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Physicochemical, microbiological, sensory and amino acid characterization of jams made with common orange peel (Citrus sinensis) in the municipality of Valledupar, Cesar (Colombia)

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Abstract

In Colombia, orange peel is an abundant and underutilized agro-industrial waste, despite its fiber, pectin and bioactive compound content. The present work evaluates whether this residue, transformed into jam, can be used as an ingredient in foods of mass consumption. Osmotically dehydrated common orange peel (Citrus sinensis) jams were characterized physicochemically, microbiologically and sensorially, their amino acid profile was determined by HPLC and their acceptability as an ingredient in stuffed bread and natural yogurt was evaluated. A 3×2 factorial design was applied with three concentrations of sucrose syrup (20, 40 and 60 % m/m) acidified with 2 % citric acid and two forced air drying temperatures (40 and 60 °C; 6 h). The husks were cut into 1×2 cm pieces and blanched at 73 °C ± 2 °C for 60 min. Data were analyzed by ANOVA and Tukey's test ($p < 0.05$) in SPSS v.21. Jams had a pH of 3.12 to 3.13; ash from 4.82 to 4.86%; crude fiber from 45.19 to 46.12 %; humidity of 10.4%; fat of 1.72%; titratable acidity of 0.77%; soluble solids of 59 °Brix and crude protein of 5.06 to 5.70%. The T6 treatment (60% sucrose, dried at 60°C) had the best organoleptic characteristics and was selected for subsequent analysis. The HPLC amino acid profile, with orthophthaldehyde (OPA) pre-column derivatization and fluorescence detection, showed five quantifiable amino acids: arginine (0.28%), cysteine (0.10%), lysine (0.20%), methionine (0.10%), and tryptophan (0.06%); three of them are essential for human beings. The microbiological quality met the limits of NTC 5247. Applied as an ingredient (10% m/m in stuffed bread and 8% m/m in yogurt), the jam reached acceptance levels of 68% and 72%, respectively, in hedonic tests with 50 untrained panelists. The results indicate that osmotically dehydrated orange peel is a harmless and nutritionally useful matrix, with industrial potential as an ingredient with fiber and essential amino acid content.

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1. Introduction

The Colombian citrus industry generates increasing amounts of by-products, mainly peels, alveoli and seeds. Oranges represent about 75% of national citrus production (Asohofrucol, 2014). Depending on the variety and degree of ripeness, the peel represents between 15% and 30% of the weight of the fruit (Rincón et al., 2005; Mahato et al., 2018). In most of the juice extraction stations in the municipality of Valledupar (Cesar), this material is discarded directly into the environment, with the consequent cost of handling, attraction of vectors and emissions associated with its decomposition.

The peel of Citrus sinensis is, however, a nutritionally rich matrix. It contains total dietary fiber between 45 and 65%, pectin between 20 and 30%, phenolic compounds such as flavanones (hesperidin and naringin), carotenoids and essential oils with limonene as the main component (Rincón et al., 2005; Badui, 2006; Sharma et al., 2017). Several recent reviews point to these extracts as antioxidant, lipid-lowering, and antimicrobial agents with nutraceutical and dietary applications (Mahato et al., 2018; Suri et al., 2022; Maqbool et al., 2023). Its transformation into food for human consumption, whether as jams, flours, pectins or functional ingredients, is part of the zero-waste technologies, and represents a concrete way for small and medium-sized agri-food companies in the Colombian Caribbean to add value to the orange chain.

Osmotic dehydration is a unitary operation with low energy consumption in which plant matter loses water and gains solutes when immersed in a hypertonic solution (Alzamora et al., 1989; Welti-Chanes & Vergara-Balderas, 1997). Applied to citrus peels, it produces a material of intermediate moisture and high microbiological stability, preserving the fibrous matrix and the bioactive profile (M'hiri et al., 2017). The concentration of the syrup, the temperature and the immersion time are the determining variables of the process (Barbosa-Cánovas et al., 2003).

Despite the regional availability of the resource, there were no published studies that characterized in an integrated manner the physicochemical, microbiological, sensory and amino acid behavior of orange peel jams produced with raw material from the department of Cesar, nor that evaluated their behavior as an ingredient in finished foods. The undergraduate work of Morales González (2012), developed at the Universidad Popular del Cesar, generated a body of experimental data that fills this gap and constitutes the empirical basis of this article. The purpose of the manuscript is to systematize, analyze and make available to the regional scientific community these results. The specific objectives were: (i) to evaluate the effect of three concentrations of sucrose syrup (20, 40 and 60 % m/m) and two drying temperatures (40 and 60 °C) on the physicochemical, microbiological and sensory quality of jams made with Citrus sinensis peel; (ii) to determine the amino acid profile of the optimal HPLC treatment; and (iii) to evaluate the applicability of jam as an ingredient in stuffed bread and natural sweetened yogurt, through hedonic tests with consumers.

The choice of bread and yogurt responds to practical criteria. Both are products of daily consumption in the Colombian Caribbean, but they belong to technologically contrasting categories: a dry bread and a creamy-acidic dairy product. If jam performs well in both vehicles, its versatility as an ingredient and, therefore, its industrial potential is demonstrated. This logic of "matrix-tested ingredient" differentiates the present work from a study that is limited to characterizing the residue.

2. Materials and methods

2.1. Origin of data and ethical considerations

The primary experimental data were obtained by Morales González (2012) in the framework of his undergraduate work at the Agroindustrial Engineering Program of the Universidad Popular del Cesar (Valledupar, Cesar, Colombia). This article constitutes an analytical systematization of this body of data, cited as a foundational reference. The panelists' participation in the sensory test was voluntary, anonymous, and unpaid. Before their intervention, each panelist received information about the ingredients of the product (wheat flour, dairy products and possible traces of citrus fruits) so that they could abstain in case of food allergy or intolerance. No personally identifiable data was collected.

2.2. Raw material

Common orange peel (Citrus sinensis) collected from juice extraction stalls in the municipality of Valledupar was used. Fruit in a state of commercial maturity was selected and pieces with stains, molds or mechanical damage were discarded. The husks were washed with pressurized water and, subsequently, with chlorinated water at 50 ppm; They were drained on mesh for 15 min before cutting.

2.3. Reagents and equipment

Commercial food-grade sucrose and USP anhydrous citric acid (Carlo Erba) were used for the osmotic solution. The physicochemical analyses followed the official methods of the AOAC (1990). A tray dryer with forced air (Memmert UFE 500) was used for drying. The amino acid analysis was performed in a high-performance liquid chromatograph (HPLC) with pre-column derivatization with orthophthaldehyde (OPA) and fluorescence detection (excitation 340 nm; emission 450 nm), in the laboratory of the Research Center for the Development of Engineering (CIDI) of the Popular University of Cesar.

2.4. Experimental design

A completely randomized design was applied with a 3×2 factorial arrangement: three sucrose concentrations (20, 40 and 60 % m/m) and two drying temperatures (40 °C and 60 °C). Each combination was performed in triplicate, for a total of 18 experimental units. Treatments were identified as T1 to T6 as indicated in Table 1.

Table 1. Definition of the treatments of the factorial design 3×2.

Treatment	Sucrose (% m/m)	Citric acid (%)	Drying temperature (°C)	Drying Time (h)	Replicas
S1	20	2	40	6	3
S2	20	2	60	6	3
S3	40	2	40	6	3
S4	40	2	60	6	3
S5	60	2	40	6	3
S6	60	2	60	6	3

2.5. Processing of the jam

From the shells, rectangular pieces of 1×2 cm were obtained by means of longitudinal cuts in the axial direction. Debittering was carried out by blanching in water at 73 °C ± 2 °C for 60 min, in a husk:water ratio of 1:5 (m/v), in order to reduce the content of naringin, the main flavanone responsible for the bitter taste. Sucrose syrups of 20, 40 and 60 % m/m, acidified with citric acid at 2 % m/m, were then prepared and boiled for 2 min. The peels were immersed in a fruit:1.5 (m/m) ratio and kept in intermittent agitation at room temperature for 72 h. This immersion time, prolonged compared to the osmotic processes typical of soft-fleshed fruits (from 4 to 24 h), responds to the structural nature of the citrus peel: the flavedo and the albedo have a porous matrix with high intracellular air retention, as well as a cell wall rich in pectins that slow down the transfer of solutes

(Barbosa-Cánovas et al., 2003; M'hiri et al., 2017). At the end of osmotic dehydration, the syrup was drained and the pieces were immersed for a few seconds in water at 95–100 °C to remove the surface syrup. Finally, they were dried by forced air at 40 or 60 °C for 6 h, the time selected to reach final humidity ≤ 12 % without excessive browning, according to the ranges reported by Welti-Chanes and Vergara-Balderas (1997). The product was packed in vacuum-sealed polyethylene laminated bags and stored at room temperature, in darkness and on ventilated shelves. The complete operational sequence is presented in Figure 1.

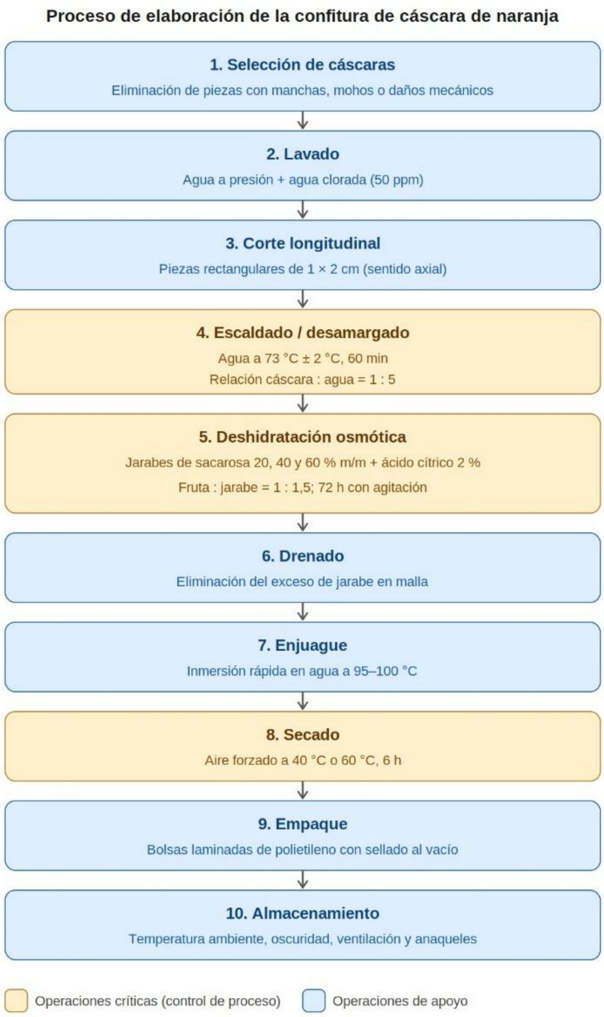

Figure 1. Flow chart of the process of making the common orange peel jam (Citrus sinensis). Yellow boxes indicate critical operations with process control: blanching, osmotic dehydration, and drying. In the original language Spanish.

2.6. Physicochemical analysis

The following parameters were determined for each triplicate treatment, according to AOAC (1990): moisture by gravimetry (105 °C, 24 h), ash by incineration (550 °C, 6 h), fat by Soxhlet extraction with petroleum ether, crude fiber by acid-alkaline hydrolysis, crude protein by Kjeldahl (factor 6.25), soluble solids by refractometry (°Brix), titratable acidity expressed as a percentage of citric acid and pH by potentiometry. Weight loss during osmotic dehydration and drying was calculated using equations (1) and (2), based on Alzamora (1997), Welti-Chanes and Vergara-Balderas (1997) and Barbosa-Cánovas et al. (2003):

$$\Delta W = (W_0 - Wt) / W_0 \quad (1)$$

$$\Delta Ww = (W_0 \times X_0 - Wt \times Xt) / W_0 \quad (2)$$

where ΔW is the weight variation, ΔWw the moisture loss fraction, W_0 and Wt the husk masses at time zero and time t, and X_0 and Xt the initial and final water contents, respectively.

2.7. Microbiological analysis

The following counts were carried out on the optimal treatment in accordance with Resolution 14712/1984 of the Ministry of Health of Colombia and the Colombian Technical Standard NTC 5247: mesophilic aerobes, molds and yeasts, total and fecal coliforms, Escherichia coli and detection of Salmonella spp. in 25 g of sample.

2.8. Amino acid profiling by HPLC

On the treatment that presented the best organoleptic characteristics, the amino acid profile was quantified by high-performance liquid chromatography (HPLC) with pre-column derivatization with orthophthaldehyde (OPA) and fluorescence detection (excitation 340 nm; emission 450 nm). Samples (approximately 100 mg) were hydrolyzed with HCl 6 N at 110 °C for 24 h in an inert atmosphere (N_2); the hydrolysate was evaporated to dryness, reconstituted with buffer borate pH 9.5 and derivatized with OPA/2-mercaptoethanol immediately before injection. Separation was performed in a C18 reversed-phase column with sodium acetate (pH 7.2)/methanol/acetonitrile elution gradient. Amino acids were identified by comparing retention times to pure standards and quantified by peak area against external calibration curves. It should be noted that the method used, based on derivatization with OPA, does not detect proline or hydroxyproline as they lack a primary amino group; Cysteine was reported after previous conversion to cystic acid. The five amino acids quantified correspond to those whose concentration was above the method's limit of quantification, which explains the absence of the remaining amino acids in Table 5.

2.9. Application of jam as an ingredient and sensory analysis

2.9.1. Formulation of enriched products

The jam of the optimal treatment was incorporated as an ingredient in two matrices of high consumption in the Colombian Caribbean region: a dry bread product (stuffed bread) and a creamy dairy product (natural sweetened yogurt). The incorporation percentages correspond to levels established in preliminary sensory tolerance tests; the base formulations were constructed from standard formulas in the literature (Badui, 2006) and will require optimization in subsequent studies. A standard commercial formulation of wheat flour, water, instant yeast, salt, sugar, milk powder and butter was used for the stuffed bread; the jam pieces were incorporated as a filling in a proportion of approximately 10% m/m over the weight of the sourdough and baked at 180 °C for 20 min. For the natural sweetened yogurt, pasteurized whole milk inoculated with commercial cultures of Lactobacillus delbrueckii subsp. bulgaricus and Streptococcus thermophilus, incubated at 42 °C to pH 4.5; jam pieces were added as chunks at a rate of about 8 % m/m to yoghurt already chilled to 4 °C.

2.9.2. Hedonic test

The sensory acceptability of both enriched products was assessed using a five-point verbal hedonic test: 1 = I don't like it, 2 = I don't like it much, 3 = I don't care about it, 4 = I like it, and 5 = I like it a lot. Each product was evaluated by 50 untrained panelists (age 18-55, both sexes), recruited from the university community, in individual booths with white light and between 10:00 and 12:00 h. The samples were served in white containers coded with random three-digit numbers. Acceptance was considered to be the sum of the categories I like and I like it a lot. The panelists gave informed verbal consent and declared that they did not have allergies to the ingredients used.

2.10. Statistical analysis

The physicochemical data were subjected to two-way analysis of variance (ANOVA). When significant differences were detected, Tukey's multiple mean comparison test was applied with a 95% confidence level ($p < 0.05$). Calculations were performed in SPSS v.21 (IBM Corp.). The results are expressed as an average of the triplicates; cell-specific standard deviations were not available for all variables at the time of writing (a limitation acknowledged in the Results section).

3. Results and discussion

3.1. Proximal physicochemical composition

Table 2 presents the proximal analysis of the jams for the six treatments. The values are within the ranges described by Rincón et al. (2005) for orange peel flours and by Priego-Nerio (2007) for dehydrated citrus peels. The high content of crude fibre (45.19 to 46.12 %) and ash (4.82 to 4.86 %) confirms the husk as an important source of minerals and dietary fibre to enrich bread and dairy matrices.

Table 2. Proximal composition of Citrus sinensis peel jam for the six treatments of the 3×2 factorial design.

Parameter	S1	S2	S3	S4	S5	S6
pH	3.12 to	3.12 to	3.13 to	3.13 to	3.12 to	3.13 to
Humidity (%)	10.4 to	10.4 to	10.4 to	10.4 to	10.4 to	10.4 to
Ashes (%)	4.82 to	4.84 to	4.85 to	4.85 to	4.84 to	4.86 to
Crude fibre (%)	45.19 to	45.40 to	45.71 to	45.90 to	46.02 A	46.12 to
Fat (%)	1.72 to	1.72 to	1.72 to	1.72 to	1.72 to	1.72 to
Acidity (% citrus)	0.77 to	0.77 to	0.77 to	0.77 to	0.77 to	0.77 to
Soluble solids (°Brix)	59 to	59 to	59 to	59 to	59 to	59 to
Crude protein (%)	5.06 to	5.30 to	5.40 ab	5.55 ab	5.65 b	5.70 b

Different letters in the same row indicate statistically significant differences ($p < 0.05$) according to Tukey's test. Values expressed in g/100 g of sample as an average of triplicates. Standard deviations per cell are not included because raw data from the original statistical analysis were not available for this systematization. Source: Authors' elaboration based on Morales González (2012).

Ashes

No significant differences were observed between treatments for ash content ($p > 0.05$), which is consistent with the fact that no sodium chloride or other minerals were incorporated into the syrup. The average value (4.85 %) coincides with that reported by Rincón et al. (2005) for common orange peel flour and is within the limits of NTC 5247 for fruit confectionery.

Crude Fiber

The crude fiber content (45.19 to 46.12 %) also showed no significant differences between treatments ($p > 0.05$). This result is slightly lower than that reported by Rincón et al. (2005) (48.03 %), but higher than that found by Priego-Nerio (2007) in citrus peels subjected to similar osmotic processes. Small variations are attributed to the degree of fruit ripeness, growing conditions, and differential loss of soluble polysaccharides during blanching.

pH and acidity

The pH remained practically constant (3.12 to 3.13) and the titratable acidity at 0.77%. The pH range is below the critical limit for the development of Clostridium botulinum (pH < 4.6), which provides a safety barrier typical of acidic foods. The incorporation of 2% citric acid in the syrup is directly responsible for this profile.

Moisture, Fat & Soluble Solids

The final humidity (10.4%) puts the product in the intermediate humidity category, which limits water activity and extends shelf life without the need for refrigeration. The fat content (1.72%) corresponds mainly to residual essential oils (mainly limonene) that contribute to the characteristic aroma. Soluble solids (59°Brix) are consistent with a stable confectionery product.

Crude protein

Crude protein showed significant differences between treatments ($p < 0.05$). The T6 treatment, the only one that combines 60% syrup with drying at 60 °C, had the highest value (5.70 %), followed by T5 (60 % syrup with drying at 40 °C) with 5.65 %. The higher concentration of sucrose in the syrup appears to reduce the leaching loss of soluble proteins, while the higher drying temperature favors the concentration by moisture loss. The content obtained is comparable to the 7.55 % reported by Rincón et al. (2005) for mandarin peel meal and the 7.71 % crude protein reported by Ojeda (2002). These values indicate that jam can provide functional protein when incorporated into bakery and dairy products.

3.2. Statistical analysis (ANOVA and Tukey)

The analysis of variance (Table 3) indicates that sucrose concentration and drying temperature had a significant effect on crude protein and weight loss ($p < 0.05$), but not on ash, fiber, pH, moisture, fat, acidity or soluble solids. The concentration × temperature interaction was not significant for any of the variables ($p > 0.05$), which suggests that the effect of each factor is independent.

Table 3. ANOVA of the physicochemical variables as a function of sucrose concentration (C), drying temperature (T) and their interaction.

Variable	C-factor (p)	T-factor (p)	C × T (p)	Significance
Ashes	0,612	0,488	0,841	N.S.
Crude Fiber	0,094	0,201	0,732	N.S.
pH	0,883	0,772	0,901	N.S.
Humidity	0,524	0,318	0,664	N.S.
Fat	0,914	0,847	0,789	N.S.
Acidity	0,701	0,802	0,690	N.S.
Soluble solids	0,058	0,142	0,377	N.S.
Crude protein	0,021	0,034	0,162	*
Weight loss	< 0.001	0,019	0,084	**

n.s. = not significant ($p > 0.05$); * $p < 0.05$; ** $p < 0.01$. The p-values are reproduced as they appear in the original analysis; raw SPSS data were not available for recalculation. Source: Authors' elaboration with SPSS v.21.

For crude protein, Tukey's test separated the means into three homogeneous groups (a, ab, b), with significant superiority of T5 and T6 over T1 and T2; the T3 and T4 treatments occupied an intermediate position. In terms of weight loss, treatments with 60% syrup achieved the highest rates (approximately 38 to 42%) versus 20% (approximately 18 to 22%), confirming that the osmotic gradient is the main driver of water transfer, in agreement with Welti-Chanes and Vergara-Balderas (1997).

3.3. Microbiological quality

All microbiological counts of optimal treatment were within the acceptable limits of NTC 5247 (Table 4). No fecal coliforms, E. coli or Salmonella spp. were detected, confirming the safety of the product. The combination of acidic pH (3.12), intermediate humidity (10.4%), high soluble solids (59 °Brix) and vacuum packaging forms a multi-barrier system that limits the development of pathogenic and spoilage microorganisms.

Table 4. Microbiological analysis of the optimal treatment (T6: 60% sucrose, dried at 60°C).

Parameter	Result	NTC 5247 Limit
Mesophilic aerobes (CFU/g)	< 10	≤ 10^4
Molds and yeasts (CFU/g)	< 10	≤ 10^2
Total coliforms (CFU/g)	< 3	≤ 9
Fecal coliforms (CFU/g)	Absence	Absence
Escherichia coli (CFU/g)	Absence	Absence
Salmonella spp. (in 25 g)	Absence	Absence

Source: CIDI laboratory, Universidad Popular del Cesar, reported in Morales González (2012).

3.4. Amino acid profile by HPLC

HPLC analysis of optimal treatment identified five quantifiable amino acids (Table 5), all above the method's limit of quantification. Of these, three are essential for humans (lysine, methionine and tryptophan) and two are conditionally essential (arginine and cysteine). The absolute content is modest in terms of g per 100 g, but it must be interpreted in context: the peel is a matrix whose main contribution is fiber and bioactive compounds, so the presence of essential amino acids constitutes a relevant added value for the design of fortified foods.

Table 5. Amino acid profile in orange peel jam (optimal treatment) determined by HPLC.

Amino acid	Classification	Content (%)	Main biological function
Arginine	Conditionally essential	0,28	Protein synthesis; nitric oxide precursor; nitrogen balance.
Cysteine	Conditionally essential	0,10	Antioxidant (precursor of glutathione); protein disulfide bridges.
Lysine	Essential	0,20	Growth; tissue repair;

			antibody synthesis.
Methionine	Essential	0,10	Initiator of protein synthesis; methyl group donor.
Tryptophan	Essential	0,06	Serotonin and niacin precursor; sleep regulation.

Source: HPLC with OPA derivatization and fluorescence detection (excitation 340 nm, emission 450 nm). Data taken from Morales González (2012).

The values obtained are comparable to those reported by Demain and Solomon (1986) for citrus extracts: arginine 0.28%; cysteine 0.11%; lysine 0.20%; methionine 0.11 % and tryptophan 0.06 %. The agreement between sources reinforces the reliability of the data and suggests a relatively stable amino acid profile in the shell matrix, regardless of small variations in geographical origin.

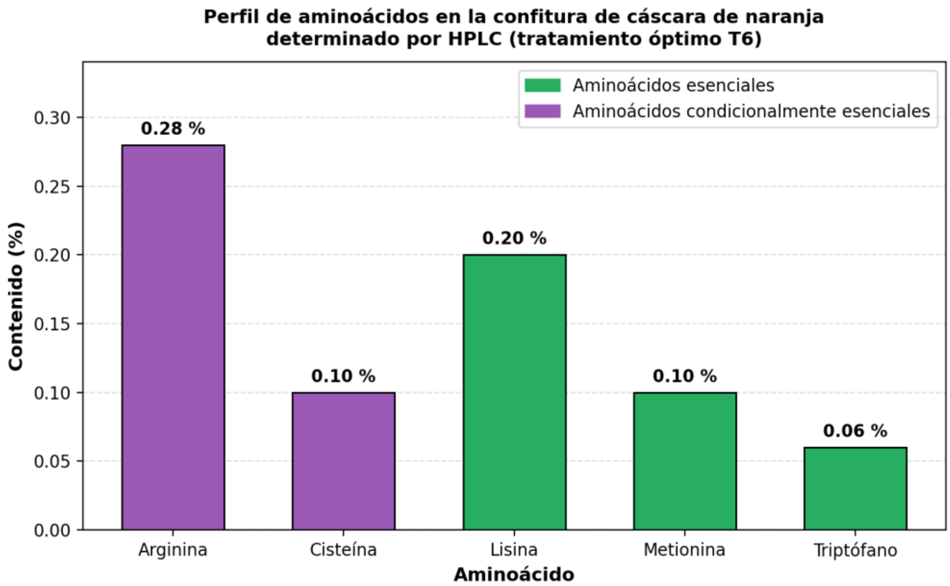

Figure 2. Amino acid profile detected by HPLC in optimal treatment (T6: 60% sucrose, dried at 60°C). In green, essential amino acids for humans; in purple, conditionally essential amino acids.

3.5. Application of jam as an ingredient: bread and yogurt

The validation of the jam as an ingredient was carried out by incorporating it into two contrasting matrices: stuffed bread (10% m/m) and sugary natural yogurt (8% m/m). The choice answers the research question: for the jam to be industrially viable, it must behave well in both a dry bread matrix and a creamy-acid milk matrix. The results showed acceptance levels of 68% in bread and 72% in yogurt, both above the threshold of 60% that is usually considered satisfactory for products in the development phase (Table 6). In bread, the predominant note was "I like it a lot" (60%); In yogurt, this category rose to 66%, suggesting greater sensory compatibility between the jam and the milk matrix. Absolute rejection ("I don't like it" category) was marginal in both cases: 4% in bread and 2% in yogurt.

Table 6. Verbal hedonic test results on bread and yogurt enriched with orange peel jam (n = 50 untrained panelists).

Category	Bread f	Bread %	Yogurt f	Yogurt %
I don't like it	2	4,0	1	2,0

I don't like it much	12	24,0	10	20,0
I am indifferent	2	4,0	3	6,0
Like	4	8,0	3	6,0
I like it a lot	30	60,0	33	66,0
Total	50	100,0	50	100,0
Acceptance (like + like a lot)	—	68,0	—	72,0

Source: Authors' elaboration based on Morales González (2012).

The greater acceptance of yogurt is explained by the synergy between the acidity of the dairy, the citrus aromas of the jam and the crunchy texture that provides contrast to a creamy base. In bread, it should be recognized that 24% of the panelists assigned the category "I don't like it much". The qualitative observations reported by the panelists attribute this score to bitter residual notes perceptible after chewing, probably associated with incomplete elimination of naringin during blanching. This result, although it does not compromise global acceptance, defines a specific line of improvement: optimising debittering by prolonging blanching, successive washes or enzymatic tests with naringinase before proposing a commercial version of enriched bread.

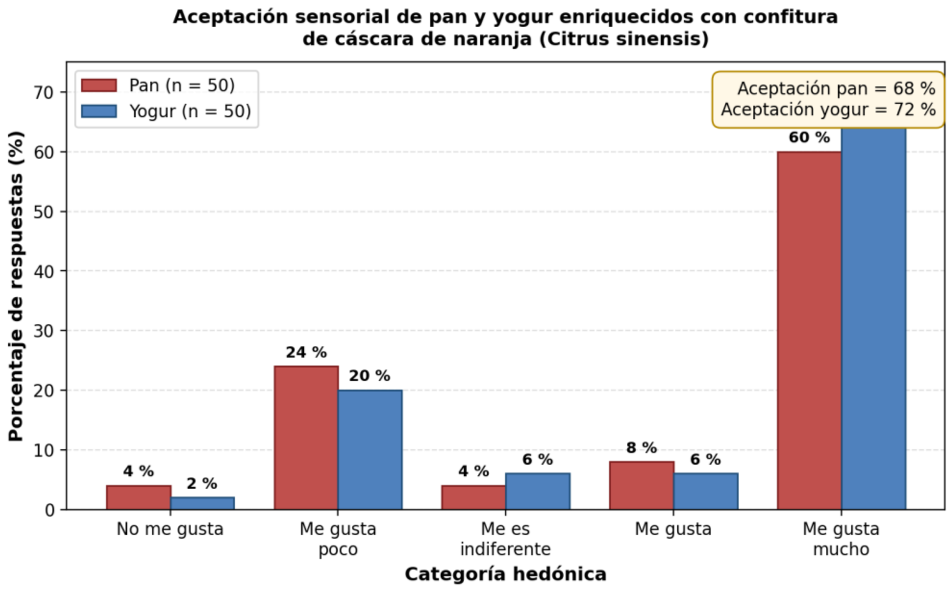

Figure 3. Percentage distribution of the responses of the verbal hedonic test applied to 50 untrained panelists on stuffed bread and natural yogurt enriched with orange peel jam. In the original language Spanish.

Figure 4 shows the gastronomic applications obtained during the experimental development: breads baked with the jam, the filling process, a sample of the stuffed bread used in the sensory evaluation and the yogurt added with the jam. These images document the operational feasibility of using jam as an ingredient in both products.

Figure 4. Gastronomic applications of orange peel jam: (a) breads baked with the jam, (b) filling process, (c) stuffed bread served as a sample of sensory evaluation, (d) natural yogurt added with the jam. Images taken from the annexes of Morales González (2012). In the original language Spanish.

4. Conclusions

The peel of the common orange (Citrus sinensis), traditionally discarded in the Cesar citrus chain, can be transformed into a harmless and nutritionally useful jam by osmotic dehydration followed by forced air drying. The process is reproducible, scalable, and compatible with cleantech practices.

The T6 treatment, with 60 % m/m sucrose syrup acidified with 2 % citric acid and dried at 60 °C for 6 h, was identified as the optimal. It maximizes weight loss during dehydration, keeps physicochemical parameters within the norm and obtains the best sensory attributes.

The proximal characterization showed a high content of crude fiber (45.19 to 46.12 %), crude protein (5.06 to 5.70 %) and ash (4.82 to 4.86 %), with acidic pH (3.12 to 3.13) and intermediate moisture (10.4 %). This profile corresponds to a stable food, with the potential to be used as an ingredient with functional contribution.

Five amino acids were identified by HPLC in the optimal treatment: arginine (0.28%), cysteine (0.10%), lysine (0.20%), methionine (0.10%), and tryptophan (0.06%). Three of them are essential for humans, which gives the product nutritional relevance.

Hedonic tests with 50 untrained panelists showed an acceptance of 68% for stuffed bread and 72% for natural yogurt enriched with jam. These values support its application as an ingredient in bakery and dairy products. The study confirms orange peel as a raw material of interest for the regional bioeconomy.

5. Future work and recommendations

- Quantify the specific content of minerals (Ca, K, Fe, Zn, Mg) and bioactive compounds (total flavonoids, hesperidin, naringin, carotenoids, antioxidant capacity by DPPH and ABTS). The percentage of ash (4.85%) suggests a relevant mineral contribution that has not yet been characterized.

- Optimize the debittering process to reduce the residual bitter notes that motivated the "I don't like it much" category in 24% of the panelists who evaluated the bread. Prolongation of scalding, successive washes or enzymatic treatment with naringinase can be tested.
- Evaluate vacuum osmotic dehydration to improve color uniformity and juiciness, since the porous matrix of the peel favors intracellular air retention that limits solute transfer.
- Implement automated temperature control during drying to prevent burnt edges that affect the appearance and bite of the final product.
- Characterize the instrumental texture profile (APL) of the final product and correlate it with sensory perception, to optimize the formulation intended for baking.
- Estimate production costs on a pilot scale, including the additional use of essential oils obtained during blanching, to build the technical-economic feasibility of industrial scaling.
- To study the shelf life of the vacuum-packed product under different storage conditions and to validate the stability of essential amino acids over time.
- To expand the amino acid profile by LC-MS or derivatization with ethyl chloroformate, techniques that allow quantifying proline, hydroxyproline and amino acids below the LOQ of the OPA method used in this study.

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Conflict of interest

The author declares that he has no conflict of interest.

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Data attribution: the primary experimental data that support this article come from the undergraduate work of Morales González (2012), developed in the Agroindustrial Engineering Program of the Popular University of Cesar. The analytical systematization and writing of the manuscript are the responsibility of the author who signs. The original thesis is cited in the reference list.

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