.70 ± 0.03 ab	15.34 ± 0.58 a	0.7294 ± 0.0028 a	37.83 ± 0.90 b	3.20 ± 0.20 a	7.84 ± 0.48 a	22.21 ± 1.76 b	8.47 ± 0.45 a
JC-OHE2	3.64 ± 0.05 b	16.91 ± 0.77 a	0.7285 ± 0.0036 a	35.84 ± 1.58 c	3.55 ± 0.93 a	4.15 ± 1.42 c	41.08 ± 2.77 a	5.47 ± 1.67 b
JC-OEE1	3.34 ± 0.16 c	15.88 ± 1.01 a	0.7375 ± 0.0003 a	40.07 ± 1.84 a	0.46 ± 0.13 bc	6.08 ± 1.95 a	4.35 ± 0.57 d	6.10 ± 1.95 b
JC-OEE2	3.93 ± 0.01 a	15.99 ± 0.27 a	0.7332 ± 0.0069 a	37.11 ± 1.25 bc	0.90 ± 0.28 b	4.77 ± 0.97 bc	10.43 ± 1.42 c	4.86 ± 1.00 bc

Different lowercase letters in the same column differ with respect to Tukey's test ($P < 0.05$). JC, jelly candy; OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract; JC-OHE1 and JC-OHE2, jelly candies containing OHE at 0.1 and 0.2 g kg^{-1}, respectively; JC-OEE1 and JC-OEE2, jelly candies containing OEE at 0.1 and 0.2 g kg^{-1}, respectively.

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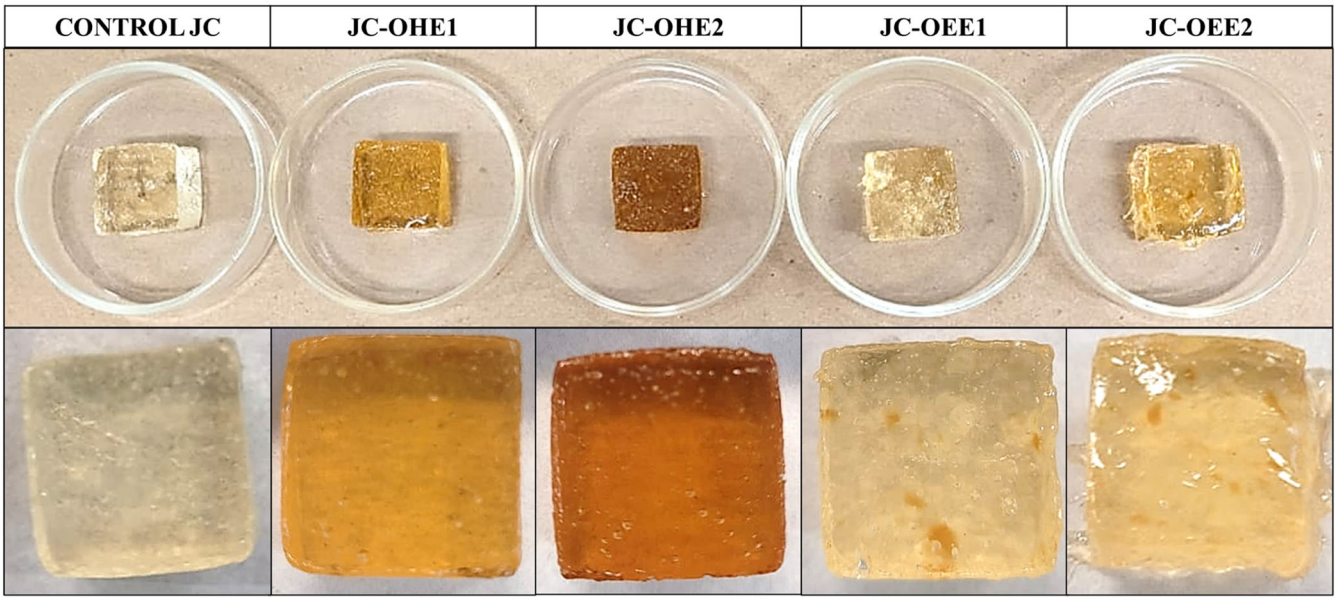

Figure 2. Visual appearance of candies added with orange by-product extracts. JC, jelly candy; OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract; JC-OHE1 and JC-OHE2, jelly candies containing OHE at 0.1 and 0.2 g kg^{-1}, respectively; JC-OEE1 and JC-OEE2, jelly candies containing OEE at 0.1 and 0.2 g kg^{-1}, respectively.

OHE. All these phenomena may generate visual microheterogeneities, as observed in the current study. However, to confirm these mechanisms, complementary investigations such as microscopy, particle size distribution analysis, and rheological analysis are recommended for future studies.

Texture analysis is also important for obtaining insights into the effects of adding extracts to foods. Therefore, hardness, adhesiveness, and stickiness were evaluated to determine the texture profile of the candies, and the results are presented in Fig. 3. The addition of OHE at both concentrations (0.1 and 0.2 g kg^{-1}) maintained the hardness of the candies similar to the control. The average hardness for these samples remained at 141.76 ± 4.20 gf. By contrast, the incorporation of OEE significantly reduced the hardness in a concentration-dependent manner, with reductions of 43.78% at JC-OEE1 (76.99 ± 2.97 gf) and 71.92% at JC-OEE2 (38.45 ± 4.39 gf) compared to the control. Regarding adhesiveness, OEE formulations resulted in less negative values, indicating reduced adhesiveness compared to the control. This means that candies with OEE required less force for the probe to detach from the sample surface.

The hardness of a material is defined as the force required to compress it between two surfaces, such as molars, resulting in deformation. Adhesiveness and stickiness in food can be characterized by the force needed to remove it from a surface, such as teeth, after chewing.[33] Accordingly, the adhesiveness and stickiness results complement the hardness findings, indicating that candies with added OEE were stickier, that is, softer.

The texture profile of gummy and jelly candies can be affected by several factors, including the addition of plant-based extracts, even when using a standard formulation. Different ingredients can interact with the pectin matrix and other gelling agents, changing properties such as firmness, adhesiveness and stickiness. In a study of jellies made from a base recipe of apple puree, pectin and agar-agar with added strawberry, raspberry or blueberry, the addition of plant extracts caused variations in texture. Although no significant differences in firmness were observed between the samples, those containing strawberries with anise and those containing strawberries with mint showed differences in stickiness.[33] Another study assessed the impact of adding orange juice to starch candies made with and without added sugar. The diet candies, formulated with a polyol sweetener, had lower hardness than both the standard candy (without juice) and the candy containing sugar. Additionally, the diet sample exhibited lower stickiness and adhesiveness.[66] Conversely, adding hibiscus extract to pectin candies[20] and propolis extract to gummy candies[67] had minimal effect on the texture properties of the samples.

These comparisons support our findings, indicating that plant-derived extracts may affect candy texture differently depending on their composition. However, the texture profile of jelly candies is influenced not only by the addition of plant extracts, but also by the structural features of the gel matrix. HM pectins (such as those used in jelly candy manufacturing) form stable gels in acidic conditions when combined with solutes such as sugar, and their gelling efficiency can be influenced by factors such as pH, co-solutes and ionic composition (e.g. sodium and potassium ions).[68] For the candies containing OEE, especially at higher concentrations, some hypotheses may explain the observed decrease in gel strength: (i) the observed pH exceeded the optimal pectin gelation range (typically 2.8–3.5), potentially compromising gel structure development; (ii) residues from the enzymatic extraction medium (e.g. salts or solutes) may interfere with gelation as a result of their higher concentration compared to the hydroalcoholic extraction medium; and (iii) hydrophilic compounds in the OEE can compete with pectin for water molecules, disrupting gel network formation. These factors collectively weaken gel strength, resulting in candies that fail to solidify properly. By contrast, despite its higher pH compared to the control, OHE maintained the candies' textural properties, indicating compatibility with the pectin matrix and minimal interference with gelation.

Therefore, the results show that it is possible to produce pectin candies with phenolic extract from orange by-products that retain

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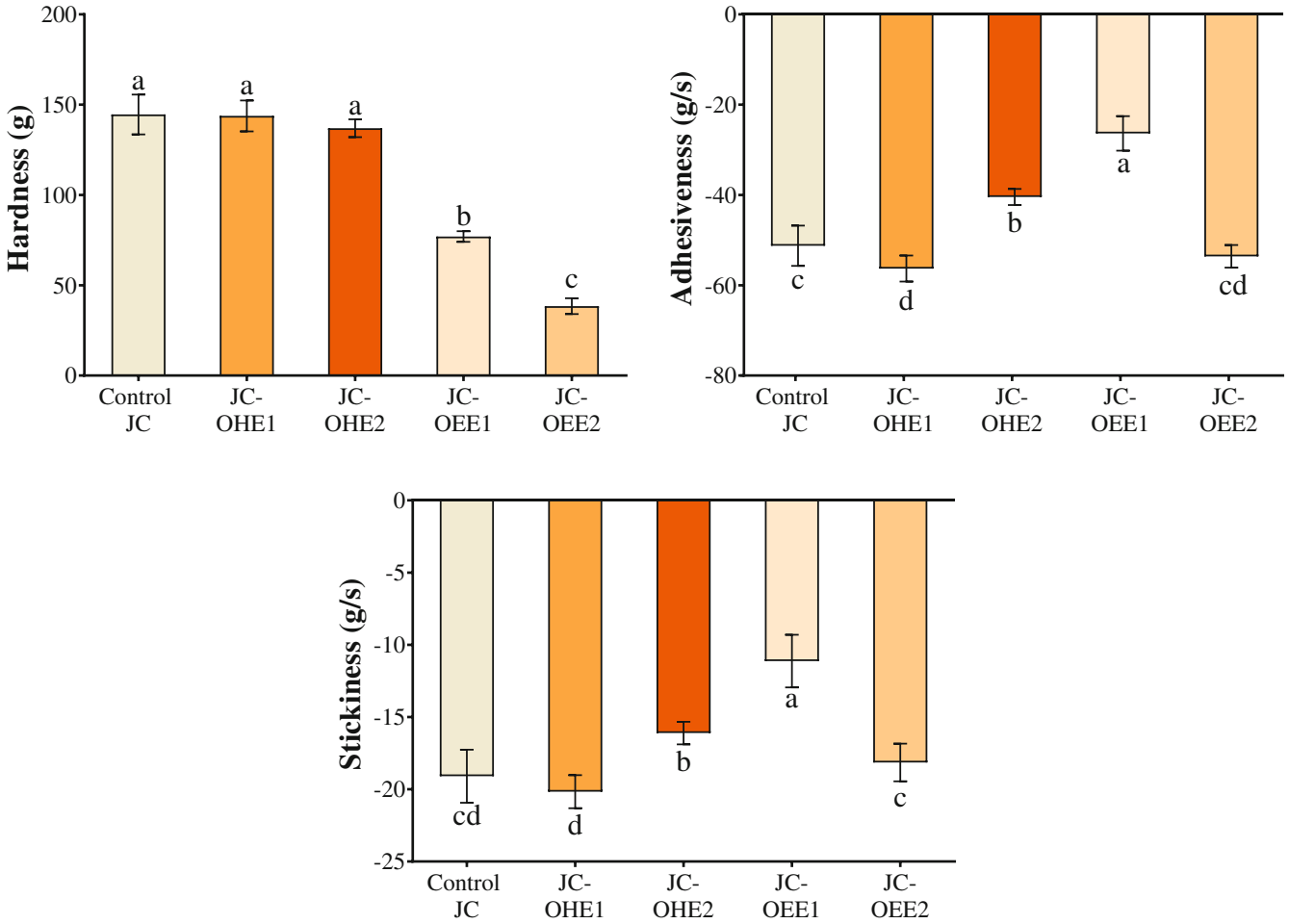

Figure 3. Texture profile obtained from the compression test of candies prepared with orange by-product extracts. Bars with the same letter do not differ in the Tukey test ($P < 0.05$). JC, jelly candy; OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract; JC-OHE1 and JC-OHE2, jelly candies containing OHE at 0.1 and 0.2 g kg^{-1}, respectively; JC-OEE1 and JC-OEE2, jelly candies containing OEE at 0.1 and 0.2 g kg^{-1}, respectively.

the expected technological characteristics. Incorporating 0.1 and 0.2 g kg^{-1} OHE effectively preserves key physicochemical properties of jelly candies, making it a viable option for industrial applications. Notably, the OHE outperformed the OEE in color intensity, likely as a result of its higher phenolic content. The addition of the extract also enhanced the candies' visual appeal, imparting richer, reddish tones. However, maintaining optimal pH levels during production and storage remains critical to ensure pectin gel stability and product quality. Therefore, texture optimization can be achieved in the formulation by adjusting the pH by incorporating an acid.

Bioaccessibility of phenolic compounds in candies

To characterize changes in phenolic composition during digestion, the phenolic profiles of candy samples (pre- and post-digestion) were analyzed by liquid chromatography. The bioaccessibility of each compound was calculated, as detailed in Table 4, where only compounds that could be quantified before simulated digestion are included. Additional compounds (diosmin and tangeritin) were identified in trace amounts in undigested candies, as provided in the Supporting information (Table S2).

The compounds narirutin, hesperidin and hesperetin were quantified in candies containing OHE. After digestion of the candies with extract concentrations of 0.1 and 0.2 g kg^{-1}, the narirutin and hesperidin content were statistically similar. In the candies containing the OEE, both hesperidin and hesperetin were quantified, along with naringenin. However, after digesting the candies, no naringenin was detected in the samples. The JC-OEE2 preserved the hesperetin content after digestion, and no reduction was observed in this compound. Although no extracts were added to the control jelly candy, hesperidin levels were quantified in this sample. After digestion, the content of this compound in the candy without extracts increased by an average of 36%. The presence of hesperidin in the control candy is likely associated with the pectin used in its formulation, given that oranges are a common source of pectin extraction. This interpretation is supported by the pectin chromatogram (see Supporting information, Fig. S1) and the similar hesperidin content in the control sample and the candies containing the OEE because this extract displayed only trace amounts of hesperidin in the chromatographic analysis. Additionally, the type of extract did not affect the bioaccessibility of the identified compounds. Despite differences in phenolic content among the candy formulations, compound bioaccessibility exceeded 90%. Notably, hesperidin

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Table 4. Content and bioaccessibility index (%) of phenolic compounds of jelly candies during an *in vitro* digestion process

Sample	Undigested (mg kg^{-1} candy)	Digested (mg kg^{-1} candy)	Bioaccessibility index (%)
Control JC			
Hesperidin	17.911 ± 0.555 b	23.563 ± 0.010 a	132
JC-OHE1			
Narirutin	29.001 ± 0.264 a	26.948 ± 0.010 a	93
Hesperidin	147.895 ± 8.325 a	136.53 ± 8.23 a	92
Hesperetin	0.867 ± 0.017	nd.	–
JC-OHE2			
Narirutin	56.488 ± 0.25 4a	54.185 ± 0.494 a	96
Hesperidin	257.411 ± 0.793 a	253.828 ± 12.572 a	99
Hesperetin	1.742 ± 0.084	nd.	–
JC-OEE1			
Hesperidin	16.890 ± 0.131 b	24.688 ± 0.282 a	146
Naringenin	1.364 ± 0.005	nd.	–
Hesperetin	4.466 ± 0.017	tr.	–
JC-OEE2			
Hesperidin	16.723 ± 0.393 b	24.563 ± 0.631 a	147
Naringenin	2.628 ± 0.016	nd.	–
Hesperetin	8.675 ± 0.046 a	8.179 ± 0.273 a	94

Different lowercase letters in the same column differ, for the same compound and jelly sample, differ via a *t*-test ($P < 0.05$). JC, jelly candy; OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract; JC-OHE1 and JC-OHE2, jelly candies containing OHE at 0.1 and 0.2 g kg^{-1}, respectively; JC-OEE1 and JC-OEE2, jelly candies containing OEE at 0.1 and 0.2 g kg^{-1}, respectively; nd., not detected; tr., traces.

in control candies and OEE-candy samples showed even higher values. Bioaccessibility rates exceeding 100% indicate that the compounds were released from the food matrix and/or metabolized from more complex phenolic compounds.[69]

Phenolic bioaccessibility and bioavailability studies are crucial for assessing the delivery efficiency of compounds and ensuring that the added bioactive compound is accessible to absorption and delivers the desired health benefits. A comprehensive overview of this topic was provided, highlighting findings from *in vivo* studies demonstrating rapid absorption of aglycones and their metabolites, detectable in plasma as early as 10 min after consumption.[6] Glycosylated flavanones, on the other hand, require hydrolysis to be absorbed and have therefore been identified on average 2–3 h after consumption. It is estimated that approximately 97% of the hesperidin consumed is converted to hesperetin by the lactase phloridzin hydrolase enzyme. Furthermore, a substantial amount of the flavanone aglycones is metabolized by the gut microbiota and may offer health benefits. For this reason, if the catabolites of the intestinal microbiota's metabolism are included, the total absorption of flavonoids can be much higher than expected, considering the undigested food.[6]

Another study evaluating different phenolics under simulated digestion found that, among flavanones, narirutin and hesperidin exhibited lower recovery rates than naringenin and hesperetin, suggesting that glycosylation may reduce the bioaccessibility of flavanones.[70] However, the polyphenol bioaccessibility varies widely depending on the food matrix and processing methods[71] because polyphenols can interact with various non-digestible or slowly digestible molecules, such as dietary fiber, gelatin and pectin, thereby increasing their resistance to gastric degradation. Overall, hesperidin has exhibited relatively high bioaccessibility among polyphenols because it remains more stable under adverse digestion conditions. In bread enriched with mandarin extract, the identified phenolic compounds were completely depleted after the duodenal phase, except for hesperidin, which exhibited bioaccessibility values exceeding 100%.[72]

Different foods containing citrus polyphenols exhibit unique phenolic bioaccessibility profiles, which are influenced by the food matrix, processing methods and the specific phenolic compounds present. Improving food formulation and processing is essential to enhance the health benefits of citrus phenolics. The extraction of polyphenols for application in food systems aims to concentrate these bioactive compounds because using the co-product in its whole form, such as flour, sometimes requires higher levels of addition and could negatively impact the product's sensory appeal. For example, in cookies made with sorghum flour enriched with hog plum (*Spondias mombin*) peel, samples with peel concentrations of 200 g kg^{-1} showed lower sensory acceptance despite having higher fiber and phenolic content.[73] Similarly, in 'whole foods' prepared with different concentrations of orange pomace, soybean bran, and wheat bran, the highest acceptance was observed in formulations containing up to 100 g kg^{-1} orange pomace, 800 g kg^{-1} soybean bran and 100 g kg^{-1} wheat bran.[74] Additionally, citrus pomace increased the fiber content of extruded snacks and lowered the glycemic index. However, extruded formulations with more than 50 g kg^{-1} pomace were not well accepted in sensory evaluation.[75]

Despite this, it is essential to emphasize that, following the principles of zero waste and the circular economy, utilizing the material that remains after phenolic compound extraction is also crucial. In addition to phenolics, the orange by-product is a valuable source of other components with nutritional and technological value, including fiber (e.g. pectin, cellulose, and hemicellulose)

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and essential oils.[76] Therefore, the orange residue remaining after phenolic extraction remains a valuable resource because it can be further exploited to recover other compounds or directed to alternative applications.[77] For example, it may serve as a functional ingredient in the development of new products. A study revealed that fruit by-products, such as buriti and orange, combined with fermented dairy matrices and probiotics, have beneficial synergistic effects on the intestinal microbiota, the production of short-chain fatty acids and bone health in an animal model. Specifically, adding orange pomace increased femoral magnesium and serum calcium levels, enhanced intestinal permeability and promoted the growth of *Lactobacillus* and *Muribaculaceae*. Formulations containing orange pomace also elevated fecal acetate and propionate levels. Meanwhile, consuming probiotic products increased the abundance of *Lachnospiraceae* NK4A136.[78] Another animal model study demonstrated that adding citrus peels (*Citrus sinensis* and *Citrus maxima*) powder to ice cream increased plasma high-density lipoprotein-cholesterol levels and enhanced the antioxidant status in the liver and heart of rats. Additionally, it lowered the glycemic index, total cholesterol, triglycerides and low-density lipoprotein compared to commercial ice creams and the control group.[79]

The observations show the potential of by-products, including citrus, in developing different foods, promoting both sustainable use and functional benefits. In jelly candies, pectin seems not only to facilitate the controlled release of phenolic compounds from the orange extracts, but also to liberate some compounds attached to the matrix during digestion as a result of the action of digestive enzymes. This may explain the high bioaccessibility rate (> 90%) of the phenolic compounds in jelly candies, indicating their classification as bioactive substances with relatively high overall bioaccessibility.[80] These findings validate orange by-products as viable sources of bioaccessible phytochemicals for industrial upcycling, at the same time as confirming the jelly formulation as an effective delivery system for bioactive-added confectionery. Nevertheless, future studies should investigate how the addition of whole orange bagasse affects bioactive parameters and impacts the technological and sensory characteristics of various products, including jelly candies, at the same time as focusing on optimizing formulations to balance functionality, technological performance and consumer acceptance.

Antioxidant capacity of candies

In addition to technological parameters and phenolic bioaccessibility, antioxidant capacity is also an important feature to evaluate in the development of new products. Therefore, the highest antioxidant capacity in the ORAC test was observed in the JC-OHE2 (12 680.23 ± 2926.35 mmol Trolox equivalent kg^{-1}), followed by JC-OHE1 (10 429.66 ± 2867.48 mmol Trolox equivalent kg^{-1}). The JC-OEE1 and JC-OEE2 samples exhibited a statistically similar antioxidant capacity, with a mean of 5208.36 ± 302.98 mmol Trolox equivalent kg^{-1}, two times lower than the OHE-added candy but higher than the control jelly candy. A similar trend was observed in the antioxidant capacity measured by the DPPH method. The addition of OEE at different concentrations showed no statistically significant differences among the samples containing these extracts, with an average of 840.65 ± 24.22 mmol Trolox equivalent kg^{-1}. By contrast, samples with the OHE demonstrated an antioxidant capacity five times higher than the OEE and twelve times higher than candies without added extract (Fig. 4). This result was expected because the OHE had a higher level of phenolics and antioxidant capacity, and, as mentioned before, both are correlated.

Both extracts improved the antioxidant capacity of the candies; however, this effect was only evident in undigested candies. Despite the HPLC profile indicating higher phenolic compound concentrations in the candies enriched with extracts, no difference in the *in vitro* antioxidant capacity of the samples was observed after digestion using either of the two methods evaluated.

The ORAC test measures a sample's antioxidant capacity by neutralizing peroxyl free radicals generated *in vitro*. In this method, a fluorescent compound (fluorescein) is oxidized by free radicals, causing a loss of fluorescence over time. Antioxidants in the sample slow down this loss of fluorescence, and the area under the curve (AUC) is used to quantify antioxidant activity. In our experiment, no difference in AUC values was identified between samples with and without extract, making it impossible to infer the contribution of phenolics to antioxidant capacity using this assay. In the DPPH method, the ability of a sample to donate electrons or hydrogen to reduce the stable free radical DPPH• is measured. The radical has an intense purple color, which is reduced to light yellow as antioxidants react with it. The clearer the medium after the reaction, the higher the antioxidant capacity of the

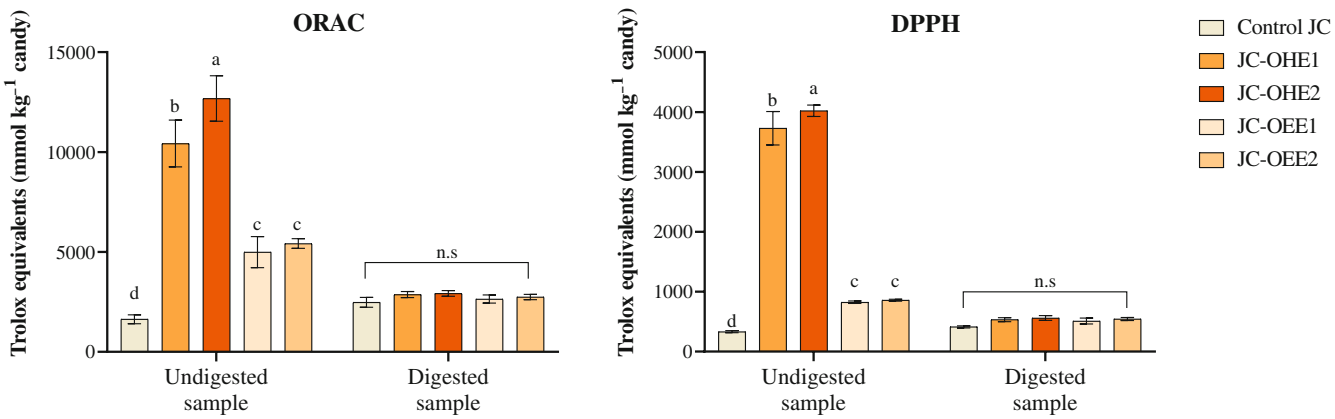

Figure 4. *In vitro* antioxidant capacity of candies containing orange by-product extract before and after simulated digestion. Bars with different letters within the same phase indicate significant differences according to the Tukey test ($P < 0.05$). JC, jelly candy; OHE, orange hydroalcoholic extract; OEE, orange enzymatic extract; JC-OHE1 and JC-OHE2, jelly candies containing OHE at 0.1 and 0.2 g kg^{-1}, respectively; JC-OEE1 and JC-OEE2, jelly candies containing OEE at 0.1 and 0.2 g kg^{-1}, respectively.

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compound. In our experiment, the reaction medium for the DPPH assay of the digested samples was visually clear after adding the reagent and the reaction time. However, even after centrifuging the digested samples, a slight turbidity was observed in the reaction medium, possibly because of micelles, suspended particles, or insoluble compounds from the simulated digestion that were not completely removed during centrifugation.

Depending on how some constituents interact in the sample, these interfering compounds can mask the antioxidant effect of phenolics, leading to inaccurate measurements of antioxidant capacity.[81] Some studies have reported findings consistent with our observations, demonstrating that incorporating phenolic extracts enhances the antioxidant capacity of candy.[18,21] Additionally, adding concentrated orange juice enhanced the antioxidant capacity of starch candies containing sugar or polyols in their formulations.[66] However, these valuable investigations focused specifically on pre-digestion analyses, making it challenging to understand how digestive processes modify both phenolic profiles and antioxidant capacity. By contrast, another study demonstrated that, although total phenolic content remained stable post-digestion, antioxidant capacity significantly declined in pomegranate-enriched gelatin candies.[16] These results suggest possible changes in the bioaccessibility or stability of bioactive compounds. However, direct comparisons between studies are challenging because of differences in candy formulations. Additionally, different classes of phenolic compounds may exhibit distinct behaviors during digestion because they can be affected by (i) specific interactions with food matrix components and (ii) variable susceptibility to simulated digestion conditions.[70]

It is essential to note that, in the present study, during simulated digestion, the end of the intestinal phase contained 125 g of candy L^{-1} of digestive fluid. In the ORAC and DPPH tests, the digested sample was pipetted without any further dilution, and no extraction was conducted. The antioxidant effects of polyphenols in candies are more challenging to detect by *in vitro* methods at low concentrations of extracts.[18] Moreover, phenolic compounds are sensitive to environmental conditions, such as pH variations during simulated digestion. These changes can lead to degradation or structural modification, reducing their biological activity *in vitro*.[69,82] On the other hand, HPLC is a more sensitive and precise analytical method, capable of identifying phenolic compounds even at low concentrations.[7] This could explain why some compounds detected by HPLC in intestinal digesta samples may have been underestimated in antioxidant capacity assays because these assays often rely on less-specific chemical reactions that can be influenced by matrix interference or compound instability.

CONCLUSIONS

The present study demonstrates that underutilized orange by-products can be valorized into flavonoid extracts, providing an economically viable source of bioactive compounds for functional food applications. Although the OHE outperformed the OEE in total phenolic content, antioxidant capacity, and candy color and texture, both extracts achieved over 90% phenolic bioaccessibility. These results highlight the effectiveness of the pectin-based jelly matrix as a controlled-release system for phenolic compounds, regardless of the extraction method. These findings offer the agroindustry valuable insights to adopt sustainable management practices, transforming orange by-products into value-added ingredients. However, one limitation of using agro-industrial by-products, especially from the orange industry, is the variability in the chemical composition of the peels and albedo. Throughout the year, the orange varieties used for juice production change with crop availability, directly affecting quality and bioactive compound content. This variability is not only a limitation of the present study, but also reflects the industrial reality, in which byproducts already contain mixed peels from different varieties after processing. Therefore, it is crucial that upcycling be carried out using robust, efficient and scalable methods. Utilization strategies must account for this heterogeneity and maximize the by-product's added value at the same time as ensuring effective availability.

Future work should focus on sensory evaluation to assess consumer acceptance and preference, as well as on industrial-scale extraction approaches that integrate emerging green technologies to balance cost-effectiveness with bioactivity retention. Additionally, the application of complementary antioxidant assays based on different reaction mechanisms could be explored to provide a broader and more robust assessment of the antioxidant potential of candy matrices. Furthermore, valorizing the extraction residue, which still contains nutritional and technological compounds, should be considered a complementary approach to strengthen the circular economy framework. Ultimately, this work advances upcycling principles by incorporating food by-products into functional confectionery with a promising bioactive delivery system.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

DISCLOSURE

All authors have read and agreed to the version of the manuscript submitted for publication.

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CONFLICTS OF INTEREST

The authors declare that they have no conflicts of interest.

SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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