

Article

Impact of Water Activity on Physical Stability and Bioactive Compound Retention in Yellow Pitaya (*Selenicereus megalanthus*) Pulp Powder

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Abstract

Yellow pitaya (*Selenicereus megalanthus*) pulp is rich in phenolic compounds with antioxidant capacity and exhibits desirable sensory properties. Dehydration and grinding into powder may enhance stability and broaden the potential for export and industrial applications. In this study, freeze-drying was used to obtain yellow pitaya pulp powder, which was stored at 20 °C under different water activity levels (a_w 0.113–0.750). Changes in physical properties (water sorption, glass transition, texture, and color) and bioactive compounds (antioxidant capacity and phenolic content) were assessed after 3 months of storage. Combining the Gordon & Taylor model with the GAB sorption isotherm, the critical water content (CWC) and water activity (CWA) related to glass transition were determined as 0.023 g water/g product and 0.110, respectively. Below these critical values, the glassy state of pitaya pulp powder was maintained, enhancing its quality and stability during storage. The greatest changes in color and bioactive compound content were observed at high a_w levels (0.680 and 0.750, respectively). Due to its high nutritional value and antioxidant properties, this powder can be incorporated into formulations or dietary supplements, offering additional functional benefits and expanding its application in the food industry.

Keywords: powder stability; critical water activity; critical water content; physical properties; antioxidant capacity

1. Introduction

Pitaya, a xerophilic plant of the *Cactaceae* family, is native to Central and South America. Its fruit, commonly known as dragon fruit or by the same name as the plant, pitaya, has been consumed since pre-Columbian societies [1]. Pitaya consumption in Europe has gained popularity in recent years due to its exotic appearance, extravagant shape, and striking colors of both skin and pulp, which are the first to catch the attention of potential consumers. There are several varieties of pitaya fruit, including red and yellow. The yellow pitaya (*Selenicereus megalanthus*) has a sweet and aromatic taste and viscous, aqueous, marbled-colored flesh containing small black seeds rich in protein and essential fatty acids. The fruit has an oblong shape with characteristic scaly structures on its peel, which develop a yellow color as they ripen [2].

Yellow pitaya is rich in dietary fiber, minerals such as calcium, magnesium, and potassium, and vitamin C, contributing to its nutritional and functional value. Its main bioactive compounds (phenolic compounds, betalains, and carotenoids such as β -carotene



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and lycopene) are primarily responsible for its antioxidant properties [3–6]. These bioactive compounds are more abundant in the peel than in the pulp and seeds [7]. Numerous studies have linked these compounds with proven beneficial effects on health, acting as antioxidants against free radicals [8] besides preventing alterations in the immune system and a wide range of diseases, including Alzheimer’s disease, Parkinson’s disease, and cancer [6,9–13].

Yellow pitaya production is seasonal (February–March and July–August), often resulting in overproduction and substantial post-harvest losses. To mitigate these losses, several products have been developed, including pitaya water, juice, jams, and liqueurs [14]. The export of yellow pitaya is an important outlet for the large volumes of production in countries such as Colombia. Between 2011 and 2020, Colombia exported nearly 40,000 tons of pitaya to countries worldwide, including Brazil, Hong Kong, France, Spain, Singapore, and Germany [15]. The development of powdered products represents a strategic innovation, enabling the diversification of pitaya-derived products while providing considerable benefits, including improved efficiency in transportation, export, storage, and extended shelf life, enhancing the overall value chain of this tropical fruit.

Freeze-drying is widely recognized as the most effective technique for producing high-quality fruit powders because of its capacity to preserve the nutritional, structural, and sensory attributes of the original fruit. From an industrial perspective, spray-drying is generally more scalable and cost-effective; however, it requires the addition of carrier agents to obtain a stable powder, which may not be desirable for clean-label or high-fruit-content products. Hot-air drying, while simpler, often fails to sufficiently reduce moisture content and makes it difficult to obtain a free-flowing powder, particularly for sugar-rich tropical fruits, while also promoting the degradation of heat-sensitive bioactive compounds. Freeze-drying, based on the sublimation of frozen water content under low pressure, minimizes the degradation of bioactive compounds and helps retain the natural color, flavor, and aroma of the product. Studies have demonstrated that freeze-dried powders exhibit superior rehydration capacity, stability, and shelf life compared with those obtained using conventional drying methods [16]. This makes freeze-drying an excellent method for creating value-added powdered products from tropical fruits, such as pitaya, ensuring their quality and market competitiveness.

Despite their advantages, freeze-dried products are prone to degradation because of their hygroscopic and thermoplastic properties. Fruit dehydration leads to the formation of an amorphous matrix, which, at ambient temperature, may undergo a transition from a glassy to a rubbery state. This behavior is mainly attributed to the high carbohydrate content of fruits, which lowers the glass transition temperature (T_g) [17]. Transition to the rubbery state is associated with increased molecular mobility, which can promote solute crystallization and the development of physical instability phenomena, such as powder caking. In addition, diffusion-driven reactions are accelerated, leading to quality deterioration, which can render the powdered product unacceptable [18]. Furthermore, fluctuations in relative humidity (RH) during storage can significantly alter the water activity, resulting in additional changes in water content and contributing to the degradation of product quality [19–21].

Although studies on pitaya drying exist, limited information is available on the stability and physicochemical behavior of yellow pitaya pulp powder, particularly regarding critical factors such as a_w , moisture content, and T_g . While extensive research has explored the relationship between these factors in other fruit powders, there is a lack of experimental data for yellow pitaya pulp powder, a tropical fruit with a unique composition and bioactive profile.

In this context, this study aimed to assess the influence of water activity on the physical properties and retention of bioactive compounds in freeze-dried yellow pitaya pulp powder after 3 months of storage. The water sorption isotherm and the plasticizing effect of water were characterized to obtain the critical water activity and the critical water content associated with the glass transition, which mark the thresholds beyond which undesirable physical changes and quality loss may occur. By understanding these critical factors, this study aims to guide for optimizing the production and storage conditions of high-quality, stable yellow pitaya powder with enhanced nutritional and functional properties, which may serve as a versatile ingredient in the formulation of functional foods, beverages, and nutraceutical products, expanding the industrial applications of the yellow pitaya.

2. Materials and Methods

2.1. Sample Preparation

Yellow pitaya (*Selenicereus megalanthus*) fruits from Colombia were purchased from Mercat Central of Valencia (Spain). The fruits were peeled, and the pulp, including the seeds, was obtained. The pulp was cut transversally into 10 mm thick slices and freeze-dried in a Telstar Lioalfa-6 Lyophiliser (Telstar, Azbil Group, Terrassa, Spain) at $-40\text{ }^{\circ}\text{C}$ and 10^{-2} Pa for 48 h, yielding a dry material with no visible signs of collapse. The freeze-dried slices were weighed to determine the freeze-drying efficiency. Subsequently, the dried slices were ground to obtain a free-flowing pitaya pulp powder. Samples (15–20 g) were placed in triplicate in aluminum cups and stored in the dark at $20\text{ }^{\circ}\text{C}$ in hermetically sealed chambers (5 L). Different saturated salt solutions (LiCl , CH_3COOK , MgCl_2 , K_2CO_3 , $\text{Mg}(\text{NO}_3)_2$, NaNO_2 , and NaCl) were used to control the RH in each chamber and to obtain, at thermodynamic equilibrium, pitaya pulp powder with a_w of 0.113, 0.230, 0.330, 0.430, 0.520, 0.680, and 0.750, respectively [22]. These water activity levels were selected to cover a broad range of moisture conditions and to explore critical thresholds relevant for powder processing, handling, and storage. The salt solutions were maintained in a supersaturated state throughout the storage period to ensure constant water activity. The mass of each sample was monitored over time, and the equilibrium was considered to be achieved when the samples reached constant weight ($\Delta m < \pm 0.0005\text{ g}$), which required between 2 and 4 weeks. At equilibrium, the a_w of each sample was assumed to correspond to that of the saturated salt solution, equal to $\text{RH}/100$.

2.2. Soluble Solid Content (SSC)

The SSC ($^{\circ}\text{Brix}$) was measured directly from the fresh pulp using a portable refractometer (RFM330+, Bellingham and Stanley Ltd., Kent, UK) at $20\text{ }^{\circ}\text{C}$. Measurements were performed in triplicate.

2.3. Moisture

Moisture content was determined in the freeze-dried sample according to the AOAC (2000) method for sugar-rich foods, using a vacuum oven (Vacuoterm, J.P. Selecta, Barcelona, Spain) at $60\text{ }^{\circ}\text{C}$ and an analytical balance (AE 100, Mettler Toledo, Greifensee, Switzerland). The moisture content of the samples equilibrated at different relative humidities was calculated based on the initial moisture content and the weight difference after equilibrium was reached in each chamber. Measurements were performed in triplicate.

2.4. Sorption Isotherms

The BET [23] (Equation (1)), GAB [24] (Equation (2)), Henderson [25] (Equation (3)), and Caurie [26] (Equation (4)) sorption models were used to fit the relationship between

water content (w_e , g water/g solids) and a_w at 20 °C. Nonlinear regression analysis was performed using the Solver tool in Excel 2019 to estimate the parameters of each model.

$$w_e = \frac{w_o C a_w}{(1 - a_w)(1 + (C - 1)a_w)} \quad (1)$$

where w_o is the monolayer water content (g water/g solids), and C is the sorption energy constant.

$$w_e = \frac{w_o C K a_w}{(1 - K a_w)(1 + (C - 1) K a_w)} \quad (2)$$

where w_o is the monolayer water content (g water/g solids), C is the sorption energy constant related to monolayer water sorption, and K is the sorption energy constant related to multilayer water sorption.

$$w_e = 0.01 \left[\frac{-\log(1 - a_w)}{10^f} \right]^{1/n} \quad (3)$$

where f and n are the model parameters for the product.

$$w_e = \exp \left[a_w \cdot \ln(r) - \frac{1}{4.5 \cdot w_s} \right] \quad (4)$$

where r is a characteristic constant of the material and w_s is the moisture security content (g water/g solids) that provides the highest stability to the dehydrated product during storage.

2.5. Glass Transition Temperature

The glass transition temperature was analyzed by differential scanning calorimetry (DSC) using a DSC25 calorimeter (TA Instruments, New Castle, DE, USA). Approximately 10 mg of each pitaya powder sample was weighed into 40 µL aluminum DSC pans (Tzero pans, TA Instruments, New Castle, DE, USA), hermetically sealed, and analyzed using an empty pan as a reference. Nitrogen gas was used as both the refrigerant and purge gas at a flow rate of 40 mL/min. The heating program ranged from −80 to 80 °C at 10 °C/min, depending on the sample moisture content. Samples were analyzed in duplicate, and the data were fitted using the Gordon and Taylor model (Equation (5)). Nonlinear regression analysis was performed using the Solver tool in Excel 2019 (Microsoft, Redmond, WA, USA), minimizing the residual sum of squares.

$$T_g = \frac{(1 - x_w)T_g(as) + kx_w T_g(w)}{(1 - x_w) + kx_w} \quad (5)$$

where x_w is the mass fraction of water (g water/g product), T_g is the mid-point glass transition temperature (°C), $T_g(w)$ the glass transition temperature of amorphous water (−135 °C), $T_g(as)$ the glass transition temperature of the anhydrous sample, and k is the model constant.

2.6. Texture

Texture was evaluated using a TA-XT Plus Texture Analyzer (Stable Micro Systems, Ltd., Godalming, UK) with a 1/2'' cylindrical Delrin probe (P/0.5R). Powdered samples were placed in containers with a diameter of 38 mm and a height of 10 mm. A compression test was performed at 1 mm/s up to a height of 5 mm, with a trigger force of 0.049 N. After the measurement, the positive peak force (N) and area under the curve (N·mm) were recorded. Six repetitions were performed for each sample.

2.7. Color

A Chroma Meter CR-400 (Konica Minolta, Tokyo, Japan) with a D65 illuminant and 10° standard observer was used to obtain the CIE $L^*a^*b^*$ color coordinates in fresh pulp, peel, and each pitaya powder sample. The hue angle (h^*) and chroma (C^*) were calculated using Equations (6) and (7), respectively. Six repetitions were performed for each sample.

$$h^* = \tan^{-1} \frac{b^*}{a^*} \quad (6)$$

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (7)$$

The total color difference (ΔE^*) was calculated in each powdered sample, using the sample with the lowest moisture content (a_w 0.113) as a reference (Equation (8)).

$$\Delta E^* = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (8)$$

2.8. Extract Preparation

Extracts were prepared by homogenizing 200 mg of each pitaya powder sample with 1 mL of 96% ethanol in Eppendorf tubes using a vortex mixer. The mixture was centrifuged at 10,000 rpm for 3 min at 4 °C (Sorvall ST1 Plus, Thermo Scientific, Waltham, MA, USA). Then, 0.9 mL of the supernatant was transferred to a 10 mL volumetric flask, and 0.9 mL of fresh ethanol was added. The extraction procedure was repeated three times. Finally, the pooled supernatant was adjusted to 10 mL and stored at −18 °C until analysis. Two extracts were prepared for each sample.

2.9. Phenolic Content and Antioxidant Capacity

The phenolic content was determined using the Folin–Ciocalteu method. The extracts were mixed with distilled water (7:1), and 0.5 mL of Folin–Ciocalteu reagent was added. After 3 min, 1 mL of 20% Na_2CO_3 and 1.5 mL distilled water were added. The mixture was kept in the dark for 90 min, and the absorbance was measured at 765 nm using a Helios Zeta UV-Vis spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). A gallic acid calibration curve (0–150 ppm) was used, and the results were expressed as mg gallic acid equivalents (GAE) per g of dry solids. Each extract was analyzed in duplicate.

Antioxidant capacity was assayed using the Ferric Reducing Antioxidant Power (FRAP) assay and modified DPPH radical methods. The FRAP assay was performed as described by Benzie et al. [27] and Pulido et al. [28]. A mixture of 30 µL distilled water, 30 µL extract, and 900 µL FRAP reagent was prepared in 1.5 mL cuvettes. After incubation at 37 °C for 30 min, the absorbance was measured at 595 nm using a Helios Zeta UV-Vis spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). A Trolox ((±)-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid) calibration curve (0–500 µmol L^{−1}) was used, and the results were expressed as µmol Trolox equivalents per g dry solid (µmol g^{−1}). Each extract was analyzed in duplicate. The DPPH method was described by Raba et al. [29]; Chiari et al. [30]; Kalantzakis et al. [31]; and Shah et al. [32]. A total of 4 mL of DPPH solution (40 µg mL^{−1}) was mixed with 1 mL of extract and incubated in the dark for 30 min. The absorbance was measured at 517 nm using 4.5 mL plastic cuvettes. A Trolox calibration curve (0–200 µmol L^{−1}) was prepared, and the results were expressed in µmol Trolox equivalents per g dry solid (µmol g^{−1}). Each extract was analyzed in duplicate.

2.10. Statistical Analysis

All data were analyzed using the Statgraphics Centurion 19 software (StatPoint Technologies, Inc., Warrenton, VA, USA). One-way analysis of variance (ANOVA) was applied to determine significant differences between measurements. The least significant differences (LSD) were calculated using Fisher's test, and the significance was determined at $p < 0.05$.

3. Results

3.1. Fresh Pitaya

The batch of yellow pitaya fruit used in this study exhibited an SSC of $18.2 \pm 1.7^\circ$ Brix, which was higher than that previously reported for this variety [7]. In Colombian-grown yellow pitaya, SSC values typically range from 10.23 to 18.84° Brix [33], with variations largely influenced by fruit ripeness, agronomic conditions, and climatic conditions. SSC, primarily composed of simple sugars such as glucose, fructose, and sucrose, is a key indicator of sweetness and overall fruit quality. Based on these results, the fruits analyzed in this study were considered physiologically mature and exhibited a favorable sweetness level, an essential sensory attribute that influences consumer preference and marketability.

The yellow pitaya peel exhibited a high luminosity ($L^* = 63 \pm 3$) and yellow hue angle ($h^* = 85.5 \pm 1.5$). The chroma value ($C^* = 54.6 \pm 1.3$) indicated a vivid and saturated color, consistent with the bright yellow appearance typically observed in ripe fruits of this species. In contrast, the pulp showed lower luminosity ($L^* = 45 \pm 6$) and a slightly higher hue angle ($h^* = 91 \pm 3$), also within the yellow range. However, the chroma was markedly lower ($C^* = 3.5 \pm 1.2$), indicating very low color saturation. Despite its underlying hue, the pulp appeared visually white. This low chromatic intensity suggests a limited presence of pigments, which is characteristic of the translucent marble or pale pulp typical of this variety.

The freeze-drying yield was 23 ± 2 g of freeze-dried pulp per 100 g of fresh pulp, exceeding the 12.7% reported by Avila Quispe et al. [34] for the same variety. Nevertheless, the observed performance deviated from prior reports on tropical fruits, remaining below that of mango [35] but exceeding that of papaya [36]. These differences may be attributed to the different sugar and fiber contents of dragon fruit compared to those of mango and papaya.

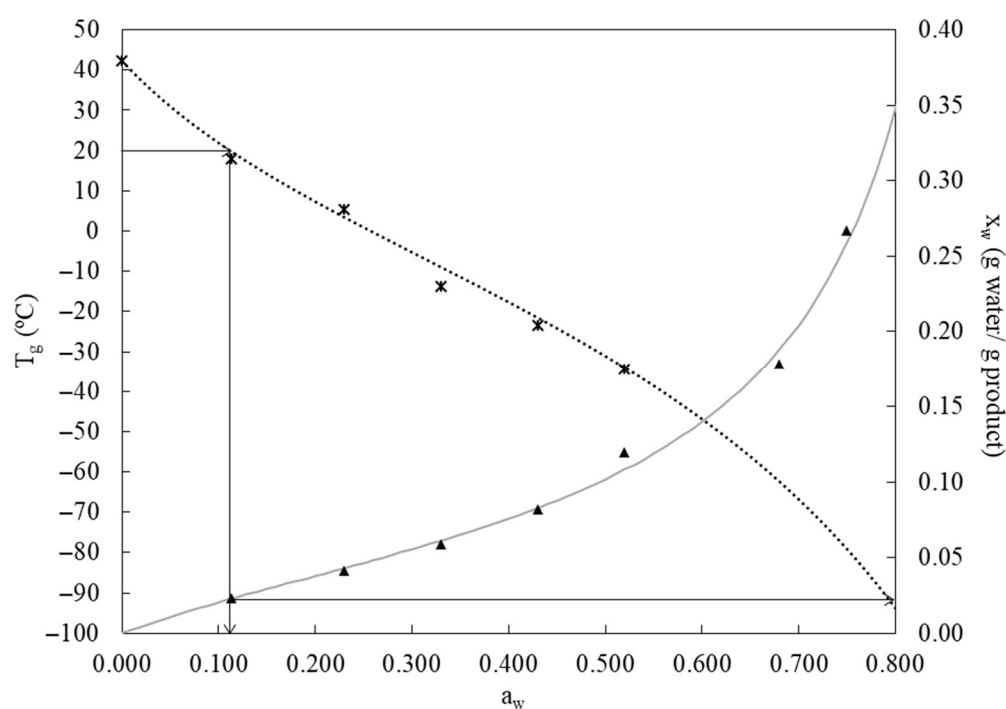
3.2. Sorption Isotherm and Glass Transition

Understanding the water sorption isotherm is useful for predicting the physical, chemical, and microbiological stability of pitaya pulp powder. Table 1 shows the parameters of the BET, GAB, Henderson, and Caurie sorption models fitted to the experimental data.

After comparing the four models, the GAB model, which is widely used to predict water sorption, was selected as the most suitable for predicting the water sorption behavior of the sample at 20°C across the studied a_w range (Figure 1). Both GAB and Caurie models exhibited the highest coefficients of determination (R^2), indicating good overall fits, while the GAB equation also showed the lowest mean relative error (%MRE = 3.18%), confirming its excellent performance. The K value above 1 may reflect complex water–matrix interactions in this sugar-rich, amorphous powder, which can deviate from an ideal behavior. The BET model was fitted only up to a_w values of 0.5, because the theoretical assumptions underlying this model are not valid at higher a_w levels. The obtained w_o value, considered a key stability parameter in powdered products, is consistent with previous studies on the same pitaya variety [37]. Nevertheless, controlling the glass transition remains essential to ensure product stability.

Table 1. Parameters of the water sorption models (BET, GAB, Henderson, and Caurie) fitted to experimental sorption data at 20 °C in yellow pitaya pulp powder.

Model	Parameters	Values
BET	w_o (g water/g solids)	0.069
	C	3.295
	R^2	0.965
GAB	w_o (g water/g solids)	0.057
	C	3.960
	K	1.123
	R^2	0.993
Henderson	n	0.900
	f	−1.534
	R^2	0.970
Caurie	w_s (g water g ^{−1} dry solids)	0.054
	r	55.118
	R^2	0.993

**Figure 1.** Relationship between glass transition temperature (T_g , *), water activity (a_w), and water content (x_w , ▲) in yellow pitaya pulp powder. Experimental data and curves predicted by the GAB and Gordon & Taylor models.

The thermograms obtained via DSC confirmed the decreasing trend of T_g with increasing moisture content, a phenomenon known as the water plasticizing effect. The T_g – x_w relationship was fitted using the Gordon & Taylor model (Equation (5)) for samples with a_w up to 0.52, as the model performed poorly for the two samples with the highest a_w . The value of the $T_g(as)$, analyzed in the anhydrous sample, was 42.3 °C, and the predicted model constant k was 6.23 ($R^2 = 0.958$). When compared to other powdered fruit products, the $T_g(as)$ value obtained for freeze-dried pitaya pulp powder was lower than those reported for hot-air-dried mango peel powders [38], grapefruit powder [39], and persimmon peel powder [40]. These differences suggest that freeze-dried pitaya may exhibit lower thermal stability under similar storage conditions.

The simultaneous modeling of the plasticizing effect of water (Gordon & Taylor model) and water sorption behavior (GAB model) enabled the determination of the critical values of water content (CWC) and water activity (CWA) associated with glass transition in freeze-dried pitaya pulp powder (Figure 1).

At 20 °C, the CWC and CWA values found in the freeze-dried pitaya pulp powder were 0.023 g water/g product and 0.110, respectively. The CWC value was lower than the w_o value predicted by the BET and GAB models, and also lower than the w_s value predicted by the Caurie model, indicating that it represents a stricter stability parameter. As illustrated in Figure 1, the sample with the lowest a_w value (0.113), which exhibited a $T_g = 18$ °C, was near the threshold for the transition from a glassy to a rubbery state, indicating that a physical state change may occur during storage. Under refrigerated conditions (4 °C), the CWC and CWA estimated by the models would be higher, at 0.042 g water/g sample and 0.226, respectively. However, the predicted CWA at 4 °C should be interpreted with caution, as it may not precisely reflect the actual behavior under refrigerated conditions, since the sorption isotherm used for its estimation was measured at 20 °C.

These critical points, particularly the critical moisture content, may also be of interest for pitaya powders obtained through other drying methods, as they provide a reference for predicting glass transition-related stability across different processing techniques. The glass transition temperature of amorphous food powders is mainly governed by water content and solute composition; therefore, the T_g - x_w relationship described by the Gordon & Taylor model is expected to be transferable across drying methods. However, since the relationship between moisture content and water activity depends on the sorption behavior of the matrix, which is affected by the drying process, critical water activity values may differ slightly between powders produced by different drying techniques.

3.3. Texture

Figure 2 shows the mechanical parameters obtained from the compression test of the yellow pitaya powder at different a_w levels. The positive peak force, which is related to hardness (N), indicates the resistance of the sample to compression. The area under the curve (N·mm) reflects the total energy required to compress the sample.

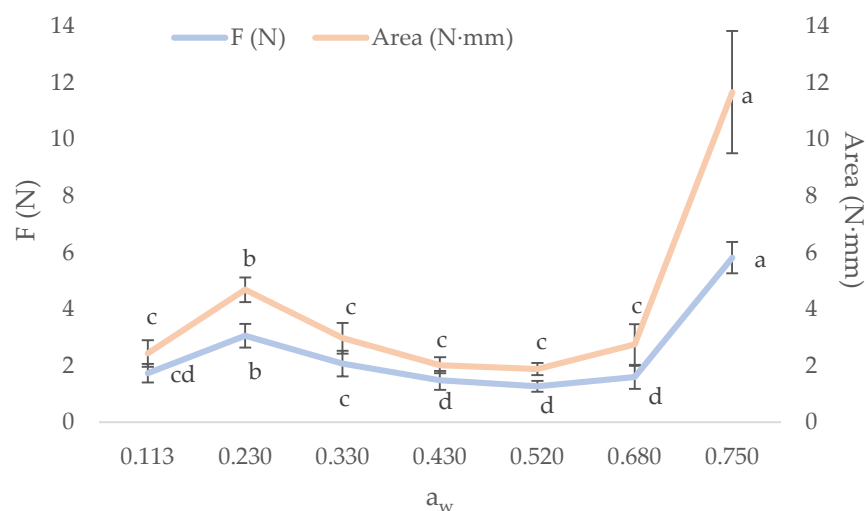


Figure 2. Maximum force (N) and area under the curve (N·mm) of yellow pitaya pulp powder at different water activity (a_w) levels. Values with different letters differ significantly ($p < 0.05$) according to ANOVA.

The mechanical behavior changed across the a_w levels. Samples with a_w values of 0.113 and 0.230 were found to be loose and hardened powders, respectively. This physical

difference was reflected in the mechanical parameters. The sample at a_w 0.230, which exhibited a compacted structure, showed significantly ($p < 0.05$) higher values of both maximum force and area, compared with the sample at a_w 0.113, indicating increased resistance to compression (Figure 2). This difference may be associated with the glassy-to-rubbery transition, a phenomenon often linked to hardening and subsequent structural collapse in powdered products [40]. From a_w 0.330 onward, extended signs of caking were observed, likely because of increased water adsorption. At these intermediate a_w levels, a significant ($p < 0.05$) decrease in both mechanical parameters was recorded, which could be attributed to the collapse of the powder structure. At a_w 0.650, the powder was transformed into a compact mass, leading to a significant ($p < 0.05$) increase in the mechanical parameters of the sample at a_w 0.750. This change could be related to mucilage wetting, as the rehydration of mucilage may increase its viscosity, raising both the maximum force and area values of the sample [41].

Based on the observed textural changes, the powder remains free-flowing and suitable for industrial handling, packaging, and mixing operations at low water activity values, below CWA. In contrast, at higher a_w values, corresponding to the rubbery state, caking and loss of structural integrity would compromise the powder's processability. These practical findings for yellow pitaya pulp powder provide useful guidance for defining appropriate handling and storage conditions.

3.4. Color

The color parameters of yellow pitaya pulp powder were significantly affected by a_w . Table 2 shows the mean values of L^* (lightness), h^* (hue), C^* (chroma), and total color difference ΔE^* as a function of a_w .

Table 2. Color attributes (luminosity L^* , hue h^* , and chrome C^*) and global color differences (ΔE^*) of yellow pitaya pulp powder at different water activity (a_w) levels.

a_w	L^*	h^*	C^*	ΔE^*
0.113	75.9 ± 1.1 ^a	89.3 ± 0.2 ^a	11.7 ± 0.5 ^b	-
0.230	73.4 ± 1.2 ^b	88.8 ± 0.2 ^{ab}	11.8 ± 0.2 ^b	2.54
0.330	70.9 ± 0.5 ^c	88.2 ± 0.8 ^b	12.1 ± 0.8 ^{ab}	5.06
0.430	69.3 ± 0.8 ^c	88.1 ± 0.4 ^b	12.7 ± 0.6 ^a	6.70
0.520	62.9 ± 1.2 ^d	87.1 ± 0.2 ^c	12.9 ± 0.6 ^a	13.16
0.680	48.9 ± 0.7 ^f	81.0 ± 0.7 ^d	9.9 ± 0.2 ^c	27.18
0.750	55.6 ± 1.2 ^e	87.2 ± 0.1 ^c	12.5 ± 0.3 ^{ab}	20.40

Values in a column with different superscripts differ significantly ($p < 0.05$) according to ANOVA.

High luminosity, related to the L^* coordinate values, was obtained in samples under low humidity conditions; however, a significant ($p < 0.05$) decrease in L^* was observed from a_w 0.113. This decrease occurred earlier than that reported in studies on other fruits, such as grapefruit [21]. The darkening of the powder progressed during water adsorption, reaching its maximum at a_w 0.680. The h^* coordinate remained within the first quadrant for all samples, indicating a predominantly yellow-orange hue. Samples with lower water content showed a more yellow hue angle, whereas as a_w increased, a gradual shift toward orange was observed, which was more marked at a_w 0.680. Similar values were obtained for C^* across most of the study range; however, a significant ($p < 0.05$) decrease was recorded at a_w 0.680, indicating a loss of chromatic saturation.

The total color difference (ΔE^*), calculated relative to the reference (a_w 0.113), increased from a_w 0.230 to 0.680. In samples with an a_w below 0.430, the color change was less noticeable. A critical value was observed at an a_w of 0.680, where the most perceptible color change occurred. This phenomenon could be mainly attributed to enzymatic browning

reactions, which may generate brown compounds and affect the product's appearance. Water activity may influence the activity of polyphenol oxidase (PPO), the main enzyme responsible for enzymatic browning in fruits. Maintaining a low a_w is essential to limit enzymatic reactions; as water activity increases, molecular mobility is enhanced, facilitating enzyme–substrate interaction and consequently favoring enzymatic browning [42]. Moreover, non-enzymatic browning may also occur. Although Maillard browning is minimal during the storage of freeze-dried fruits, it can intensify if a_w increases [43]. In addition, color changes may also be associated with pigment degradation and/or structural changes such as matrix collapse. The decrease in ΔE^* values in the sample with a_w 0.750 could be related to the dilution of coloring compounds due to the higher moisture content, as reported in previous studies [21]. Comparable trends have been reported for similar fruit-based products, showing that color changes become more pronounced with increasing a_w , while remaining minimal in the glassy state due to the limited molecular mobility and slow diffusion of reactants [17,40,44].

3.5. Phenolic Content and Antioxidant Capacity

Figure 3 illustrates the phenolic content and antioxidant capacity of yellow pitaya pulp powder at different water activity levels.

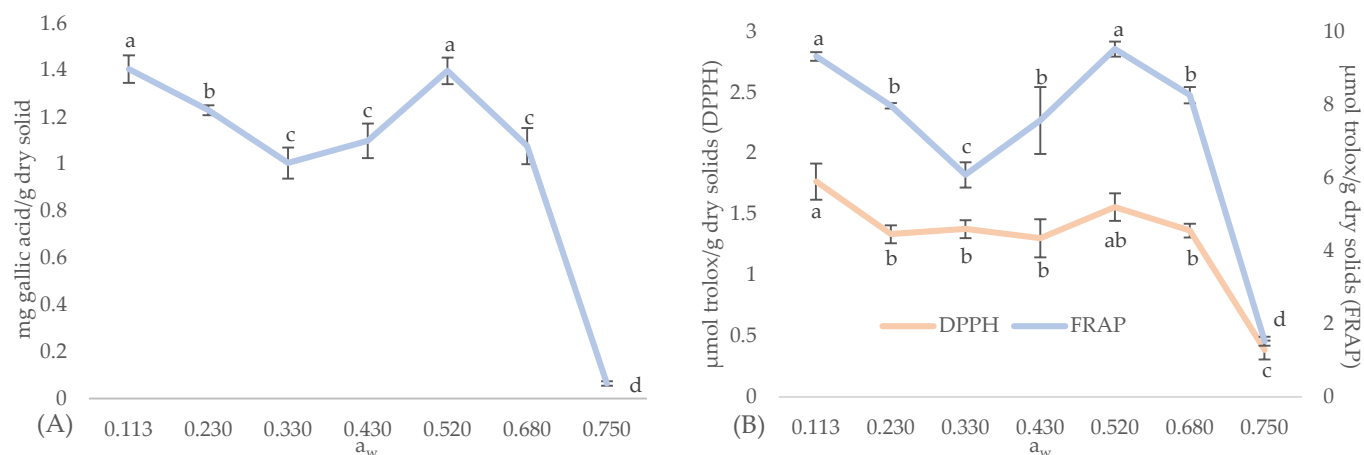


Figure 3. Phenolic content (mg gallic acid/g dry solids) (A) and antioxidant capacity (μmol Trolox equivalents/g dry solids) by DPPH and FRAP assays (B) of yellow pitaya pulp powder at different water activity (a_w) levels. Values with different letters differ significantly ($p < 0.05$) according to ANOVA.

The influence of water activity on the total phenolic content of pitaya powders showed a nonlinear behavior, with significant ($p < 0.05$) differences among samples (Figure 3A). Phenolic values decreased from a_w 0.113 to 0.230 and reached lower levels at a_w 0.330 and 0.430. The recovery of phenolic values observed at a_w 0.520, without significant differences ($p > 0.05$) to those at a_w 0.113, suggests that the decrease observed at intermediate a_w may not reflect actual loss or degradation of phenolic compounds, but rather a reduction in their extractability related to structural changes in the powder matrix. At a_w 0.113, the freeze-dried, free-flowing powder, in a highly porous form, may allow efficient solvent penetration and, consequently, high extractability of phenolic compounds. As water activity increased to intermediate levels (a_w 0.230–0.430), the observed decrease in detected phenolic content could be related to structural collapse of the powder matrix, induced by the plasticizing effect of water and the transition from a glassy to a rubbery state. At these intermediate moisture levels, hydrogen bonding between water molecules and matrix components may have promoted particle agglomeration and compaction limiting solvent accessibility and reducing phenolic compound extractability, rather than reflecting actual molecular

degradation. This trend was reversed at a_w 0.520, where a significant ($p < 0.05$) increase in phenolic extractability was observed. Under these conditions, increased water availability and molecular mobility within the matrix may have led to the relaxation of the collapsed structure, facilitating solvent diffusion and the release of soluble phenolic compounds, thereby improving their extractability. Similar moisture-enhanced extractability has been described for other fruit powders [40]. From a_w 0.520, a significant ($p < 0.05$) decrease in phenolic content was observed, reaching a 22.97% loss at a_w 0.680 and a 95.38% loss at a_w 0.750, likely attributable to increased PPO activity at high a_w levels.

The same pattern was observed in the antioxidant capacity measured by FRAP (Figure 3B). Changes in FRAP values reflected variations in phenolic extractability at low and intermediate water activity levels and their subsequent degradation at higher a_w , supporting the strong contribution of phenolic compounds with reducing capacity to this assay [27]. In contrast, DPPH values showed smaller variations at intermediate a_w levels, indicating that radical-scavenging activity was less sensitive to changes in total phenolic content.

4. Conclusions

Yellow pitaya pulp powder is a promising functional ingredient with strong potential for international commercialization and year-round food applications; its physical properties and bioactive compound content are strongly influenced by water activity. The critical water activity and the water content associated with the glassy-to-rubbery transition were identified, providing key thresholds for maintaining powder quality. The findings indicate that maintaining the product in a glassy state (at 20 °C, below a_w 0.110, and 0.023 g water/g product) is essential to obtain a light and free-flowing powder with the highest levels of phenolic compounds and antioxidant capacity. Exceeding these thresholds compromises the physical quality and functional stability of the powder.

Caking and structural collapse, both related to glass transition phenomena, are identified as the primary deterioration mechanisms and occur when the powder exceeds the critical water activity and water content, rendering it unsuitable for commercialization. However, the most pronounced alterations in color and bioactive compound content were observed at higher water activity levels (a_w 0.680 and 0.750, respectively), indicating that these quality attributes are affected beyond the critical thresholds for physical stability.

These results underscore the importance of strict moisture control in preserving both the physical quality and functional potential of the final product, providing guidance for handling, packaging, and storage under real-world conditions. Refrigerated storage may be beneficial for enhancing powder stability and handling properties; however, its economic feasibility should be evaluated.

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