SCIENTIA PLENA
www.scientiaplena.org.br
VOL. 20, NUM. 06 2024
doi: 10.14808/sci.plena.2024.061502

Valorization of waste organic pitaya seeds (*Hylocereus undatus*): a technological approach

Valorização do resíduo de semente de pitaya orgânica (*Hylocereus undatus*): uma abordagem tecnológica

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(Recebido em 04 de março de 2024; aceito em 16 de junho de 2024)

Pitaya seeds (*Hylocereus undatus*) constitute agro-industrial waste with a high potential for developing new products and adding value. Studies characterizing these materials are scarce, though. This work aimed to characterize proximal composition, technological properties, thermal properties, and moisture adsorption isotherms. Carbohydrates, lipids, and proteins were the major components. Thermogravimetric analyses showed three mass loss events, with the onset of degradation processes at 200 °C and first-order transitions in a temperature range between -42 °C to 205 °C, with a glass transition at 1 °C. Water and oil retentions were 153 % and 89 % of their mass, respectively. The seeds showed hygroscopic characteristics, with a monolayer moisture value between 3.4-5.9 g H_2O.100 g solids^{-1} (dry basis). The GAB and Oswin were the best models to describe the moisture adsorption isotherms at 25 °C. The results indicate that pitaya seeds are promising materials for use as ingredients in the formulation of food products.
Keywords: dragon fruit, by-products, waste.

Sementes de pitaya (*Hylocereus undatus*) constituem um resíduo agro-industrial de elevado potencial para o desenvolvimento de novos produtos e produtos de valor agregado. Contudo, estudos de caracterização desses materiais são escassos. Este trabalho teve o objetivo de realizar a caracterização da composição proximal, propriedades tecnológicas e térmicas e isotermas de adsorção de umidade. Carboidratos, lipídios e proteínas foram os maiores componentes. As análises termogravimétricas indicaram três eventos de perda de massa, com processo de degradação iniciando a 200 °C e transições de primeira ordem na faixa de -42 °C a 205 °C, com transição vítrea a 1 °C. As retenções de água e óleo foram de 153% e 89% de sua massa, respectivamente. As sementes apresentaram características higroscópicas, com teor de umidade da monocamada entre 3,4-5,9 g H_2O.100g.sólidos^{-1} (base seca). GAB e Oswin foram os melhores modelos para descrever as isotermas de adsorção de umidade a 25 °C. Os resultados indicam que as sementes de pitaya são materiais promissores para utilização como ingredientes na formulação de produtos alimentícios.
Palavras-chave: fruta dragão, subprodutos, resíduos.

1. INTRODUCTION

According to the Food and Agriculture Organization of the United Nations (FAO), 1.3 billion tons of food and nearly half of all produced fruits and vegetables are lost or wasted yearly [1]. About 50% of the waste in agribusiness corresponds to roots, fruits, vegetables, and seeds; in fruit processing, approximately 40 % of the waste comes from peels and seeds [1, 2]. However, these wastes can be managed more efficiently, as they are potentially rich in nutrients and bioactive components [3].

In this perspective, the Circular Economy rules focus on reducing the waste produced and reusing them as by-products. Those residues are potentially rich in compounds of industrial interest such as sugars, polysaccharides, proteins, fibers, aromas, vitamins, fatty acids, and bioactive compounds. Thus, its reuse involves environmental and technological interests [4, 5]. Among these by-products, we draw attention to pitaya, specifically pitaya seeds.

Hylocereus undatus (*H. undatus*), popularly known as pitaya, pitahaya, pitaia, or dragon fruit belongs to the *Cactaceae* family, originated in Mexico and Central America, and is cultivated in many tropical and subtropical areas around the world [6]. Besides its attractive appearance, it became an economical alternative in the exotic fruit market due to its high added value [7]. Pitaya

stood out for its bioactive, nutritional, and nutraceutical properties, giving it status as a superfruit [6, 8, 9].

Regarding nutritional characteristics, pitaya seeds have protein levels comparable to legumes and high concentrations of fatty acids, 75 % of which are unsaturated [10-12]. Furthermore, its extracts stand out for the presence of tocopherols, flavonoids, sterols, and phenolic compounds, demonstrating antioxidant activities. Also, the phenolic and flavonoid contents are higher in the pitaya skin and pulp [13-16]. Despite those reports, relevant technological properties of pitaya seeds are not available yet. In fact, the seed separation method itself was only recently discussed [17].

Technological properties such as water and oil holding capacities (WHC and OHC), and emulsifying capacity (EC) are essential to assess a potential application of a given material as an ingredient in new product information [18]. Thermal degradation and component phase changes are also relevant information, since some constituents of seeds are thermosensitive and may change during usual processing and storage configurations. Knowing the thermal events occurring in the food when subjected to heat fluxes allows establishing limits to which one can submit the food during processing, without thermal damage [19].

Pitaya seeds are food matrices with a complex composition of amorphous nature, and given the hydrophilic nature of some components, they can absorb water, causing plastification process. Plastification, in turn, changes glass transition properties and water activity, altering the physicochemical and microbiological stability of the seeds during storage. This understanding helps define packaging and storage conditions and can be achieved by moisture sorption isotherms assays, which allow predicting the behavior of food materials facing different relative humidities [20, 21].

Although other works have explored the nutritional potential and the drying process [10, 12-14, 17], there is no data about technological properties of pitaya seeds available yet. It is also worth mentioning that the natural intraspecific variability expected for natural products justifies the investigation of nutritional properties, even if already reported, contributing to the understanding of the potential of the raw material. Thus, in the present work, we aim to characterize the seeds of organic pitaya (*H. undatus*) in terms of nutritional, technological, and thermal properties.

2. MATERIAL AND METHODS

2.1 Material

The chemical reagents and salts for isotherms, all of analytical grade, were from Vetec Química Fina (Rio de Janeiro, Brazil), Lefan Química Fina (São Paulo, Brazil) and Neon Comercial (São Paulo, Brazil). The Pectinex enzyme (Ultra SP-L, declared activity 3,300 polygalacturonase units per gram) was provided by the Latin American LNF (Rio Grande do Sul, Brazil).

The organic pitayas (*H. undatus*) were donated by the Recanto das Pitayas farm, located in Turvo - Santa Catarina - Brazil (28°55.117'S; 49°41.572'W), 2017-2018 crop with harvest between February and April 2018. The pulps presented average pH of 4.5 and soluble solids content of 9 °Brix.

2.2 Samples preparation

The pitayas were washed in running water and sanitized in sodium hypochlorite solution (100 ppm) for 15 minutes. Then, the peels were removed, and the pulps were manually cut into approximately 1 cm x 1 cm cubes.

The pulp-seed separation was performed by enzymatic hydrolysis (Pectinex), as de Santana et al. (2022) [17] described. The enzymatic reaction occurred in jacketed glass reactors at 51 °C ± 1 °C for 45 minutes. After washing with running water and filtration, the seeds were

dehydrated in an oven with forced circulation (Tecnal TE-394/2, Brazil), air velocity of 0.78 $m.s^{-1}$, 45 °C ± 1 °C for 180 minutes (final moisture content of 5.5 %) and, finally, vacuum packed in polyethylene packaging and kept at -18 °C until analysis.

The seeds were ground in an adjustable knife grinder (Hamilton Beach, USA) in fine mode, sieved, and particles with a size of -16/+32 mesh were selected to undergo the analysis.

2.3 Proximate composition

The moisture and volatile content were gravimetrically determined by heating in a drying oven (FANEN, 325 SE, Brazil) at 105 °C [22]. The protein content was determined by the Kjeldahl method (N = 6.25), the ash content by muffle incineration at 500 °C, and the lipids were extracted in a Soxhlet extractor with petroleum ether, following the methodologies described by AOAC [23].

The dietary fiber determination (soluble and insoluble fractions) followed the methodology described by Freitas et al. (2008) [24], using the Sigma-Aldrich Fiber Determination Kit containing the enzymes α-amylase, protease, and amyloglucosidase [25].

The carbohydrate percentage was calculated by subtracting all the contents mentioned above from 100 % (carbohydrate % = 100 – water and volatiles % - lipids % - proteins % - ash % - dietary fiber %).

All analyses were performed in triplicate, and the results were expressed as mean ± standard deviation in grams of analyte per 100 grams of dry solid ($g.100g\ solids^{-1}$ dry basis).

2.4 Thermal analysis

The thermogravimetric curves (TGA and DTG) were obtained using TGA 50 (Shimadzu, Japan). Approximately 10 mg ± 2 mg of sample were transferred to an aluminum pan and fitted to the TGA thermobalance. The analysis conditions were: heating rate of 10 $°C.min^{-1}$ with a temperature range of 25 °C to 800 °C in a dynamic nitrogen atmosphere with a flow rate of 50 $mL.min^{-1}$.

The phase transitions were evaluated by Differential Scanning Calorimetry (DSC) in the whole seed (S1) and the separated parts, namely tegument (C1) and endosperm (E1). Samples of 8 mg ± 3 mg were transferred to an aluminum pan and analyzed in a Jade-DSC calorimeter (Perker Elmer, USA), using an empty aluminum pan as a reference and a dynamic nitrogen atmosphere with a flow rate of 50 $mL{\cdot}min^{-1}$. The tests were conducted with two heating/cooling cycles. At first, samples were heated from - 60 °C to 350 °C with a heating rate of 10 $°C{\cdot}min^{-1}$. Once at 350 °C, the isotherm was evaluated for 2 minutes. Then, samples were cooled at a rate of 2 $°C{\cdot}min^{-1}$ up to - 60 °C, and a second isotherm was taken for 10 minutes.

2.5 Technological properties

2.5.1 Water-holding capacity (WHC) and water solubility index (WSI)

The WHC determination followed the methodology of Capitani et al. (2012) [26], where 1 g ± 0.2 g of sample (n = 3) was placed in a 15 mL falcon tube containing 10 g of distilled water and homogenized for 1 minute in a vortex tube shaker (VTX-F-220, Biomixer, Brazil) at 2,800 rpm. The suspension was centrifuged (Centrifuge 5804R, Eppendorf, Germany) at 2,200 g for 30 minutes. The supernatant volume was measured, and the WHC was expressed as the mean of the values and standard deviation in $g\ H_2O.g\ solids^{-1}$ (dry basis).

A 5 mL supernatant aliquot from the WHC analysis was transferred to aluminum capsules and oven-dried at 105 °C to determine the WSI. The solubility fraction (w/w) was calculated as the ratio between the final and initial supernatant mass and expressed as mean ± standard deviation in grams of dry solid per gram of water ($g\ solids.g\ H_2O^{-1}$ dry basis).

2.5.2 Oil-holding capacity (OHC)

The OHC determination followed the methodology described in item 2.5.1, substituting water for canola oil (density of 0.92 $g.mL^{-1}$). Results were expressed, as mean ± standard deviation, in grams of retained oil per gram of dry solid (g O.g $solids^{-1}$ dry basis).

2.5.3 Emulsifying activity (EA)

The EA was determined according to Capitani et al. (2012) [26] methodology, with modifications. 1.0 g ± 0.1 g of powdered seed (n = 3) was suspended in 100 mL of distilled water and homogenized in Ultra-Turrax (T25 digital, IKA, Germany) at 7,800 rpm for 2 minutes. Then, 100 mL of canola oil was added to the suspension and homogenized in Ultra-Turrax at 15,000 rpm for 2 minutes. A fraction of it was transferred to 15 mL falcon tubes and centrifuged (Centrifuge 5804R - Eppendorf, Germany) at 460 g for 10 minutes. The EA was expressed in volume (mL) of the emulsified layer in 100 mL of the original amount.

2.6 Moisture adsorption isotherms

The isotherms were determined according to Barbosa-Cánovas et al. (2007) [27], whose methodology is based on static-gravimetric tests. Seven saturated saline solutions were used to provide water activity (a_w), in the range of 0.11 to 0.90: lithium chloride (LiCl – 0.11); magnesium chloride (MgCl – 0.33); potassium carbonate (KCO_3 – 0.43); sodium bromide (NaBr – 0.58); sodium chloride (NaCl - 0.75); potassium bromide (KBr – 0.80) and barium chloride (BaC_{l2} – 0.90) [28].

The samples remained in a desiccator with silica gel for ten days prior to the analysis. Triplicate samples with 1 g ± 0.2 were weighed in porcelain crucibles and placed in the saturated saline solutions recipients. The recipients were hermetically closed and kept in a B.O.D. (TE-371 Tecnal, Brazil) at constant temperature of 25 °C. The crucibles were weighed on an analytical balance (Bioprecisa, FA2104N, Brazil) at regular time intervals until they reached a constant mass (variations smaller than 0.001 g) which was considered the equilibrium condition.

Finally, the samples were dehydrated in a drying oven (DeLeo, Brazil) at 60 °C until constant mass, and the equilibrium moisture content (Xe) on a dry basis (g H_2O.100 g $solids^{-1}$ dry basis) was determined by Eq. 1.

$$Xe = \left(\frac{me - ms}{ms}\right).100 \quad (1)$$

where: Xe = equilibrium moisture (db), me = equilibrium sample mass, and ms = sample dry mass.

The isotherms curves were obtained by plotting the values of Xe against a_w. The models of Guggenheim, Anderson, and Boer (GAB); Peleg; Bruauer; Emmet and Teller (linearized BET); Smith; Oswin, and Langmuir were fitted to experimental data of equilibrium moisture as a function of a_w, according to Eqs. 2-7 (Table 1).

Table 1. Isotherm representation models to evaluate organic pitaya seeds moisture adsorption (Hylocereus undatus).

Model	Equation	
GAB - Guggenheim-Anderson-de Boer [29]	$Xe = \frac{Xm.C.K.aw}{(1-K.aw)(1-K.aw+C.K.aw)}$	(2)
Peleg [30]	$Xe = k_1.aw^{n1} + k_2.aw^{n2}$	(3)
Linearized BET [31]	$\frac{aw}{(1-aw).Xe} = \frac{1}{Xm.C} + \frac{(C-1).aw}{Xm.C}$	(4)
Smith [32]	$Xe = A - B.[\ln(1-aw)]$	(5)
Oswin [33, 34]	$Xe = A.\left(\frac{aw}{1-aw}\right)^B$	(6)
Langmuir [35]	$Xe = \frac{Xm.C.aw}{1+(C.aw)}$	(7)

Xe = equilibrium moisture on a dry basis (g H_2O.100 g solids^{-1} db); aw = water activity; Xm and A = moisture in the molecular monolayer (g H_2O.100 g solids^{-1} db), C, K, k1, k2, n1, and n2: fit parameters.

2.7 Statistical analysis

The adsorption isotherm models fit was performed by non-linear regression, with 95% probability, using the Statistica® 7.0 software (Statsoft, Tulsa, OK, EUA). To assess the quality and select the best model to describe the equilibrium moisture content in pitaya seed, the magnitude of the coefficient of determination (R^2, ideally close to 1), the Mean Relative Error (MRE, desirably lower than 10%), the Standard Deviation of the Estimate (SDE, desirable closer to 0) and the residual distribution (ideally random) [36] were evaluated, according to Eqs. 8-10.

$$R^2 = \frac{\sum_{i=l}^{n}(Xe_{pred,i} - \overline{Xe})^2}{\sum_{i=l}^{n}(Xe_{expj} - \overline{Xe})^2} \quad (8)$$

$$SDE = \sqrt{\frac{\sum_{i=1}^{n}(Y_i - \hat{Y}_i)^2}{GLR}} \quad (9)$$

$$MRE = \frac{100}{n}\sum_{i=1}^{n}\frac{|Y_i - \hat{Y}_i|}{Y_i} \quad (10)$$

Where: Yi is the experimentally observed value (% db, or g H_2O.100 g solids^{-1} db), $\hat{Y}i$ is the value calculated by the model (g H_2O.100 g solids^{-1} dry basis), N is the number of observed data, and GLR is the residual degrees of freedom (number of observed data subtracted from the number of model parameters).

3. RESULTS AND DISCUSSION

3.1 Proximate composition

The results of proximal composition for organic pitaya seeds (dry basis), were: moisture 5.5 ± 0.2 %; lipids 30.0 ± 0.1%; proteins 24.50 ± 0.3 %; crude fiber 36.80 ± 0.2 % (31.1 ± 0.2 % of insoluble and 5.7 ± 0.2 % of soluble fiber); ash 2.6 ± 0.1 %; and other carbohydrates 0.60 %.

The moisture content was similar to those reported by Chemah et al. (2010) [13] for pitaya seeds of the genus *H. undatus*, *H. polyrhizus*, and *Selenicereus megalanthus*. The lipid content

was similar to that reported by other authors for *H. undatus* seeds but higher than *H. polyrhizus* and *Selenicereus megalanthus* [10, 13, 15]. When compared to seeds from other cacti species, such as *Opuntia ficus-indica* L. and *Opuntia stricta*, pitaya seeds have a lipid content at least twice as high [37-39].

The protein content corroborates that reported in other studies with pitaya seeds [12-15]. It was higher than in other seeds such as flaxseed (19.5%), sesame (17.8%), amaranth (15.3%), and white rice (6.8%), comparable to the content found in legumes such as chickpeas (25.0%), black beans (24.3%) and green peas (25%) [40]. The high protein content can make pitaya seeds interesting ingredients for the food industry.

Fibers represented the highest carbohydrate content, as well as the most significant fraction of macronutrients evaluated in the seeds and were predominantly insoluble. This high concentration is mainly due to lignocellulose, cellulose, and hemicellulose, which are common constituents of seed peels. According to Banerjee et al. (2017) [41], research interest in lignocellulosic biomass is growing, especially when treating food waste as a renewable source of bioactive polysaccharides. The carbohydrate and fiber values obtained in this work are similar to those reported by Villalobos-Gutiérrez et al. (2012) [12] for pitaya seeds of the genus *H. polyrhizus*.

3.2 Thermal analysis

The organic pitaya seeds thermograms (TGA and DTG) are shown in Figure 1. The values of the initial (To), maximum peak (Tp), and final (Tc) temperatures of each thermal event are shown in Table 2.

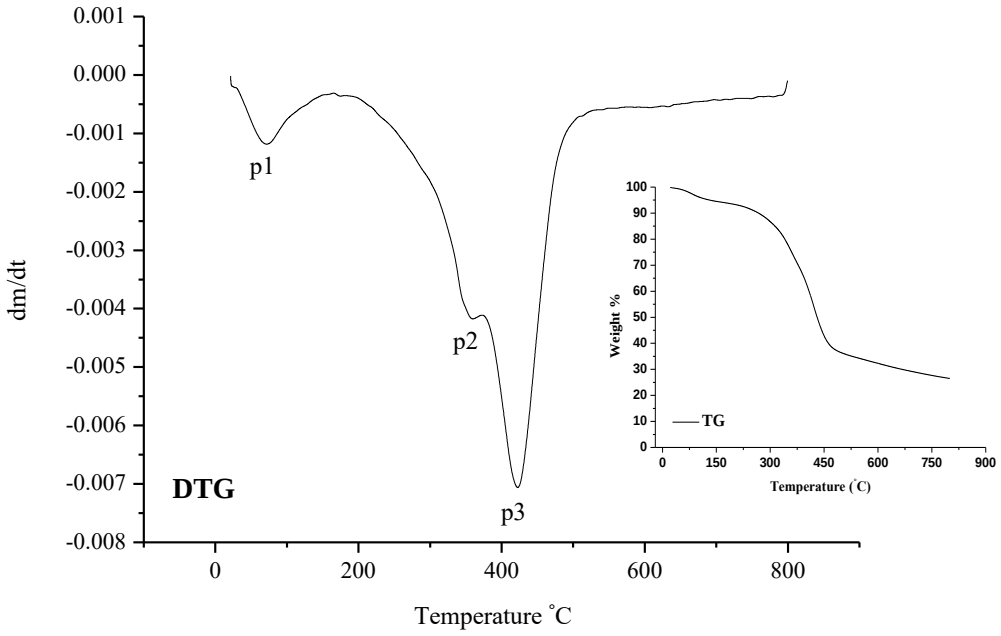

Figure 1. TGA-DTG thermograms of organic pitaya seeds (Hylocereus undatus).

The seeds showed three mass-loss events (p1, p2, and p3 in Figure 1), which indicate the components' volatilization and thermal decomposition processes, completed at 550 °C, followed by carbonization process of the material (Table 2).

Table 2. Degradation temperatures of thermogravimetric curves (DTG) of organic pitaya seeds and transition temperatures of DSC tests for whole seeds (S1), seed coat (C1), and endosperm (E1).

DTG					
	Event	**T_o (°C)**	**T_p (°C)**	**T_c (°C)**	
	p1	28	68	150	
	p2	200	357	374	
	p3	375	423	550	
DSC					
Sample	**Event**	**T_o (°C)**	**T_p (°C)**	**T_c (°C)**	**Run**
S1, E1	(a), (g)	- 42.0	- 32.0	- 27.2	1
S1, E1, C1	(b), (h), (n)	- 27.0	- 22.3	- 17.7	1
S1, E1	(c), (i)	- 17.0	- 13.8	- 4.5	1
S1, E1	(d), (j)	5.0	9.5	14.0	1
S1	(e)	14.0	95.5	200.5	1
S1	(f)	205.0	223.6	242.0	1
S1	Endo	- 40.0	- 1.0	12.0	2
E1	(k-l)	16.5	111.0 139.0	187.0	1
E1	(m)	197.0	215.0	229.0	1
E1	Endo	- 40.0	- 1.0	12.0	2
C1	(o)	12.0	95.8	205.0	1
C1	Tg	- 1.8	1.0	3.5	1
C1	Tg	- 4.0	1.0	8.5	2

T_o – onset tempereture; T_p – peak temperature; T_c – "endset" temperature; ΔH – Melting enthalpy in joule per gram.

The first mass-loss event (p1) corresponds to the dehydration and evaporation of volatile compounds with low molar mass, as reported in blackberry, chokeberry, raspberry, and açaí seeds [42, 43]. According to Henrique et al. (2013) [44], the loss of these compounds can occur in a temperature range between 30-180 °C.

The second mass-loss event (p2) started at 200 °C and may be associated with the protein decomposition. For protein extracts from legumes (chickpeas, lentils, peas, and beans), Ricci et al. (2018) [45] report the volatilization of protein fragments occurring in the temperature range of 178-228 °C, and a maximum mass-loss rate at 220-330 °C. In this same temperature range, specifically below 260 °C, the decomposition processes of polyunsaturated fatty acids (PUFAs) such as linoleic and linolenic acids, which are in high concentrations in pitaya seeds, can also start [10, 13, 15, 46].

It is worth highlighting that 200 °C, the initial temperature of the decomposition process in p2, may be the limiting temperature for thermal processing of the studied seeds since the PUFAs, due to their chemical structure, might be the more thermosensitive lipids in these seeds. The lipids' thermal decomposition depends on chemical structure and fatty acid composition. The longer and more saturated the fatty acid chain, the higher the boiling point and, therefore, the greater the thermal stability [47, 48]. Thus, in the temperature range in which the p2 mass-loss event occurs, the monounsaturated fatty acid decomposition process can start, extending to the p3 event temperatures for saturated fatty acid degradation once the two peaks overlap [47].

Fibers (cellulose and hemicellulose) and other carbohydrates, can also undergo decomposition at temperatures related to peaks p2 and p3. Yang et al. (2007) [49] evaluated the pyrolysis process in hemicellulose, cellulose, and lignin and reported degradation temperatures for these components that corroborate the values found in this study. Piasecka et al. (2021) [43] evaluated the thermal degradation of blackberry, chokeberry, and raspberry seeds and associated mass-loss events between 120-230 °C with carbohydrate degradation. According to González Martínez et al. (2019) [50], the thermal degradation of hemicellulose occurs between 250-320 °C, the cellulose, as a polymer with a more crystalline structure, degrades at higher temperatures (280-400 °C) while lignin starts at 400 °C. Similar behavior was observed by Shadangi and Mohanty (2014) [51] sin a study with inedible oil seeds. In açaí seeds, a temperature close to 300 °C was associated with depolymerization and decomposition of hemicellulose and cellulose [42]. In contrast, blackberry, chokeberry, and raspberry seeds, temperatures of 300-500 °C were associated with carbohydrate and lipid pyrolysis processes [43]. According to Hernández-Montoya et al. (2009) [52], the complexity of lignocellulosic materials contributes to the observed overlap in the mass-loss peaks, as seen in orange, lemon, and grapefruit seeds.

The TGA curve (Figure 1) shows about 25 % residual mass. Similar behavior was observed in blackberry, chokeberry, and raspberry seeds under a nitrogen atmosphere, as in the present study. The authors attributed to thermostable inorganic compounds [43].

Although seeds are complex food matrices, this analysis allowed us to observe the temperature limits of degradation, indicating that seeds can be submitted to usual processing temperatures, preventing significant degradation of their more thermosensitive compounds.

The DSC curves (Figure 2A) for S1, C1, and E1 showed endothermic peaks (downward-facing) indicating first-order transitions related to melting processes. Second-order transitions, such as the glass transition (Tg), are represented as a gradual change from baseline (Figure 2B). Table 2 presents the transition temperatures for each event.

Pitaya seeds have components with amorphous, semi-crystalline, and crystalline structures. Thus, identifying each constituent responsible for the observed thermal event becomes challenging since many of these events are simultaneous, and their curves overlap. Separating the tegument (C1) and the endosperm (E1) was a strategy to separate different compositional structures and promote a better visualization of those events. Furthermore, the second run (Figure 2B) allowed blanking of the thermal history of the samples since the crystalline material would have gone through the thermal transition during the first run. The observation of the Tg became, thus, possible.

The thermogram S1 showed five endothermic peaks of thermal transition (Figure 2A). The peaks from (a) to (d) refer to the peaks (g) to (j), and peak (f) refers to the peak (m) observed in E1. Peak (e) was an overlap of peaks (k-l) in E1 and (o) in C1 (Table 2). Separate E1 and C1 analyses made it possible to determine a Tg at C1 (Figure 2B), not detected in the S1 assay. This separation also favored identifying the temperature range in which the lipids underwent the phase transition. Also, it confirmed that the other seed constituents perform overlapping phase transitions in a wide temperature range between 14 °C and 205 °C (Table 2).

Six thermal events were observed in E1, four of which (g, h, i, j) were associated with lipid melting processes, with T_p between - 32.0 °C and 9.5 °C (Table 2). That is due to the unsaturated character of fatty acids, which correspond to 75% of the fatty acids present in pitaya seeds [11, 13, 15]. Similar endothermic peaks reported for pitaya (*H. undatus* and *H. polyrhizus*) seed extracts were related to fatty acids and triacylglycerol thermal transitions [14, 15]. That strongly suggests that melting peaks (k-l) involve the melting of other components present in the seed endosperm, such as water, proteins, and carbohydrates. In trials with pea and lentil seeds and protein isolates, Piasecka et al. (2021) [43] and Ladjal-Ettoumi et al. (2016) [53] respectively, associated the thermal transition of carbohydrates and protein denaturation with the temperature range observed at the peak (k-l) of E1. The (m) peak, although showing a melting process, is more associated with the beginning of the degradation of the seed components, as seen in the TGA assays.

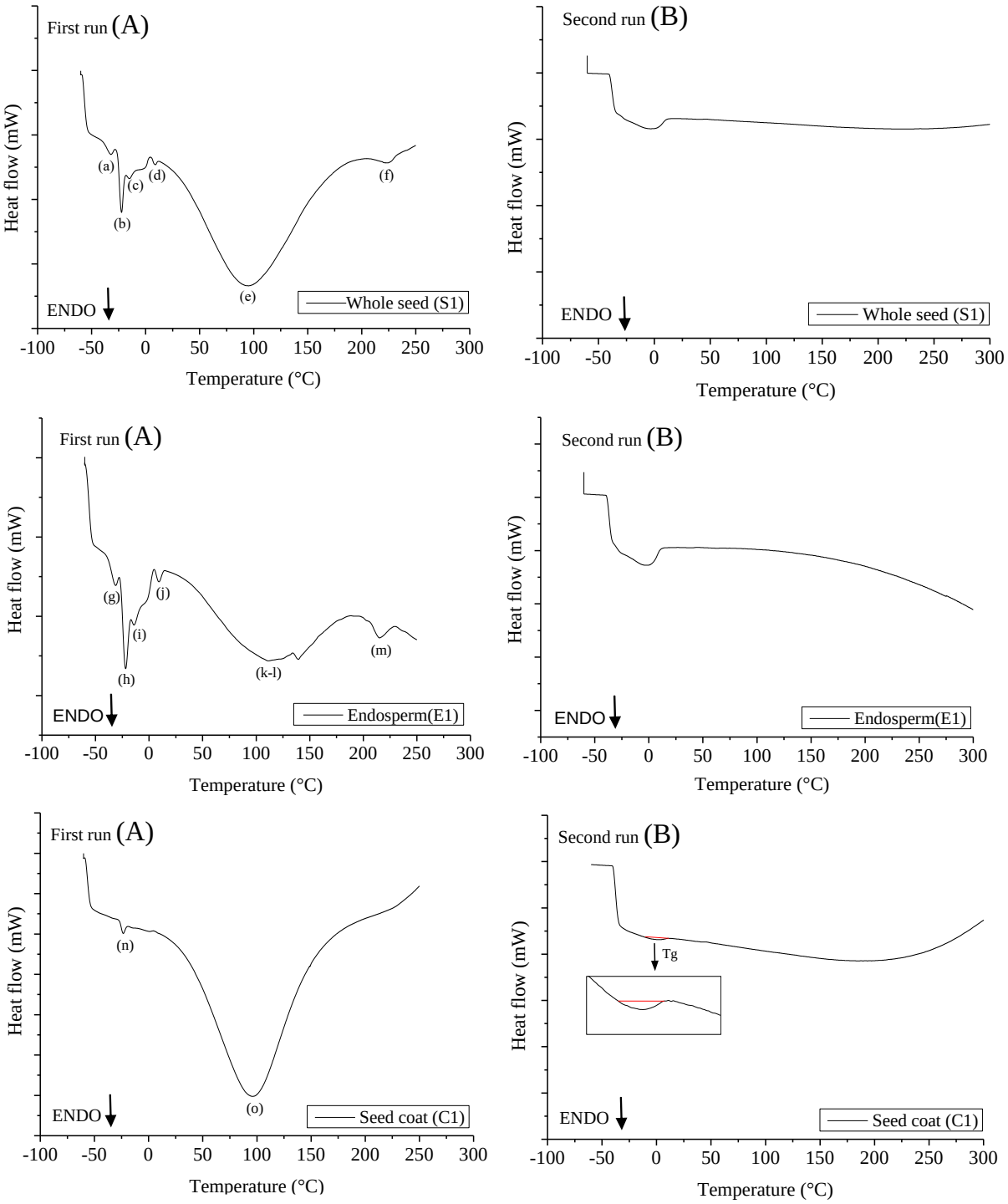

Figure 2. DSC thermograms analysis of powder organic pitaya seeds (Hylocereus undatus) as whole seeds (S1), seed coat (C1), and endosperm (E1).

The C1 thermogram (Figure 2A) showed two endothermic peaks (n), with T_p of - 23.4 °C, and (o), with T_p of 95.8 °C, and a Tg with T_p of 1 °C (C1-B), the latter due to the presence of amorphous material. The seed coat is mainly composed of cellulose, hemicelluloses, and lignin polymers and may contain smaller amounts of pectic substances and waxes. These substances differ in terms of their structural organization, having crystalline (cellulose) and amorphous (cellulose, hemicellulose, and lignin) regions, which interfere with the thermal transition [54]. While the (n) peak may be due to the melting of waxes present in small amounts, the (o) peak is due to the thermal transition of polymers, which occurred in the same temperature range as the

peak (e) observed in E1. That shows that both the seed coat polymers and the endosperm constituents undergo thermal transitions in the same temperature range, with peaks overlapping, as seen in S1. This overlap did not allow directly associating the transition temperature of each seed constituent separately. Thermal transitions involving cellulose and lignin components and their complexes, as well as water phase changes and volatile compounds at temperatures close to 100 °C, can complicate evaluating the thermal transitions in seeds [49, 50].

3.3 Technological properties

The water and oil retention capacities (WHC and OHC) and emulsifying activity (EA) are crucial functional properties for the formulation of new food products, influencing their quality and stability [55]. Their determination is essential to understanding the interactions between components, structure, physicochemical properties, and nature of the environment or food matrices [25]. Since the present work is pioneering the investigation of those properties of pitaya seeds, the comparison discussed in the following paragraphs is based on other reference seeds and grains.

Pitaya seeds presented WHC of 1.53 ± 0.07 g H_2O. g solids^{-1} and WSI of 0.01 g solids. g H_2O^{-1} (dry basis). That means, despite having low solubility, pitaya seeds can retain 153 % of their mass in water. This value was similar to that reported for seeds of six varieties of quinoa and peas [56, 57] and higher than that reported for moringa seeds, Brazil nuts, cashew seed, hazelnut, macadamia, pecan, pistachio, walnut, peanuts and rice [58]. However, it was lower than reported for chickpea, soybean, and wheat [59]. According to Mune et al. (2016) [58], polar amino acid residues in proteins and the composition of carbohydrates are the main factors affecting WHC in foods. The presence of polysaccharides, such fibers and mucilage, influences WHC due to its ability to swell. The high protein content and the presence of hydrophilic amino acids promote an increase in WHC in seeds [60, 61]. In a study conducted with both fresh and heat-treated canola, soybean, and linseed seeds, Khattab and Arntfield (2009) [62] observed intra and interspecies WHC variations, attributed to three factors: the polysaccharides composition, the conformational characteristics of proteins, and the gelation process. The WHC values found in powdered pitaya seeds indicate that they could be used as food ingredients in products with high water content where phase separation is undesirable.

Pitaya seeds presented OHC values of 0.89 ± 0.07 gO.g solids^{-1} (dry basis). These results indicate that, although pitaya seeds can retain 89 % of their mass in oil, they have a greater affinity for water. Similar OHC values are reported for Bolivian black real-quinoa [56]. However, pitaya seeds showed higher oil retention than rice and lower than chia, chickpeas, lentils, and peas [26, 57, 59]. According to Capitani et al. (2012) [26] and Aider and Barbana (2011) [60], high oil contents in food matrices reduce the OHC, as well as high values of WHC. It is noteworthy that pitaya seeds had both high lipid content (30 %) and high WHC, which may have negatively affected the OHC. However, OHC can be affected by protein content and the presence of non-polar amino acids or hydrophobic domains of proteins which, when exposed (favored by denaturation), can establish hydrophobic interactions, increasing oil retention [61].

Although it is common in seeds, we did not observe the emulsifying capacity in pitaya seeds, nor were studies found for comparison purposes.

3.4 Moisture adsorption isotherms

Figure 3 shows the experimental values of Xe of organic pitaya seeds as a function of a_w. The experimental data represent the mean of three repetitions for Xe, whose standard deviations are within the range of 0.0003 to 0.0008 (g H_2O.100 g solids^{-1} – dry basis). The solid lines represent the fits of the most suitable models, as will be discussed throughout this topic.

An increase in Xe with increasing a_w was observed, characterising materials of a hygroscopic nature, corroborating the results of WHC. This effect is due to the increase in the surrounding vapor pressure, forming a pressure gradient towards the interior of the food, thus increasing

moisture adsorption [63]. Similar behavior was observed in other seeds such as chia, pine nuts, sunflower seed, quinoa, and hazelnut [21, 36, 63-65].

At the highest value of a_w (0.9), Xe was 14.6 g H_2O.100 g solids^{-1} (dry basis) being, therefore, the highest amount of water absorbed by seeds under the conditions studied. In chia seed, Xe at 25 °C was 18-20 g H_2O.100 g^{-1} while, for grape seed, it was 9.51 [64, 66]. The moisture adsorption properties of foods, aside from being influenced by pressure and relative humidity, are affected by the composition, physicochemical structure, and hygroscopicity of the food, as well as by the temperature [67], making the isotherm specific for each food and case.

The isotherm presented a sigmoid shape, typical of the S form or Type II, [68]. The sigmoid shape of the curves describes the behavior of the isotherms of most foods [67, 69] and was observed in studies with quinoa seeds, Cumari-do-Pará pepper, safflower, chia and oilseeds [36, 63, 64, 70, 71], corroborating the results obtained in this study.

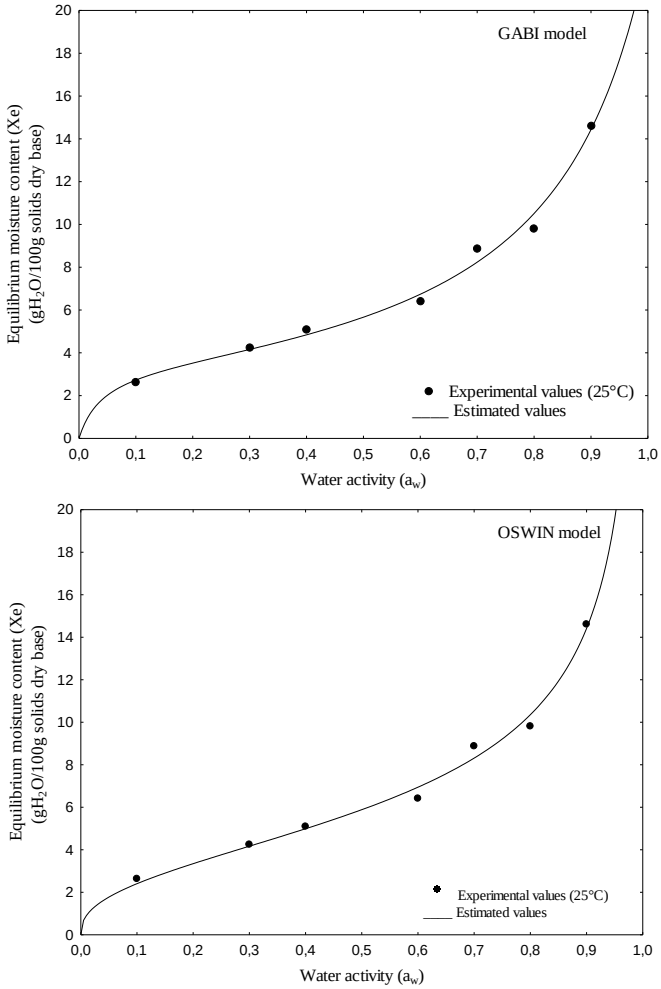

Figure 3. Experimental values of equilibrium moisture content as a function of the a_w of organic pitaya seeds (Hylocereus undatus) at 25 °C and the mathematical fit for the GAB and Oswin models.

The adjusted parameters of the six models evaluated and the respective parameters of fit quality evaluation (R^2, SDE, MRE, and residual distribution) are shown in Table 3.

Except for the linearized BET and Smith models, the others could describe well the pitaya seed isotherms, as they presented $R^2 > 0.99$, SDE < 1 %, MRE < 10 %, and randomized distribution of residues (Table 3). However, the GAB and Oswin models (Figure 3) were the ones that best described the experimental data on the moisture adsorption process in these seeds. GAB is an important and broadly accepted semi-empirical model to describe many foods' isotherms [71-75]. The Oswin model, in turn, is widely considered adequate to describe seed adsorption

isotherms [63, 76, 77], being adopted as a standard model by the American Society of Agricultural and Biological Engineers (ASABE) [69].

Table 3. Parameters of hygroscopic equilibrium models for the moisture adsorption of pitaya seeds, with their respective: determination coefficients (R^2), the standard deviation of the estimate (SDE), mean relative error (MRE) and analysis of residual distribution with significance level of 95 %.

Model Type	Model coefficients (25°C)	Goodness of fit parameters			
		R^2	SDE (% db)	MRE (%)	Residual Distribution
GAB	Xm = 3.401 K = 0.852 C = 29.527	0.994	0.539	4.605	Random
PELEG	K_1 = 8.238 K_2 = 13.715 n_1 = 0.521 n_2 = 13.781	0.994	0.620	4.344	Random
Linearized BET	Xm = 0.071 C = 20.782	0.981	0.875	12.060	Random
Smith	A = 2.147 B = 5.236	0.993	0.527	5.418	Biased
Oswin	A = 5.887 B = 0.407	0.995	0.454	4.867	Random
Langmuir	Xm = 8.887 K = 1.371	0.937	1.551	17.044	Biased

Note: Xm, K_1, and A equate to monolayer moisture (g H_2O.100 g solids^{-1} dry basis); K_2 = g H_2O.100 g solids^{-1} dry basis; n_1 and n_2 are dimensionless; K and C = constants related to the heat of sorption; B equates to the K of GAB; % db = g H_2O.100 g solids^{-1} dry basis.

The Xm parameter is relevant designing packaging systems and defining storage conditions that reduce foods physicochemical changes, such as deterioration, oxidation, darkening, and loss of crispness, among others, extending their shelf life [64]. The GAB model Xm parameter represents the moisture content in the monolayer, corresponding to the mass fraction of water absorbed in the material in the form of the monolayer. In the pitaya seeds, this content was 3.4 and 5.9 g H_2O.100 g solids^{-1} (dry basis) according to the GAB and Oswin models. For the Oswin model, the parameters A and B are equivalent to the parameters Xm and K of the GAB model, respectively [78]. Similar values of Xm were reported for rapeseed, chia, and hazelnut [64, 65, 71].

The residual dispersion analysis is recommendable to assess the fit quality of the models. That is because even when the statistical parameters indicate a satisfying fit ($R^2 > 0.99$, SDE < 1 %, and MRE < 10 %), the model may have a biased residual distribution. Thus, the selected model must present residual values around zero and a non-biased distribution [79]. Figure 4 shows the residual distribution graphs, consisting of the difference between the observed values and the values estimated by the GAB and Oswin models as a function of the predicted values.

The analysis presented residual values around zero and their random distribution, meaning that both models satisfactorily described the behavior of the moisture adsorption isotherms of pitaya seeds under the conditions studied.

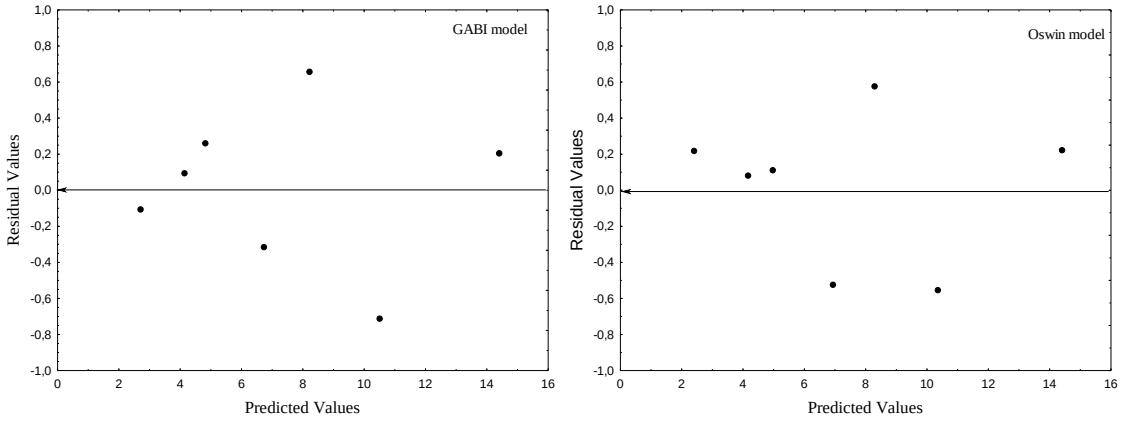

Figure 4. Residual graph for the GAB and Oswin models of the moisture adsorption isotherms of organic pitaya seeds (Hylocereus undatus).

4. CONCLUSION

Thermal analysis showed that the degradation of thermosensitive compounds starts at 200 °C. This means that the seeds can endure usual processing temperatures without significant changes in these compounds. In the whole seed DSC curves, the endothermic peaks were associated with melting processes and ranged from 109 °C to 179 °C. The separate analysis of the seed's parts allowed us to observe the thermal transition of the integument starting at 22 °C with high enthalpy variation, as well as two melting peaks in the endosperm with the second transition ending at 230 °C, which did not occur in the whole seed evaluation. That demonstrated the complexity in the composition of seeds causes thermal transitions to occur or simultaneously or at close temperatures.

The technological properties showed that pitaya seeds retain 153 % and 89 % of their mass in water and oil, respectively, highlighting their hygroscopic nature. This behavior was corroborated by the moisture adsorption isotherm of the seeds, which have a Xe value of 14.6 g H_2O/100 g solids at 0.90 a_w and a type II sigmoid shape. Among the six fitted models, the GAB was the one that best described the pitaya seed isotherm, according to the established quality parameters, presenting a calculated monolayer value of 3.4 (dry basis), similar for both pitaya seeds.

Therefore, in a pioneering way in the scientific literature, we explored the technological potentialities of pitaya seeds (*H. undatus*). We demonstrated their high protein and carbohydrate contents, associated with water and oil holding capacities that are interesting for incorporation in food matrices and thermal profiles that confirm the suitability of the raw material at the usual thermal processing conditions in foods. Therefore, and guided by the ideals of sustainability, we point out the great potential of this until then considered “waste” for use as an ingredient, thus a co-product, functional both nutritionally and technologically in the food industry.

5. ACKNOWLEDGMENT AND FUNDING

The authors thank Recanto das Pitayas for the kind supply of pitayas and LNF Latino Americana for the donation of Pectinex.

This work was supported by the Coordination for the Improvement of Higher Education – Brazil (CAPES) – Finance Code 001.

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