



Thermal degradation of phenolic compounds and water adsorption isotherms of flours from jenipapo peel, pulp, and seeds

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Abstract

Jenipapo (*Genipa americana* L.) is a Brazilian fruit known for its abundance of phenolic compounds, which have potential nutritional and technological applications. This study investigated the production of flours from the pulp (FP), peel (FC), and seeds (FS) of jenipapo, dried at 70 °C, by evaluating the degradation kinetics of total phenolic compounds (TPC), thermogravimetric analysis, Fourier-transform infrared spectroscopy (FTIR), and hygroscopic behavior through the determination of water adsorption isotherms. The results indicated that first-order models were effective in predicting the degradation of TPC in all flours, with a reduction in these compounds as storage temperature increased. The peel flour (FC) demonstrated a higher content of TPC and lower susceptibility to degradation at 30 and 40 °C, while the seed flour (FS) showed lower TPC content and shorter half-life times. Thermogravimetric analysis revealed three mass loss events in the flours, related to dehydration, decomposition of components such as proteins and lipids, and the formation of fixed mineral residue, with the greatest loss observed during the third event. Endothermic peaks were observed in all three flours during the gelatinization process. FTIR analysis confirmed the presence of various functional groups, highlighting the similarity between the samples. The water adsorption isotherms of all flours were well fitted by the GAB model and classified as type II at the studied temperatures. The peel flour stood out for having a higher content of phenolic compounds and greater stability during storage, favoring its bioactive potential. These findings highlight the potential application of jenipapo flours, particularly peel flour, as ingredients with relevant bioactive stability and hygroscopic behavior, supporting strategies for product development in the food industry. The demonstrated retention of phenolic compounds at lower storage temperatures and the defined water adsorption patterns reinforce their technological viability and potential functional contribution.

Keywords *Genipa americana* L. · Phenolic compounds · Temperature coefficient · Thermogravimetry · Total phenolic contents · Water absorption

Introduction

Jenipapo (*Genipa americana* L.) is a fruit with excellent nutritional properties and potential for processing. Its pulp contains a high concentration of phenolic compounds and a strong capacity to inhibit enzymes such as α -glucosidase,

α -amylase, and lipase, with possible antidiabetic benefits. Additionally, the peels of jenipapo are notable for their exceptional levels of these compounds and antioxidant activity, often surpassing those found in the pulp itself [1, 2].

Known for their antioxidant properties and potential health benefits, phenolic compounds include flavonoids, phenolic acids, and tannins, which have demonstrated anti-inflammatory, antimicrobial, and antiviral activities, potentially contributing to immune system modulation and improved metabolic function [3]. These compounds, found in the pulp, peels, and seeds of jenipapo, can be preserved through controlled drying, which not only concentrates

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the phenolic compounds and other solids but also helps inhibit enzymatic activity that contributes to their degradation. However, environmental factors such as temperature, humidity, and oxygen exposure may influence degradation reactions during storage, affecting the bioactive stability and functional potential of these ingredients [4, 5].

Thermal analysis techniques are essential for investigating how biological products respond to temperature variation over time, providing valuable thermodynamic and kinetic data. These techniques offer detailed information on thermal stability, degradation of bioactive compounds, and other essential physicochemical characteristics, contributing to a better understanding of the structural and compositional changes that occur during heating processes [6].

Despite the documented phytochemical richness of jenipapo and the growing industrial and scientific interest in recovering bioactive compounds from fruit residues, research on the thermal stability and storage behavior of jenipapo byproducts remains limited. Studies addressing the pulp, peel, and seed fractions are particularly scarce and often fragmented, resulting in a lack of integrated data on thermal degradation, thermogravimetric behavior, and water sorption processes. These knowledge gaps hinder the development of technological strategies for valorizing byproducts from this fruit and restrict advances toward reducing agroindustrial waste.

Therefore, aiming to optimize processing and storage conditions and ensure the quality and nutritional efficacy of jenipapo flours, this study was conducted to evaluate the storability of flours produced from the pulp, peels, and seeds of jenipapo dried at 70 °C, determining the degradation kinetics of total phenolic compounds, thermogravimetric analysis, and hygroscopic behavior through water adsorption isotherms.

Materials and methods

Raw material and processing

For this experiment, jenipapo fruits (*Genipa americana* L.) were obtained from the local market in João Pessoa, Paraíba, Brazil (7°07'08" S, 34°52'53" W), at the ripe stage, identified by the yellowish-brown color of the peel. After acquisition, the fruits were transported to the laboratory, where they were selected and sanitized in a sodium hypochlorite solution at 150 ppm for 10 min, followed by rinsing under running water.

After cleaning, the fruits were manually peeled, and the pulp, peels, and seeds were separated. The pulp was cut into 10-mm cubes using a manual aluminum cutter, while the peels and seeds were ground using a domestic blender (Arno®, Power Mix model, São Paulo, Brazil) at maximum

speed for 1 min. Each fraction was individually packaged in high-density polyethylene bags, vacuum-sealed, protected from light, and stored in a freezer (≈ -18 °C) until use in the experimental procedures.

It is important to note that changing the processing sequence (i.e., drying intact pieces of pulp, peels, and seeds prior to grinding, instead of applying the grind–dry–grind approach used in this study) may influence the retention of total phenolic compounds (TPC). Drying intact structures may reduce the exposed surface area, potentially limiting oxidative reactions, whereas pre-grinding increases surface exposure to heat and oxygen, which may accelerate phenolic degradation. However, no comparative treatment was performed in this work. Therefore, the results presented here are specifically associated with the processing conditions applied, and future studies are recommended to evaluate the effect of alternative drying–grinding sequences on TPC retention.

Drying of jenipapo pulp, peels, and seeds

The previously processed pulp, peels, and seeds of jenipapo were distributed in stainless steel trays, forming a thin layer of about 10 mm in height, and dried in a forced-air oven (Marconi®, model MA035, Piracicaba, Brazil) at 70 °C with an air velocity of 1.0 m s⁻¹ until reaching a moisture content of around 5%. This temperature was chosen based on preliminary tests, as it proved more effective in preserving bioactive compounds and required a shorter drying time. After drying, the samples were ground in a domestic blender (Arno®, Power Mix model, São Paulo, Brazil) at maximum speed for 5 min and sieved through a 40-mesh screen, resulting in flours from the pulp (FP), peels (FC), and seeds (FS).

Degradation kinetics of total phenolic compounds in jenipapo flours

The degradation kinetics of total phenolic compounds (TPC) in the flours were conducted under controlled temperature and relative humidity conditions. The FP, FC, and FS flours were weighed in triplicate and stored in low-density polyethylene plastic bags, with 0.08 g m⁻², thickness between 0.80 and 0.90 µm, and dimensions of 5 × 7 cm, similar to commercial flour packaging. The sealed bags containing the flours were placed in desiccators with a saturated sodium chloride (NaCl) solution, producing an environment with an average relative humidity of 75.32%, and in chambers with controlled temperatures of 20, 30, and 40 °C for a period of 40 days.

Every 10 days, TPC quantifications were performed according to the methodology described by Waterhouse [7]. Absorbance readings were taken at 765 nm using a spectrophotometer (Agilent Cary 60, Santa Clara, USA),

and a standard curve was prepared using gallic acid at a concentration of $100 \mu\text{g mL}^{-1}$. The results were expressed in mg of gallic acid equivalent per 100 g on a dry basis (mg GAE/100 g db).

The degradation kinetics curves of TPC in the flours as a function of storage time at different temperatures were plotted, and zero-order and first-order mathematical models (Table 1) were fitted to the experimental data using Origin/OriginPro (2024b) version 10.15 software. To evaluate the quality of the fits, the coefficient of determination (R^2) was used as a parameter.

To calculate the half-life ($t_{1/2}$) for the zero-order and first-order models, Eqs. (3) and (4) were used, respectively.

$$t_{1/2} = \left(\frac{A_0}{2k} \right) \quad (3)$$

$$t_{1/2} = \frac{0.693}{k} \quad (4)$$

To evaluate the effect of temperature on the reaction rate constant (k), an Arrhenius-type equation (Eq. 5) was applied. The k values used were from the model that best fit the experimental data of TPC degradation kinetics.

$$k = k_0 \exp \left(-\frac{E_a}{RT} \right) \quad (5)$$

where: k is reaction rate constant (1/day); k_0 pre-exponential factor (1/day); E_a activation energy (kJ mol^{-1}); R universal gas constant ($0.008314 \text{ kJ mol}^{-1} \text{ K}^{-1}$); and T absolute temperature (K).

To calculate the Q_{10} factor, Eq. (6) was used, which demonstrates the dependence of TPC changes on temperature variations. The k parameter from the best-fit model was used.

$$Q_{10} = \left(\frac{k_{(T+10)}}{k_T} \right) \quad (6)$$

where: k is reaction rate constant (1/day) and T absolute temperature (K).

The thermodynamic properties, enthalpy (ΔH), Gibbs free energy (ΔG), and entropy (ΔS), associated with the

TPC degradation kinetics were calculated according to Eqs. (7), (8), and (9), respectively [8].

$$\Delta H = E_a - R.T \quad (7)$$

$$\Delta G = -R.T \cdot \ln \left(\frac{K \cdot h_p}{K_B \cdot T} \right) \quad (8)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (9)$$

where: ΔH is differential enthalpy (J mol^{-1}); ΔS differential entropy ($\text{J mol}^{-1} \text{ K}^{-1}$); ΔG Gibbs free energy (J mol^{-1}); K_B Boltzmann constant ($1.38 \times 10^{-23} \text{ J K}^{-1}$); h_p Planck constant ($6.626 \times 10^{-34} \text{ J s}^{-1}$); and T absolute temperature (K).

Thermogravimetric analysis

The thermogravimetric curves (TG/DTG) and differential scanning calorimetry (DSC) analysis of the flours were obtained using a DTG 60H thermal analysis system from Shimadzu (Japan). Five mg of the samples was placed in alumina crucibles, with a heating rate of $10^\circ \text{C min}^{-1}$, in a nitrogen atmosphere, and subjected to a temperature range of 25 to 600°C . The onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c), and gelatinization enthalpy (ΔH) were measured throughout the gelatinization process. The data obtained were plotted using Origin/OriginPro (2024b) version 10.15 software, while the experimental data associated with thermodynamic properties were obtained from variance analysis (ANOVA) applying the F test of significance. The comparison between means was made using the Tukey test at a 5% probability level using Assistat software version 7.7 beta [9].

Fourier-transform infrared spectroscopy (FTIR)

The FTIR analyses were conducted using a Fourier-transform infrared spectrophotometer (PerkinElmer®, model Spectrum One, Waltham, USA). The spectra were generated with a resolution of 1 cm^{-1} , covering a spectral range of 500 to 4000 cm^{-1} . Data interpretation and graph preparation were performed using Origin/OriginPro (2024b) version 10.15 software.

Scanning electron microscopy (SEM)

The flour samples were coated with a fine layer of gold alloy under vacuum using an ion sputter coater G20. The morphological features of the samples were examined using a scanning electron microscope (SEM) (Tescan®, model VEGA3 SBH, Brno, Czech Republic) operating at 15 kV.

Table 1 Mathematical models used to estimate the degradation kinetics of total phenolic compounds totais

Model	Equation	Eq. No.
Zero-order	$A = A_0 - kt$	(1)
First-order	$\ln \left(\frac{A}{A_0} \right) = -kt$	(2)

A —concentration after a certain time; A_0 —initial concentration; k —reaction rate constant; t —time

Observations and images of the samples were captured at magnifications of 300 ×, 500 ×, and 1000 ×.

Water adsorption isotherms

The water adsorption data of the flours were obtained using the static indirect method proposed by Crapiste and Rotstein [10]. Water activity was measured using a water activity meter (Aqualab®, model 3TE, Pullman, USA) at temperatures of 20, 30, and 40 °C. The mathematical models presented in Table 2 were fitted to the data using Statistica 7.0 [11], through nonlinear regression by the quasi-Newton method.

To assess the quality of the fits to the experimental data, the coefficient of determination (R^2) and the mean percentage deviation (P), calculated according to Eq. (13), were used.

$$P = \frac{100}{n} \cdot \sum_{i=1}^n \frac{|(X_{\text{exp}} - X_{\text{pred}})|}{X_{\text{exp}}} \quad (13)$$

where: P is mean percentage deviation (%); X_{exp} experimentally obtained values; X_{pred} values predicted by the model; and n number of experimental data points.

Results and discussion

Degradation kinetics of phenolic compounds in jenipapo flours

Figure 1 presents the graphical representation of the degradation kinetics of total phenolic compounds in jenipapo pulp (FP), peel (FC), and seed (FS) flours over 40 days of storage in polyethylene packaging at temperatures of 20 °C, 30 °C, and 40 °C.

The highest initial phenolic compound levels were observed in FC, followed by FP, both exceeding 2000 mgGAE/100 g db and maintaining their relative positions throughout storage. FS had significantly lower values, below

1000 mgGAE/100 g db, but exhibited less pronounced losses over time. These reductions are attributed to the instability of phenolic compounds in response to heat and humidity, which accelerate oxidation reactions. Increased environmental humidity facilitates oxygen diffusion, enhancing oxidation processes that can be further intensified by heat [15, 16].

In percentage terms, FS exhibited the highest degradation rate, losing 40.62% of its initial phenolic content, followed by FP (36.34%) and FC (24.35%) at 40 °C. Conversely, the lowest losses occurred at 20 °C, with FC showing the greatest stability, retaining 81.22% of its initial phenolic content after 40 days, followed by FP (76.81%) and FS (66.29%). These results highlight FC's superior quality, as it not only contains higher phenolic concentrations but also demonstrates better stability over time.

To understand the degradation behavior of phenolic compounds over time, zero-order and first-order kinetic models were applied to the experimental data. The reaction rate constant (k) and half-life ($t_{1/2}$) values are presented in Table 3.

Across all three flours, the first-order model provided the best predictions, yielding the highest determination coefficients. The half-life times indicate slower degradation rates for FP at 20 °C and FC at 30 °C and 40 °C, with values ranging from 58 to nearly 100 days. However, in FS, even at 20 °C, the longest half-life was less than 45 days. These half-life values correlate with the first-order reaction rate constant (K), which reflects the degradation rate influenced by temperature. As expected, k values increased progressively with rising temperatures from 20 to 40 °C, a trend consistent with the principles of chemical kinetics [17]. Similar findings have been reported by Aprodu et al. [18] in a study on the degradation kinetics of anthocyanins extracted from purple corn flour, where increasing temperatures led to reduced half-life values and higher k constants.

Table 4 presents the activation energy, determination coefficient (R^2), and Q10 reaction coefficients for phenolic compound degradation in jenipapo flours.

Activation energy (E_a) is a key parameter indicating the sensitivity of a reaction to temperature. Higher activation energy values imply stronger temperature dependence, leading to slower reactions at lower temperatures and faster reactions at higher temperatures [19]. FP required significantly higher activation energy compared to FC and FS, suggesting greater thermal stability.

The Q10 coefficients, which indicate degradation rates over 10 °C temperature intervals, reveal more pronounced degradation between 20 and 30 °C. FP exhibited the highest Q10 values, while FC and FS showed similar values.

Table 5 presents the thermodynamic parameters associated with the degradation kinetics of phenolic compounds.

Positive ΔH and ΔG values indicate an endergonic degradation process, meaning that the reaction is non-spontaneous and kinetically controlled [21]. In contrast, the negative ΔS

Table 2 Mathematical models used to fit water adsorption data

Designation	Model	Eq.
Oswin [12]	$X_{\text{eq}} = a \left(\frac{a_w}{1-a_w} \right)^b$	(10)
GAB [13]	$X_{\text{eq}} = \frac{X_m C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)}$	(11)
Peleg [14]	$X_{\text{eq}} = k_1 \cdot a_w^{n_1} + k_2 \cdot a_w^{n_2}$	(12)

a_w —water activity (dimensionless); X_{eq} —equilibrium moisture content (% db); X_m —monolayer moisture content (% db); C —constant related to the heat of sorption of the molecular layer; a , b , c , K , k_1 , k_2 , n_1 , and n_2 —model constants

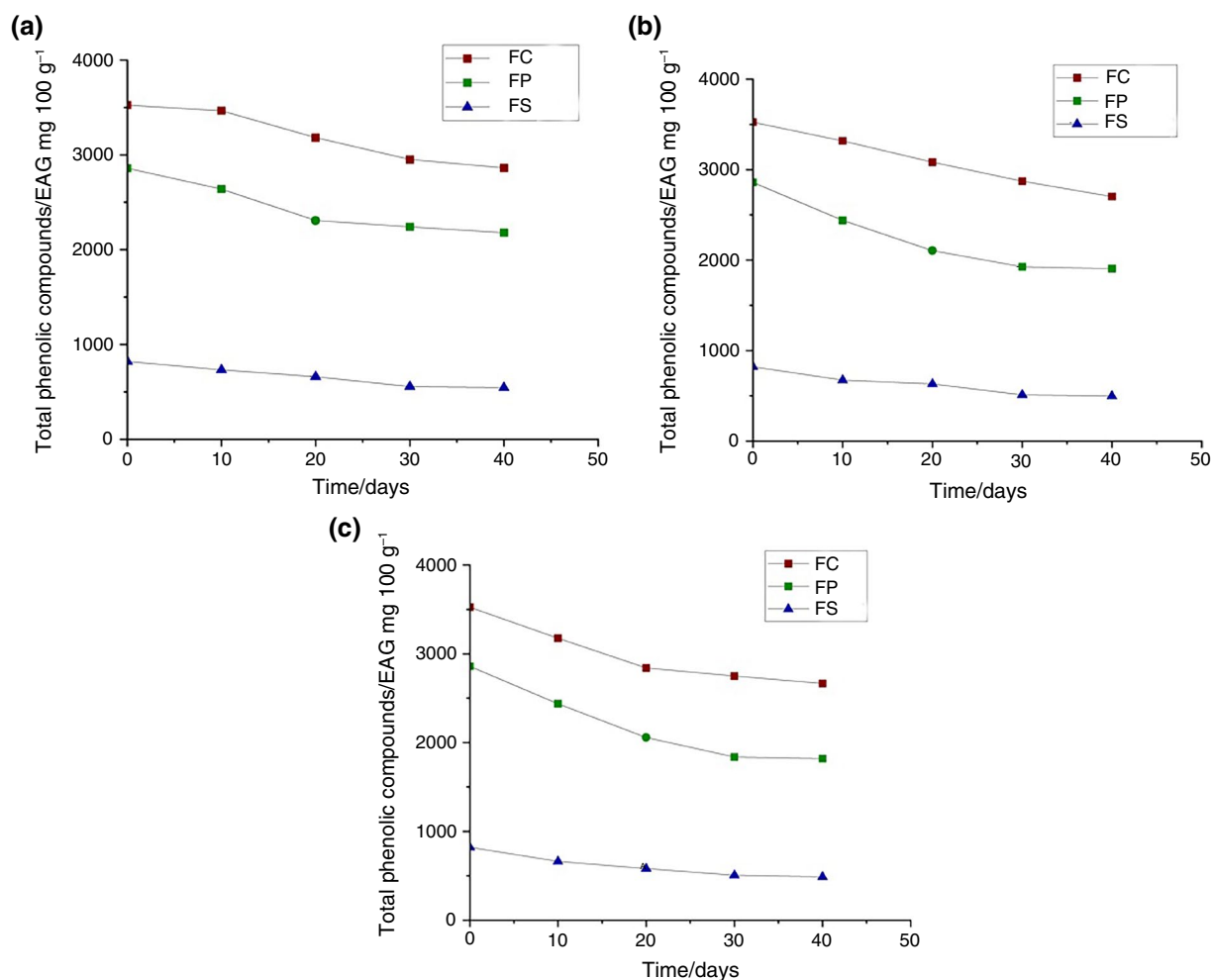


Fig. 1 Degradation kinetics of total phenolic compounds (mgGAE/100 g db) in genipap pulp (FP), peel (FC), and seed (FS) flours over 40 days of storage at temperatures of 20 °C (a), 30 °C (b), and 40 °C (c). Standard deviations across replicates were small ($\leq 1\%$

of the mean), indicating data consistency. Due to overlapping values, these deviations are not shown in this figure to maintain graphical readability

values suggest a transition state with reduced structural freedom, leading to decreased reactivity and increased time required for complex formation. A similar behavior was observed by Silva et al. [22] in germinated peanut flours. These thermodynamic properties help explain differences in the degradation behavior of phenolic compounds observed at lower temperatures, since the structural organization and thermal resistance of matrix components influence their exposure and susceptibility to oxidation during storage. Therefore, the endothermic characteristics and energy transitions described here are directly related to the kinetic and hygroscopic responses evaluated in this study.

Thermogravimetric analysis

Figure 2 displays the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves for the genipap

flours. Overall, all three samples exhibited similar behavior, with three distinct mass loss regions. The flours showed exothermic characteristics typical of food products, where moisture loss occurs with increasing temperature, followed by the onset of thermal decomposition beyond 400 °C. Similar patterns were observed by Athma-selvi et al. [23] in lyophilized tropical fruit flours, where three thermal events were identified in lyophilized guava (*Psidium guajava* L.) and sapodilla (*Manilkara zapota*) powders. Although storage temperatures used in the phenolic degradation and isotherm studies were substantially lower than the thermal ranges evaluated through TGA/DTG, these results provide essential structural information. The number and magnitude of mass loss events are linked to the chemical nature of the matrix, which contributes to explaining the differences in phenolic stability and water sorption capacity among the flours.

Table 3 Parameters of zero-order and first-order models, determination coefficients (R^2), and half-life times ($t_{1/2}$) for total phenolic compounds in genipap flours stored at 20 °C, 30 °C, and 40 °C

FP					
Model	$T/^\circ\text{C}$	Parameters		$R^2/\%$	$t_{1/2}/\text{day}$
		A_0	k		
Zero-order	20	2796.2	17.54	90.73	79.59
	30	2730.4	24.17	89.92	56.47
	40	2734.4	26.77	91.18	51.13
First-order	20		0.0071	91.79	97.61
	30		0.0105	92.07	66.00
	40		0.0119	93.17	58.24
FC					
Model	$T/^\circ\text{C}$	Parameters		$R/\%$	$t_{1/2}/\text{day}$
		A_0	k		
Zero-order	20	3564.5	18.4	96.01	97.0
	30	3517.7	20.9	99.72	84.2
	40	3419.5	21.4	90.87	79.8
First-order	20		0.0082	96.13	84.5
	30		0.0096	99.88	72.2
	40		0.0100	92.43	69.3
FS					
Model	$T/^\circ\text{C}$	Parameters		$R/\%$	$t_{1/2}/\text{day}$
		A_0	k		
Zero-order	20	809.3	7.29	96.28	55.5
	30	789.3	8.10	93.36	48.7
	40	777.5	8.25	91.70	47.1
First-order	20		0.0156	96.69	44.4
	30		0.0182	94.99	38.1
	40		0.0188	94.86	36.9

Table 4 Activation energy (E_a), determination coefficients (R^2), and Q_{10} reaction coefficients for the degradation of total phenolic compounds in genipap flours

Flours	$E_a/\text{kJ mol}^{-1}$	R^2	Temperature coefficient/ Q_{10}	
			(20–30 °C)	(30–40 °C)
FP	19.81	0.92	1.4789	1.1333
FC	7.61	0.90	1.1707	1.0417
FS	7.16	0.89	1.1667	1.0330

The first region exhibits a lower mass loss, ranging from 13.27% (FS) to 9.25% (FC), an initial mass reduction associated with the loss of absorbed water in the temperature range of 120–260 °C. The second mass loss event, occurring between 201 and 351 °C, may be related to a more complex volatilization process resulting from the degradation of hemicellulose and cellulose, which are thermally less stable

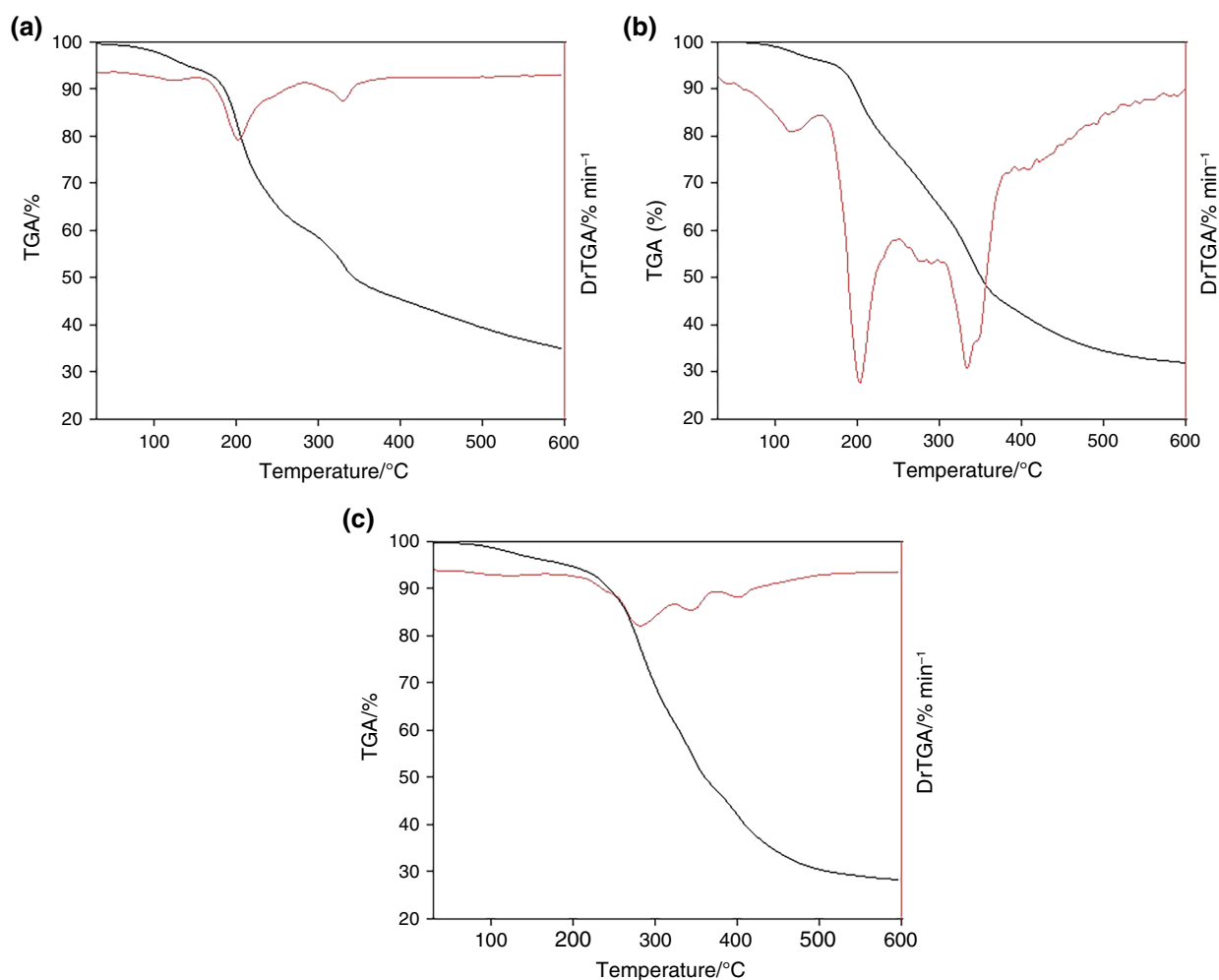
than lignin, as well as the degradation of proteins, lipids, fibers, and starch [20]. The third event, observed above 351 °C up to 600 °C, represents the complete degradation of the material, involving the cleavage of C–O and C–C bonds, releasing CO and CO₂, leading to the formation of a graphite layer and the release of fixed mineral residues. Additionally, dehydration, demethoxylation, and decarboxylation reactions may occur in compounds such as pectin and similar polysaccharides [24].

Table 6 summarizes the estimated temperature ranges and mass loss percentages for each thermal degradation event in the genipap flours.

For all three events, the highest mass losses were recorded in FS flour, followed by FC and FP. This pattern suggests that FS flours are more susceptible to thermal degradation compared to FC and FP. Additionally, in all the studied flours, the lowest mass losses occurred during the first degradation event, followed by the second and

Table 5 Activation enthalpy (ΔH), activation entropy (ΔS), and Gibbs free energy of activation (ΔG) for the degradation kinetics of total phenolic compounds in jenipapo flours

Flours	Temperature/ $^{\circ}\text{C}$	$\Delta H/\text{kJ mol}^{-1}$	$\Delta S/\text{J mol}^{-1} \text{K}^{-1}$	$\Delta G/\text{kJ mol}^{-1}$
FP	20	17.375	-0.2266	83.813
	30	17.292	-0.2259	85.770
	40	17.209	-0.2272	88.358
FC	20	5.173	-0.2671	83.462
	30	5.090	-0.2669	85.996
	40	5.006	-0.2676	88.811
FS	20	4.723	-0.2632	81.894
	30	4.640	-0.2631	84.384
	40	4.556	-0.2638	87.167

**Fig. 2** Thermogravimetric (TGA) and derivative thermogravimetric (DTG) curves for jenipapo pulp (FP), peel (FC), and seed (FS) flours

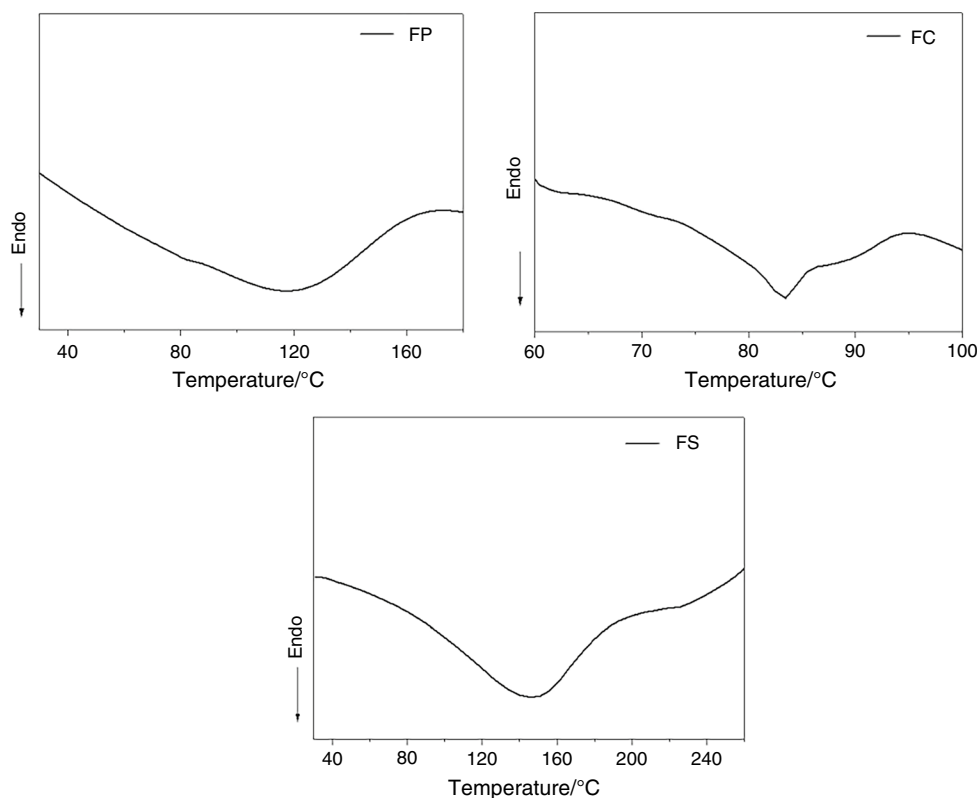
third events. This indicates that lower temperatures (first event) are already sufficient to initiate significant degradation in the flours, while higher temperatures (third event) lead to greater mass losses, mainly due to the sensitivity of organic matter to heat, resulting in increased degradation of its components. Flours exhibiting lower thermal

stability, particularly FS, also demonstrated higher phenolic degradation rates and less hygroscopic stability during storage, suggesting that the extent of matrix decomposition is functionally related to chemical reactivity and water interaction capacity.

Table 6 Temperature ranges and mass loss percentages during the thermal degradation of genipap flours

Flour	Event	Temperature/°C	Mass loss/%
FP	First	161–201	12.25
	Second	201–301	29.01
	Third	331–600	33.1
FC	First	120–202	9.25
	Second	202–331	35.91
	Third	331–600	42.93
FS	First	188–270	13.27
	Second	270–351	36.45
	Third	351–600	45.63

Figure 3 represents the heat flow curves obtained from differential scanning calorimetry (DSC) analyses of genipap flours.

Fig. 3 Heat flow curves obtained from differential scanning calorimetry (DSC) analyses of genipap flours from pulp (FP), peel (FC), and seeds (FS)**Table 7** Thermal properties of gelatinization of genipap flours

Parameter	Farinhas		
	FP	FC	FS
Initial temperature— T_0 /°C	84.89 ± 0.55 a	73.01 ± 0.33 b	34.80 ± 0.57 c
Peak temperature— T_p /°C	121.03 ± 0.69 b	83.49 ± 0.79 c	147.37 ± 0.49 a
Conclusion temperature— T_c /°C	162.63 ± 0.33 b	86.49 ± 0.61 c	198.76 ± 0.84 a
Gelatinization enthalpy— $\Delta H/J\ g^{-1}$	1.35 ± 0.28 c	3.07 ± 0.86 b	11.94 ± 1.15 a

The DSC curves show the formation of endothermic peaks in all three flours during the gelatinization phenomenon, revealing a broad apparent endothermic peak. According to Ćujić Nikolić et al. [25], these broad peaks may be associated with the evaporation of absorbed water, structural water, and volatile compounds from the material. Similar broad endothermic peaks were also reported by Santos et al. [26] in dried carrot cubes. These calorimetric transitions complement the thermogravimetric results, reinforcing the interpretation that thermal stability and crystalline organization within the flours influence molecular mobility and water affinity. Consequently, DSC data provide structural evidence that supports the observed kinetic and hygroscopic behavior.

Table 7 presents the transition temperatures and enthalpies for the gelatinization processes of the flours.

The high values of initial, peak, and conclusion temperatures (T_0 , T_p , and T_c) in the three flours indicate their high thermal stability. Although FS had the lowest initial

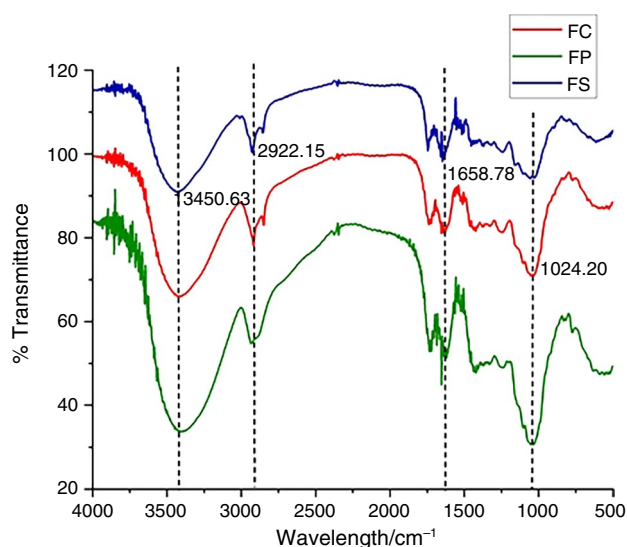


Fig. 4 Fourier-transform infrared spectroscopy (FTIR) of genipap flours from pulp (FP), peels (FC), and seeds (FS)

temperature, its peak and conclusion temperatures were the highest among the three. Peaks at higher temperatures may result from the low water content in the flours, leading to the formation of an amylose–lipid complex [27, 28]. Elevated initial gelatinization temperatures, ranging from 41.87 to 98.66 °C, were also reported by Moraes et al. [29] in germinated pumpkin seed flours of the Coroa, Jacarezinho, and Italian varieties, associated with the thermal processing applied to the samples.

FS showed the highest ΔH value (11.94 J g⁻¹), with a significant reduction when compared to FP (1.35 J g⁻¹) and FC (3.07 J g⁻¹), a difference attributed to the distinct physicochemical composition and physical structure of the different genipap parts. According to Xu et al. [30], high gelatinization enthalpy values may be due to the starch granules present in the flours, which exhibit greater crystallinity and molecular order. Akhila et al. [31] reported a gelatinization enthalpy close to that of the pulp and peel, at 2.72 J g⁻¹, in Hausa potato (*Plectranthus rotundifolius*) flour obtained after convective drying at 40 °C.

The lower ΔH values in FP and FC are beneficial for food processing, as they result in reduced energy consumption in heat-involving processes, facilitating production and improving product quality [26].

Fourier-transform infrared spectroscopy (FTIR)

Figure 4 presents the graphical representation of the Fourier-transform infrared spectroscopy (FTIR) spectra of flours obtained from the pulp, peels, and seeds of genipap.

FTIR analysis allowed the identification of functional groups present on the surface of the flours. The samples exhibited similar behavior, with absorption peaks occurring in the same spectral ranges.

The peaks observed at 3450.63 cm⁻¹ correspond to the stretching vibration of –OH groups, indicating the presence of hydroxyl groups. This region is associated with complex vibrational bands involving both free intermolecular and intramolecular hydroxyl groups, influencing the carbohydrate structure [32]. The peaks at 2922.15 cm⁻¹ can be attributed to the asymmetric –CH group vibration, suggesting the presence of lipids [29].

The peak at 1658.78 cm⁻¹ characterizes the –COO stretching vibration, related to the amide I band, a sensitive spectral region reflecting the secondary structural organization of proteins. This band is typical of peptide bonds and functional group stretching vibrations [33].

Finally, peaks around 1024 cm⁻¹ indicate an intense stretching of the –C–O group, which may be related to the vibrations of alcohols and primary and secondary ethers in the polymeric chains of starch, confirming its presence in the analyzed flours [34].

A similar pattern was observed by Moraes et al. [29] with peaks in the 1030 cm⁻¹ band in germinated pumpkin seed flour of the Jacarezinho variety, associated with the presence of starch in the flours.

Scanning electron microscopy (SEM)

SEM evaluation was conducted not only to describe flour morphology but also to investigate structural traits associated with phenolic degradation susceptibility and water adsorption capacity, supporting the primary objectives of this work.

The microstructural characterization of genipap flours, performed using Scanning Electron Microscopy (SEM), is presented in Figs. 5–7, corresponding to the flours from pulp (FP), peels (FC), and seeds (FS), respectively. In general, a similar microstructural behavior was observed among the flours, evidenced by visually comparable structures. This similarity may be attributed to all samples undergoing the same convective drying process under identical temperature conditions, indicating that, despite differences in chemical composition, their external structures appeared visually alike.

At 300× magnification, particles of varying sizes can be identified, characterizing a heterogeneous granular distribution. This granularity is a typical feature of dried polymeric raw materials, with the formation of agglomerates occurring during the drying process.

At 500× magnification, the rough surface of the particles becomes more evident, marked by irregularities caused by convective drying. This process contributes to the formation

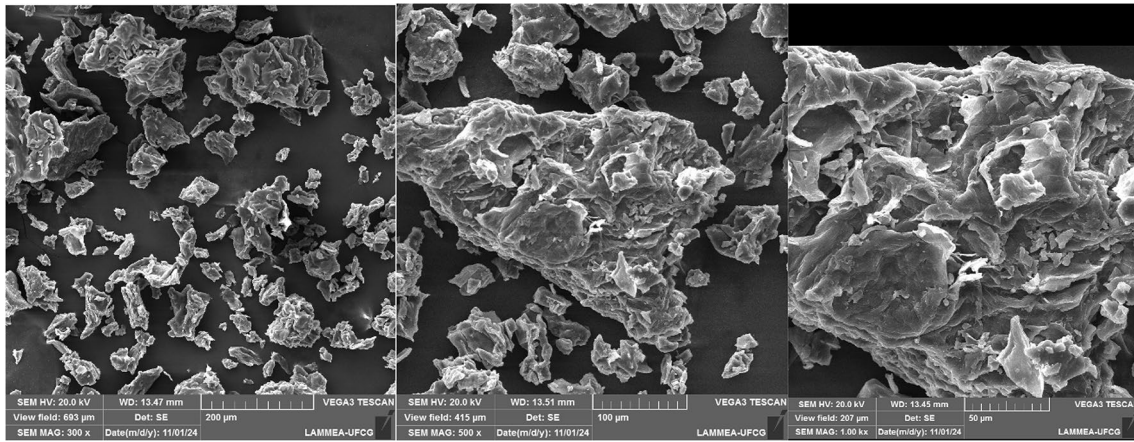


Fig. 5 Scanning electron microscopy of genipap pulp flour (FP) obtained by convective drying: **a** 300× magnification; **b** 500× magnification; **c** 1000× magnification

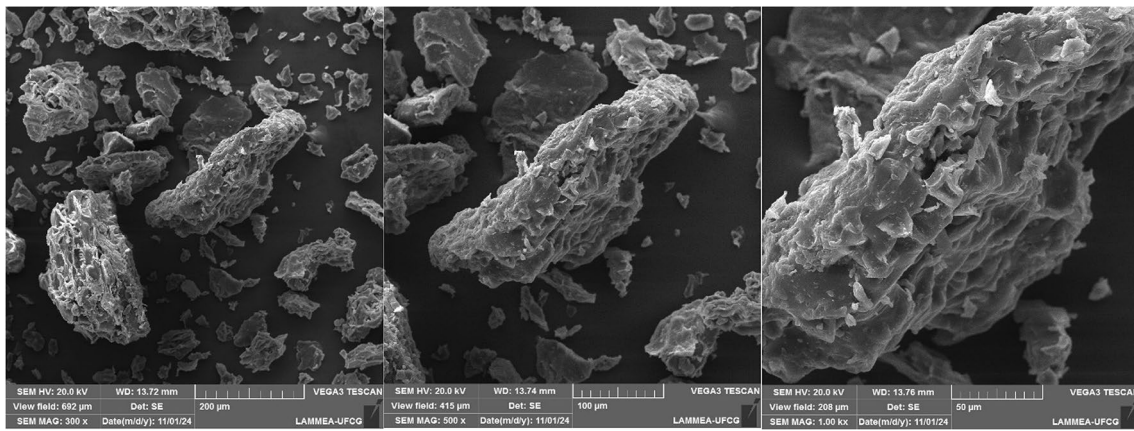


Fig. 6 Scanning electron microscopy of genipap peel flour (FC) obtained by convective drying: **a** 300× magnification; **b** 500× magnification; **c** 1000× magnification

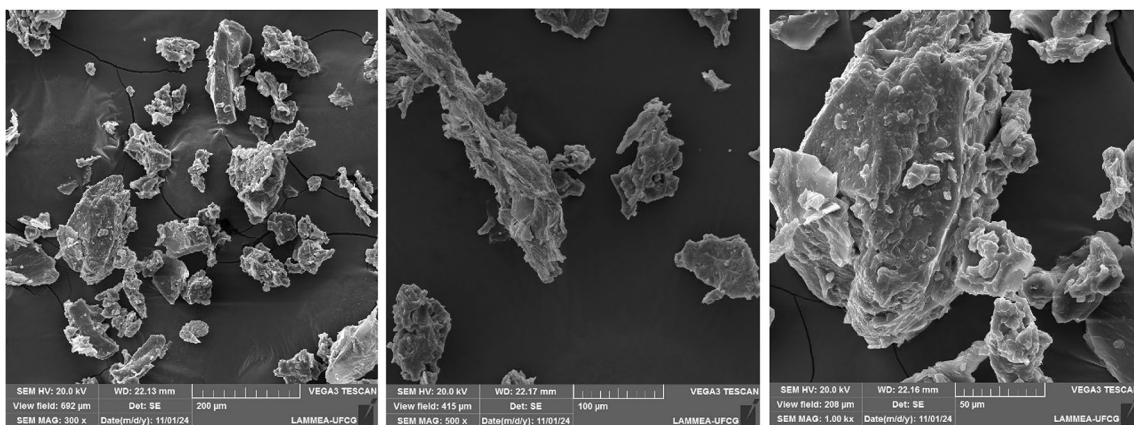


Fig. 7 Scanning electron microscopy of genipap seed flour (FS) obtained by convective drying: **a** 300× magnification; **b** 500× magnification; **c** 1000× magnification

Table 8 Parameters, coefficients of determination (R^2), and mean percentage deviations (P) of models fitted to the experimental water adsorption data of genipap flours at 20, 30, and 40 °C

FP						
Model	$T/^\circ\text{C}$	Parameters		R^2	$P/\%$	
		a	B			
Oswin	20	9.1236	0.5760	0.9965	5.08	
	30	8.4497	0.5207	0.9960	8.38	
	40	11.072	0.4553	0.9935	7.21	
GAB	20	6.6922	3.0347	0.9992	1.86	
	30	5.2647	4.8332	0.9971	3.86	
	40	6.2937	8.8710	0.9990	1.75	
Peleg	20	20.788	1.2584	35.064	8.4132	0.9990
	30	38.761	17.615	24.929	1.6491	0.9981
	40	38.544	10.022	21.784	1.0549	0.9987
FC						
Model	$T/^\circ\text{C}$	Parameters		R^2	$P/\%$	
		a	B			
Oswin	20	8.7855	0.6704	0.9950	6.90	
	30	10.309	0.6513	0.9990	4.40	
	40	9.1236	0.5760	0.9972	4.26	
GAB	20	6.9427	2.1081	0.9257	0.9963	3.75
	30	7.5558	2.7950	0.9279	0.9996	1.46
	40	7.3743	3.2740	0.9331	0.9988	2.60
Peleg	20	46.948	8.9184	22.918	1.4573	0.9954
	30	57.800	11.091	29.256	1.4986	0.9995
	40	28.304	1.4372	61.262	10.816	0.9991
FS						
Model	$T/^\circ\text{C}$	Parameters		R^2	$P/\%$	
		a	B			
Oswin	20	8.2862	0.6782	0.9932	4.25	
	30	8.4497	0.5207	0.9960	8.38	
	40	11.072	0.4553	0.9935	7.21	
GAB	20	8.2921	8.6463	0.9655	0.9980	3.90
	30	10.628	2.1926	0.9037	0.9970	4.58
	40	6.6922	3.0347	0.8988	0.9992	1.86
Peleg	20	28.214	1.6807	16.8391	2.5216	0.9979
	30	38.761	17.615	24.929	1.6491	0.9981
	40	38.544	10.022	21.784	1.0549	0.9987

Bold values indicate the best-fitting model

of a rigid film around the particles, which can directly impact the technological properties of the flours, such as solubility and dispersibility in aqueous systems.

At the maximum magnification of 1000 $\times$, finer details of particle morphology can be observed, including fissures and pores that are more noticeable than at lower magnifications. These structural defects suggest weaknesses in the uniformity of the powders formed during drying, indicating the need for process adjustments to improve formation, increasing uniformity and resistance [35]. The SEM results are relevant because microstructural characteristics influence both phenolic compound stability and water adsorption behavior. Porosity, particle size distribution, and surface roughness affect oxygen diffusion, moisture accessibility, and surface area exposure, which may contribute to the different rates of TPC degradation and hygroscopic responses observed among the flours. In this context, the higher porosity and surface irregularities identified in FC and FS may help explain their greater susceptibility to structural interaction with oxygen and moisture when compared to FP, supporting the interpretation of kinetic and isotherm results.

Water adsorption isotherms

Table 8 presents the fitting parameters of mathematical models to the water adsorption data at 20, 30, and 40 °C for genipap flours, as well as the coefficients of determination (R^2) and mean percentage deviations (P).

Good fits to the experimental data ($R^2 > 0.99$ and $P < 10\%$) were observed for the Oswin, GAB, and Peleg models. Mean percentage deviations below 10% indicate high accuracy of the water adsorption isotherms, according to Lomauro et al. [36]. Among these models, the GAB model demonstrated the best performance, with R^2 values above 0.996 and P values below 4.59. It is common for the GAB model to provide the most precise fits, as reported in studies by Hawa et al. [37] on cassava flour (*Manihot esculenta*) and Omole et al. [38] on breadfruit flour (*Artocarpus altilis*).

Based on the parameters obtained from the GAB model, all flours can be classified as Type II adsorption isotherms according to Blahovec [39], since the GAB model constant

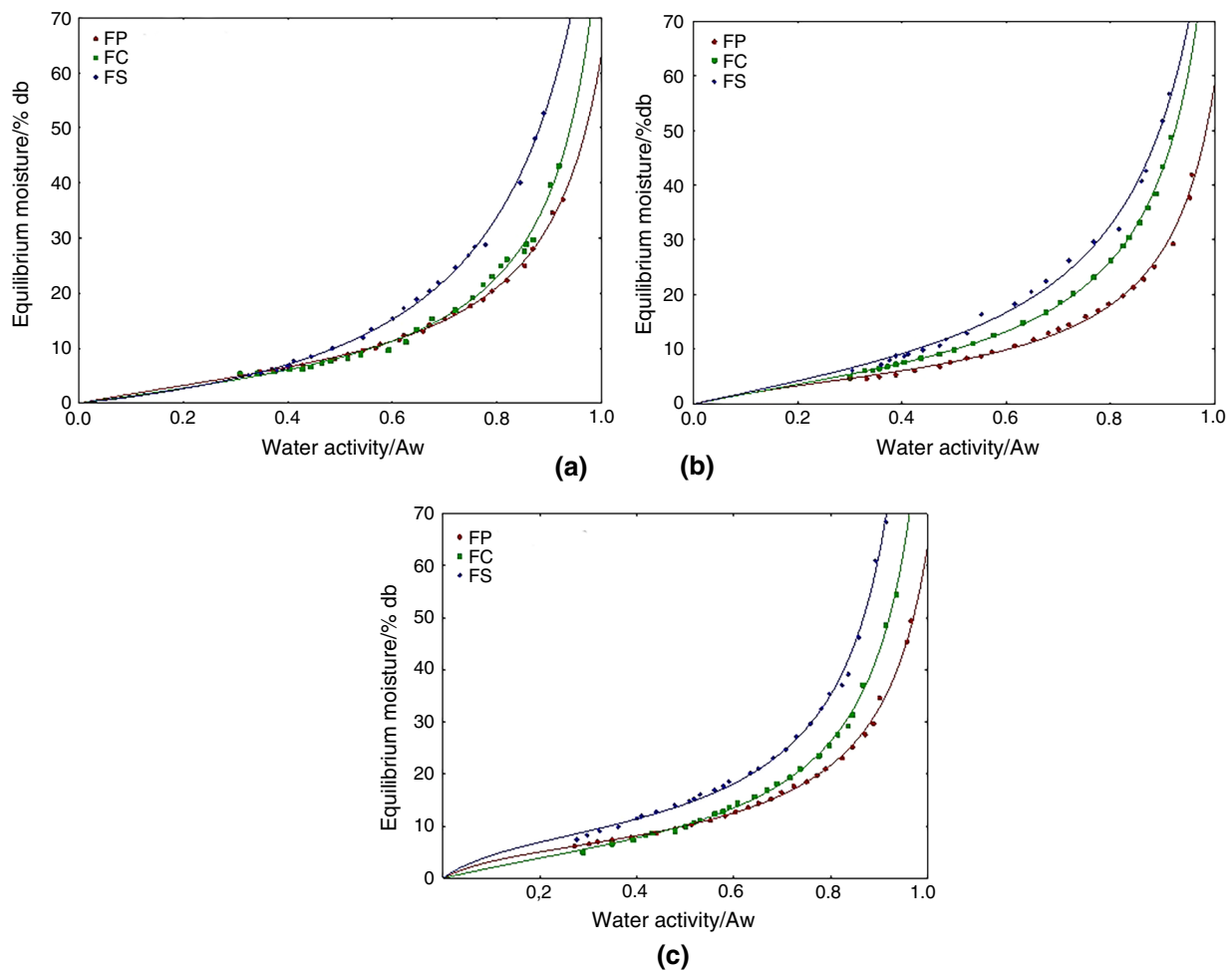


Fig. 8 Water adsorption isotherms of genipap flours from pulp (FP), peels (FC), and seeds (FS) fitted using the GAB model at temperatures of 20 °C (a), 30 °C (b), and 40 °C (c)

K falls between 0 and 1, while C remains above 2. This type of isotherm is associated with multilayer adsorption, commonly observed in macroporous adsorbents.

The monolayer moisture content (X_m) from the GAB model indicates the amount of strongly adsorbed water, a critical value for product stability. The observed values, ranging from 5.26 to 10.63, suggest that the material has a good water retention capacity, which may benefit the stability of bioactive compounds and nutrients [40].

In the Oswin model, the “a” parameter values showed an increasing trend, while “b” values decreased with rising temperature. According to Blahovec [39], this behavior is significant as it indicates the absence of an inflection point in the curve. The lack of concavity changes in the functions demonstrates that these parameters exhibit both mathematical and physical consistency.

Figure 8 presents the adsorption isotherms of genipap pulp, peel, and seed flours fitted using the GAB model.

At all temperatures, FS flour reached the highest equilibrium moisture contents, while pulp flour consistently showed the lowest values, occasionally coinciding with FC at some water activity levels below 0.6 at 20 and 40 °C. This behavior can be attributed to structural similarities between the pulp and peel, which differ from the seed, characterized by a more rigid and fibrous structure. A similar trend was observed by Mallek-Ayadi et al. [41], where increasing Aw promoted an increase in equilibrium moisture content in melon seed (*Cucumis melo* L.) flours.

Conclusions

Peel flour (FC) had the highest phenolic content, exceeding 3000 mg GAE/100 g dry basis, while seed flour (FS) had lower phenolic levels, with its initial content surpassed by the other flours after 40 days of storage. The first-order model showed that pulp flour (FP) had the longest half-life, approximately 100 days at 20 °C. Peel flour (FC) exhibited longer half-life values at 30 and 40 °C, with the highest value reaching 70 days at 40 °C, while seed flour (FS) had a half-life of 36.9 days at 40 °C. The temperature coefficients (Q_{10}) indicated that degradation was more significant between 20 and 30 °C, especially in pulp flour, which also had the highest activation energy.

Thermogravimetric analysis revealed mass loss events associated with dehydration, decomposition, carbonization, and the formation of fixed mineral residue, with the most significant mass loss occurring during the third event. FTIR analysis confirmed the presence of starch, lipids, and proteins in all flours, showing structural similarity. The water adsorption data were best fitted by the GAB model, classifying the flours as Type II isotherms. Peel flour's high phenolic content and stability suggest it can reduce

agricultural byproduct waste and enhance the functional and nutritional properties of food products, making it a promising ingredient for various applications.

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Author contributions Juvêncio Olegário de Oliveira Neto conceived the study, designed the experimental protocol, conducted all laboratory analyses, performed data treatment and interpretation, prepared the figures and tables, and wrote the full manuscript draft. Alexandre José de Melo Queiroz and Rossana Maria Feitosa de Figueirêdo supervised all stages of the research, provided scientific guidance throughout the study, contributed to the interpretation of results, and led the critical revision and refinement of the manuscript. The remaining co-authors (Francislaine Suelia dos Santos; Yaroslávia Ferreira Paiva; Josivanda Palmeira Gomes; Mailson Gonçalves Gregório; Michael Marcos de Aquino Gomes; Morgana Aragão Araújo; Nailton de Macedo Albuquerque Junior; Juane Queiroz Farias; Henrique Valentim Moura; Eugenia Telis de Vilela Silva) contributed to specific components of the study, including sample preparation, instrumental analysis support, data interpretation, and technical revision of the final manuscript version.

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