Shelf life assessment and antioxidant degradation kinetics of a pineapple–ginger agglomerated powder: Insights into storage stability

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Abstract

Agglomerated food powders may undergo degradation reactions during storage, resulting in the loss of nutritional quality and functional properties. This research aimed to evaluate the stability of an agglomerated pineapple–ginger powder mixture (APGPM), obtained through the fluidised bed process. The mixture was stored at different temperatures (T) (15, 25 and 35°C) over different periods (t) (0, 30, 60, 90, 120, 150 and 180 days). The samples were stored in a controlled environment at 65% relative humidity, packed in PET foil-laminated film bags. Moisture (X_w), water activity (a_w), Carr's index (CI), Hausner's ratio (HR), solubility, hygroscopicity, wettability, color (L, a*, b*), antioxidant capacities (ABTS and DPPH), total phenols (TP), total flavonoids (TF), vitamin C (Vit. C) and β-carotene (β-car) were monitored. Overall, ANOVA showed statistically significant differences ($P < 0.05$) in most of the dependent variables concerning t, T, and their interaction (t-T). At the end of storage, moisture and a_w showed values conferring stability to APGPM. Fluidity was classified as good and very good, and cohesiveness as low. Colour was stable, with no significant differences observed. The lowest retention values were found for ABTS (33.6–50.7%), DPPH (34.0–61.2%), TP (64.8–70.8%), TF (30.1–0.53%), Vit. C (12.8–40.0%) and β-car (0–39%).

Keywords: antioxidants; bioactive compounds; degradation kinetics; shelf life

Introduction

Pineapple (*Ananas comosus*) is a tropical fruit with attractive sensory properties and nutritional characteristics. It is considered a rich source of vitamins B and C, as well as minerals such as calcium, phosphorus and iron, among other compounds (Ancos *et al*., 2016). Ginger (*Zingiber officinale*) is known for its spicy taste and is an excellent source of nutrients, including minerals (Mn, Fe, Mg, Zn, K, P and Ca) and vitamins (C, B3, B6, B1, B2, B9 and E). It also contains bioactive phenols such as gingerols, paradols, shogaols and zingerones, among others (Mbaveng and Kuete, 2017).

In general, food powders obtained through spray drying and fluidised bed agglomeration are typically found in a non-thermodynamic amorphous state, where their behaviour depends on the glass transition temperature (Tg). This condition strongly influences the required storage conditions for the product. If the storage temperature is lower than Tg, the product may retain a glassy state. However, at higher temperatures, it can transition into a rubbery amorphous state, where the molecular mobility of water significantly increases, leading to important changes in its physical, physicochemical, chemical, sensory, microbiological and nutritional properties (Rahman, 2019). Additionally, water content (X_w) in powdered foods is a critical parameter during storage due to its plasticising effect on amorphous systems (Carter and Schmidt, 2012). An increase in X_w levels due to adsorption phenomena further enhances molecular mobility by reducing local viscosity (Staniszewska *et al*., 2022). Thus, the physical, chemical, and microbiological stability of powdered products is strongly influenced by the relationship between equilibrium X_w and a_w at a given temperature.

Studies on food stability over time enable the determination of shelf-life behaviour under different conditions. In addition, modelling the kinetics of quality parameter changes allows for the prediction of product behaviour during storage (Chang *et al*., 2018a), making it an important tool for decision-making regarding the conditions required to ensure that the product meets established quality standards for consumers (Udomkun *et al*., 2016).

In general, powdered foods are microbiologically stable due to low a_w and X_w values. However, these products can be affected during storage by intrinsic factors, such as the type and concentration of additives, chemical composition, the ratio of functional groups present (proteins, lipids, and carbohydrates), polarity and particle morphology, among others; as well as extrinsic factors, including environmental conditions (temperature, time, relative humidity, light, O_2), packaging, and others (Muzaffar and Kumar, 2016; Rannou *et al*., 2015). The combined effect of these independent variables leads to deterioration in nutritional value (Zorić *et al*., 2017), as well as in quality characteristics such as fluidity, agglomeration, and colour (Lucas-Aguirre *et al*., 2019; Pereira *et al*., 2020).

With regard to the behaviour of active compounds in food, the development of kinetic models has enabled the simulation of quality attributes, or dependent variables of food, at a given t, thereby addressing the needs of both science and the food industry while also determining shelf life. In this context, kinetic models provide a structural framework that enables the quantitative description of the changes occurring within a food system (Chang *et al*., 2018a). The measurement or availability of appropriate kinetic constants for these changes allows for the estimation of the magnitude of change of a given compound during food processing or storage (Heldman, 2011).

The evolution or changes in the properties of fruit powders during storage have been reported in several studies: myrtle (Cheng *et al*., 2017), jambolan (de Carvalho *et al*., 2020), blueberry (Staniszewska *et al*., 2022), soursop (Chang *et al*., 2019), mango peel (Ferrari *et al*., 2021), blackberry (Ferrari *et al*., 2013), papaya (Udomkun *et al*., 2016; Wong and Lim, 2016), pink guava (Shishir *et al*., 2017) and coconut (Lucas-Aguirre *et al*., 2019). Within this context, the present research aimed at evaluating the stability of an agglomerated pineapple–ginger powder mixture (APGPM) obtained via fluidised bed processing and stored at different temperatures (15, 25 and 35°C) and times (0, 30, 60, 90, 120, 150 and 180 days).

Materials and Methods

An APGPM was obtained using a Lemar (China) FL5 fluidised bed dryer under the following operating conditions: air inlet temperature (TEA) of 70°C, atomisation pressure of 1 bar, fluidising air fan frequency of 30–34 Hz, peristaltic pump frequency for atomisation water feed of 3–4 Hz (10–15 mL/min), process time of 1 h and 45 min, material load of 3000 g, fluidising agent of 0.5% w/w silica dioxide, binding solution (SL) composed of ginger extract (50%) + water (50%) + ascorbic acid (AA) (3.33 g/L total solution), and an SL consumption of 2000 mL. The powder mix used in the formulation was obtained from the pineapple fruit MD2 variety, purchased at a supermarket in the city of Medellín.

Determination of physical and physicochemical variables

X_w was determined according to the official AOAC 930.15/90 method. Water activity (a_w) was measured using a dew point hygrometer at 25°C (Aqualab 3TE series, Decagon). Bulk density (ρb) was determined following the methodology described by Pereira *et al*. (2020), with modifications, by weighing 2 g of sample and recording the volume in a 10 mL test tube. Compacted density (ρc) was determined by placing 2 g of sample in a Falcon tube and centrifuged at 5000 rpm for 10 min, after which the recorded volume was measured (Gagneten *et al*., 2019). Fluidity was determined using the Carr Index (CI) (Carr, 1965) (Equation 1), while cohesiveness was assessed using the Hausner Ratio (HR) (Hausner, 1967) (Equation 2).

$$CI = \frac{\rho_c - \rho_b}{\rho_c} \times 100 \quad (1)$$

$$HR = \frac{\rho_c}{\rho_b} \quad (2)$$

Solubility was determined according to the methodology of Chauhan and Patil (2013), modified by Marulanda *et al*. (2018). Wettability was assessed as the time required for 1 g of sample to disappear from the surface of a 100 mL volume of water at 20°C (Marulanda *et al*., 2018). Hygroscopicity was determined using the sorption isotherm methodology described by Daza *et al*. (2016), employing the gravimetric method at a constant and controlled relative humidity of 68%, maintained with a supersaturated potassium iodide solution at 25°C. Particle size was measured using a Mastersizer 3000 analyser (Malvern Instruments) and reported as the volume-equivalent diameter (D[4,3]). Colour was determined using CIE-Lab coordinates, measured with an X-RITE sphere spectrophotometer (model SP64) under illuminant D65, with a 10° observer and the specular component included.

The total phenolic (TP) content was determined from a methanolic extract obtained following the methodology described by Gallón *et al*. (2020) and quantified using the Folin–Ciocalteu reagent. Gallic acid calibration curves, ranging from 0 to 300 µg/mL (R^2 = 0.989), were used, and TP results were expressed as mg gallic acid equivalent (GAE)/100 g db.

Antioxidant capacity was assessed through DPPH and ABTS free radical scavenging activity in the methanolic extract (Gallón *et al*., 2020). Trolox equivalent (TE) calibration curves were used for ABTS (50–250 µM, R^2 = 0.998) and DPPH (0.02–0.12 mg/mL, R^2 = 0.997), with results expressed as mg TE/100 g db.

The total flavonoid (TF) content was determined using the colorimetric method described by Sharma *et al*. (2016), with modifications. A 500 µL aliquot of extract was mixed with 150 µL of $NaNO_2$ (5% w/v), 150 µL of $AlCl_3$ (10% w/v), and 700 µL of NaOH (1 M). A standard calibration curve was prepared using quercetin in the range of 10–30 µg/mL, and absorbance was measured at 510 nm. Results were expressed as mg quercetin equivalent (QE)/100 g db.

Vitamin C (Vit. C) content was determined by placing 1.5 g of sample in a graduated test tube and adding a 0.02 M KH_2PO_4 buffer solution, adjusted to pH 3.0 with 85% orthophosphoric acid, to a final volume of 20 mL. The mixture was vortexed for 2 min and centrifuged at 5000 rpm for 15 min at 4°C. The supernatant was then filtered through a 0.45 µm cellulose acetate membrane filter and collected in an amber vial. Quantification was performed using HPLC (Shimadzu Prominence 20A) with a Luna® 5 µm C18(2) 100 Å column (250 × 4.6 mm). The mobile phase consisted of 0.02 M KH_2PO_4 (pH 3.06), with a flow rate of 1 mL/min and a pressure of 1172 psi. Retention times ranged from 4.317 to 4.456 min, with an injection volume of 5 µL and detection at 244 nm. An analytical standard solution of L-ascorbic acid (Sigma-Aldrich 47863, Lot LRAC1812) was used, and results were expressed as mg AAE/100 g db (Lucas *et al*., 2018).

β-carotene (β-Car) content was determined following the methodology of Etzbach *et al*. (2020), with minor modifications. A 1 g sample was dissolved in 10 mL of distilled water before carotenoid extraction, mixed with 10 mL of a hexane-acetone solution (60:40) and centrifuged at 8965 g for 5 min at 4°C. The supernatant was filtered through a 0.45 µm PVDF syringe filter. Identification and quantification were performed using HPLC on a Prominence UFLC 20A system (Shimadzu, Kyoto, Japan), coupled to a Prominence SPD-M20A diode array detector, with a Luna® C18(2) 100 Å column (250 × 4.6 mm ID, 5.0 µm Dp). The mobile phase consisted of acetonitrile:-methanol:acetone (60:30:10), with a flow rate of 1.2 mL/min, a column temperature of 45°C, an injection volume of 20 µL and detection at 450 nm. The separation was carried out in isocratic mode with an analysis time of 45 min. The β-Car concentration was determined using the external standard method, with a β-carotene standard curve (Sigma-Aldrich C4582) in the range of 1–30 µg/mL (R^2 = 0.999), and a retention time of 19.52 min. Results were expressed as µg/100 g db.

Experimental design and data analysis

A completely randomised factorial design was employed, considering the independent variables: storage time (0, 30, 60, 90, 120, and 180 days) and temperature (15, 25, and 35°C), and the dependent variables: X_w, a_w, solubility, hygroscopicity, wettability, CI, RH, colour parameters (L, a*, b*), antioxidant activity (ABTS and DPPH), TP, TF, β-Car and Vit. C. The samples were packed in multilayer bags (Alico S.A) with a laminated film thickness of 12 µm (PET), 8 µm aluminium foil, and a polyethylene sealing layer of 100 µm, resulting in a total weight of 136.5 g/m^2. The bags had a water vapour permeability of <1 cm^3/(m^2·24 h·atm) and an oxygen permeability of <1 cm^3/(m^2·24 h·atm). They were stored in climatic chambers with controlled relative humidity (65%) and packed under ambient conditions. Each package contained 100 g of APGPM. The results, obtained from three replicates, are presented as mean ± standard deviation.

A two-way analysis of variance (ANOVA) was employed to evaluate the effect of temperature and storage time on the response variable. In the model, time and temperature were treated as continuous variables, with different experimental levels. Each specific combination of time

and temperature corresponded to an independent sample, as no repeated measurements were taken on the same sample, even though all samples originated from the same initial batch.

When significant differences were found, Tukey's test was applied as a post hoc analysis. Statistical analysis was performed using Statgraphics Centurion XVII.II software.

Degradation kinetics of the active compounds

Variations in active compound content and antioxidant activity were analysed using zero-, first-, and second-order kinetic models, as described in Equations 3, 4, and 5, respectively. The reaction order was determined by regression analysis in Microsoft Excel® to identify the best-fitting model (R^2). Additionally, the effect of temperature was evaluated using the Arrhenius equation (Equation 6) to determine the activation energy (Ea) (Heldman, 2011).

$$C_t = C_0 - kt \quad (3)$$

$$C_t = C_0 \exp(-kt) \quad (4)$$

$$\frac{1}{C_T} = \frac{1}{C_0} + kt \quad (5)$$

$$Ln(k) = \frac{E_a}{RT} + Ln(k_0) \quad (6)$$

Results and Discussion

The properties of pineapple + ginger agglomerate powder (APGPM) were X_w = 4.8 ± 0.2%, a_w = 0.143 ± 0.003, bulk density (ρb) = 0.38 ± 0.01 g/mL, solubility = 95.5 ± 0.3%, wettability = 8.1 ± 0.1 s, hygroscopicity = 17.3 ± 0.2%, CI = 4.1 ± 0.1%, HR = 1.05 ± 0.01, antioxidant activity: DPPH (324.1 ± 6.8 mg Trolox equivalent (TE)/100 g dry basis (db)), ABTS (287.4 ± 5.1 mg TE/100 g db), TP = 373.8 ± 4.1 mg gallic acid equivalent (GAE)/100 g db, TF = 232.7 ± 2.5 mg quercetin equivalent (QE)/100 g db, Vit.C = 253.4 ± 4.0 mg ascorbic acid equivalent (AAE)/100 g db, β-Car = 753.2 ± 4.7 µg/100 g db, and particle size ($D_{[4,3]}$) = 111.6 ± 2.6 µm.

Table 1 presents the significant differences ($P < 0.05$) of the dependent variables according to ANOVA.

Table 1. ANOVA (P value) of the APGPM storage study.

Variable	Time	Temperature	Interaction Time–Temperature
X_w	0.000	0.003	0.124
a_w	0.000	0.565	0.274
Solubility	0.000	0.295	0.346
Wettability	0.000	0.239	0.014
Hygroscopicity	0.000	0.185	0.048
CI	0.000	0.002	0.026
HR	0.039	0.879	0.790
L	0.000	0.005	0.016
a*	0.000	0.000	0.000
b*	0.000	0.244	0.270
TP	0.000	0.042	0.182
TF	0.000	0.027	0.001
ABTS	0.000	0.003	0.712
DPPH	0.000	0.000	0.006
β-Car	0.000	0.000	0.003
Vit-C	0.000	0.000	0.000

Moisture and water activity

Figure 1 presents the mean values and standard deviations for X_w and a_w of APGPM during storage. X_w and a_w are crucial parameters influencing the stability of powdered food products. In dehydration processes, the rate of mass transfer is pivotal in determining the final state of the material. When these rates are high, the product tends to acquire an amorphous state, which can range from vitreous to gummy.

The latter state is prone to accelerating deterioration reactions due to increased water mobility within the food matrix, thereby reducing its quality over storage time (Karaca *et al.*, 2015). Nevertheless, the inherent characteristics of the product and the interaction between temperature, X_w, and a_w play a crucial role in determining its shelf life.

These results position the product within the established range for dehydrated powdered products, thereby ensuring reduced rates of physicochemical and biochemical deterioration reactions, as well as microbial growth (Shishir *et al.*, 2017).

X_w exhibited a progressive increase during storage, with the highest values observed at 35°C. Consequently, ANOVA revealed significant statistical differences concerning the storage time and temperature of the independent variables. In general, the X_w and a_w values obtained for APGPM were consistently higher than those reported for pineapple powder or mixtures obtained via spray drying (4.815 ± 0.176% and 0.143 ± 0.003, respectively). This difference arises because agglomerated powders absorb moisture from SL, which is not entirely removed during processing, resulting in a final X_w higher than the

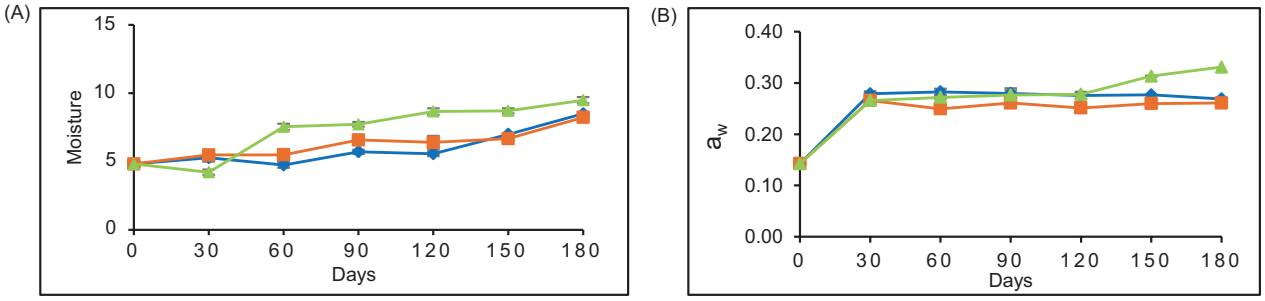

Figure 1. X_w (A) and a_w (B) of the APGPM during storage (blue: 15°C, orange: 25°C, and green: 35°C).

initial value. The X_w behaviour observed aligned with previous studies on other spray-dried products, such as sweet guava (Shishir *et al.*, 2017), black cumin powder (Varastegani *et al.*, 2019), soursop (Chang *et al.*, 2018a) and coconut (Lucas-Aguirre *et al.*, 2019), as well as products obtained through alternative technologies, including orange fibre (Fernández-López *et al.*, 2009) and pineapple (Shiby *et al.*, 2017).

Conversely, a_w exhibited a trend similar to X_w; however, a sharp increase was noted mainly at 30 days for all temperatures. Subsequently, an asymptotic trend was observed at 15 and 25°C, whereas at 35°C, a_w continued to rise progressively, reaching its highest values. This behaviour is primarily attributed to the driving force for mass transfer generated by the water chemical potential differential between APGPM (a_w = 0.142 at day 0) and the storage chamber (a_w = 0.650), which induces water adsorption at the active sites on the surface of APGPM until equilibrium is achieved (from Day 30 onwards) (Wilson *et al.*, 2014).

This water transfer from the storage chamber to APGPM is regulated by the water vapour permeability of the packaging used (<1 $cm^3/[m^2 * 24\ h\ atm]$).

Reconstitution properties

Figure 2 presents the mean values and standard deviations for solubility, hygroscopicity and wettability of the APGPM during storage.

In general, during storage, APGPM exhibited acceptable solubility (83.8 ± 0.6% to 92.3 ± 1.3%), with a gradual decrease over 180 days, becoming more pronounced

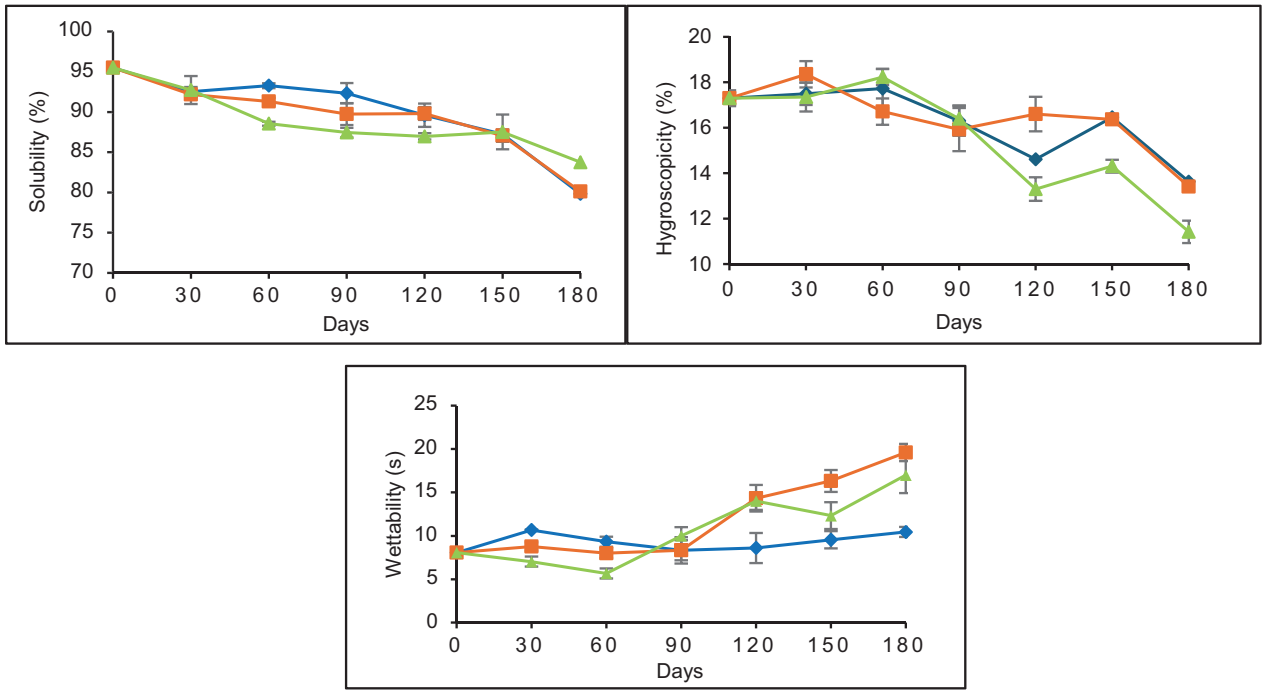

Figure 2. Solubility, hygroscopicity, and wettability of the APGPM during storage.

between days 150 and 180. The ANOVA showed significant differences ($P < 0.05$) only for storage time but not temperature. Similar behaviours have been reported during the storage of *Nigella sativa* powder at 4°C for 12 months (92.6% to 91.9%) (Varastegani *et al.*, 2019). Other studies have observed reductions in solubility during storage, including: a 35% decrease in soursop powder at 35°C for 90 days (Chang *et al.*, 2018b); a decline from 97.1 to 85.1% in papaya powder stored at 38 ± 2°C, 90% relative humidity, for 7 weeks (Wong and Lim, 2016); and a decrease in mango peel powder over 6 months under ambient conditions (Wilson *et al.*, 2014).

Wettability values ranged between 5.7 ± 0.5 and 19.6 ± 0.9 s, with ANOVA showing significant statistical differences ($P < 0.05$) concerning storage time and the storage time–temperature interaction. Wettability increased over time, revealing two homogeneous groups: one comprising the 25 and 35°C samples, which exhibited higher values, and another consisting of the 15°C samples, which showed lower values. In general, the results obtained could be considered characteristic of powders with good instantaneity (Cuq *et al.*, 2013), similar to those observed in other agglomerated food powders: *N. sativa* (6.56 ± 0.73 s) (Mohammed *et al.*, 2019), hydrolysed collagen (12 s) (Andreola *et al.*, 2015), spinach with maltodextrin (6 s) and gum arabic (5 s), among others.

The behaviour of solubility and wettability is influenced by the increase in X_w over time. This phenomenon enhances the wetting of the solid bridges within the agglomerated particles, leading to a restructuring of the microstructure. As a result, pores collapse, causing caking that restricts water diffusion into the interior, thereby reducing solubility and increasing wetting time (Haider and Palzer, 2016).

Hygroscopicity is a key property of low-moisture foods, which represents the ability of a product to absorb water molecules from the surrounding environment. Greater adsorption capacity occurs when the food structure is drier and when there is a higher affinity between the surface's active sites and the surrounding water molecules (Janiszewska-Turak *et al.*, 2017). This response corresponds to the X_w equilibrium reached by APGPM under the conditions to which it is subjected (temperature: 25°C, relative humidity: 68%). ANOVA revealed significant statistical differences in hygroscopicity ($P < 0.05$) concerning storage time and the storage time–temperature interaction. Hygroscopicity exhibited a progressive decline over time, with lower values observed at 35°C after 180 days (17.3 ± 0.5% to 11.4 ± 0.5%). This behaviour was consistent with the X_w of APGPM over time, where lower hygroscopicity values reflect higher X_w levels. In this context, hygroscopicity is a variable influenced by the water vapour permeability of the packaging used. Therefore, ensuring minimal permeability to H_2O and O_2 vapour contributes to improved APGPM stability during storage.

Flowability and cohesiveness

Figure 3 presents the mean values and standard deviations for flowability and cohesiveness, measured as CI and HR, of the APGPM during storage.

The CI represents the percentage of powder compressibility, with flowability classified as very good (<15%), good (15–20%), medium (20–35%), poor (35–45%), and very poor (>45%) (Carr, 1965; Thakur and Nanda, 2022). CI was highly sensitive to storage conditions, with the ANOVA revealing significant statistical differences ($P < 0.05$) to time, temperature and their interaction. In this context, and according to the fluctuations observed in CI, APGPM exhibited good flowability (4.1 ± 0.9% to 19.7 ± 0.9%) during storage.

On the other hand, HR represents a measure of APGPM cohesiveness, classified according to Hausner (1967), as low (<1.2), intermediate (1.2–1.4) and high (>1.4). The mean HR values ranged from 1.05 ± 0.01 to 1.24 ± 0.03,

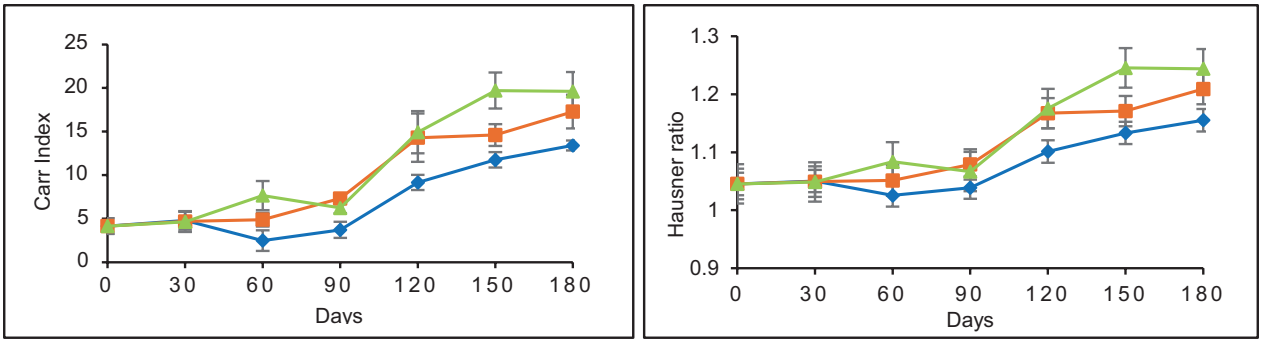

Figure 3. Flowability and cohesiveness, measured as CI and HR, of the APGPM during storage.

indicating classification of cohesiveness as low to intermediate. The ANOVA identified significant statistical differences ($P < 0.05$) for HR only with time, showing an increasing trend with temperature. However, the most pronounced changes occurred after 90 days, with higher values observed at higher temperatures.

APGPM demonstrated good flow properties during storage, primarily due to the presence of SiO_2, which possesses fluidising and anticompacting properties. SiO_2 reduces the likelihood of particles sticking together by minimising cohesive forces, thereby preventing caking. In general, the flow characteristics of powdered foods are affected by storage conditions (time, temperature and packaging materials), density, particle size, capillary activity and the migration of cohesive chemical components to the particle surface (Cruz-Tirado *et al.*, 2021).

For both CI and HR, the most significant increase was observed at 35°C between days 120 and 180. This behaviour aligns with the increase in X_w over time and temperature, which enhances material plasticity, increases molecular mobility and promotes the formation of liquid bridges between particles (Ferrari *et al.*, 2013; Zungur *et al.*, 2017). The flow characteristics of APGPM were consistent with those reported for olive powder stored at 4, 25 and 60°C for 180 days (Zungur *et al.*, 2017), soy milk powder stored at 10, 25, 35 and 55°C for 150 days (Cruz-Tirado *et al.*, 2021), and bee pollen-enriched milk powder stored at 5, 25 and 40°C for 365 days (Thakur and Nanda, 2022).

Colour

Figure 4 presents the mean values and standard deviations for Colour (L, a*, b*), of the APGPM during storage.

Colour is a fundamental sensory attribute of food, playing a crucial role in consumer perception. It influences purchasing and consumption decisions while also serving as an indicator of product quality and condition, which is perceived visually (Mathias-Rettig and Ah-Hen, 2014). Food colour results from biochemical reactions occurring during processing and storage (de Carvalho *et al.*, 2020). Its stability during storage depends on multiple factors, including storage time, temperature, relative humidity, light exposure, moisture content and product composition (Chang *et al.*, 2018a).

ANOVA revealed significant statistical differences ($P < 0.05$) in luminance (L) and chromaticity (a) in relation to storage time, temperature and their interaction. Additionally, chromaticity (b) exhibited significant differences ($P < 0.05$) only with storage time. Luminance (L) is closely associated with browning in food matrices, ranging from black (L = 0) to white (L = 100). For APGPM, L was the most relevant colour parameter, with values ranging from 51.3 ± 0.7 to 71.2 ± 0.6 at 15°C, 51.3 ± 0.7 to 64.9 ± 0.6 at 25°C and 51.3 ± 0.7 to 64.8 ± 0.1 at 35°C.

The evolution of L over storage time showed a significant increase during the first 30 days at all temperatures: by 38% at 15°C and by 26% at 35°C. Subsequently,

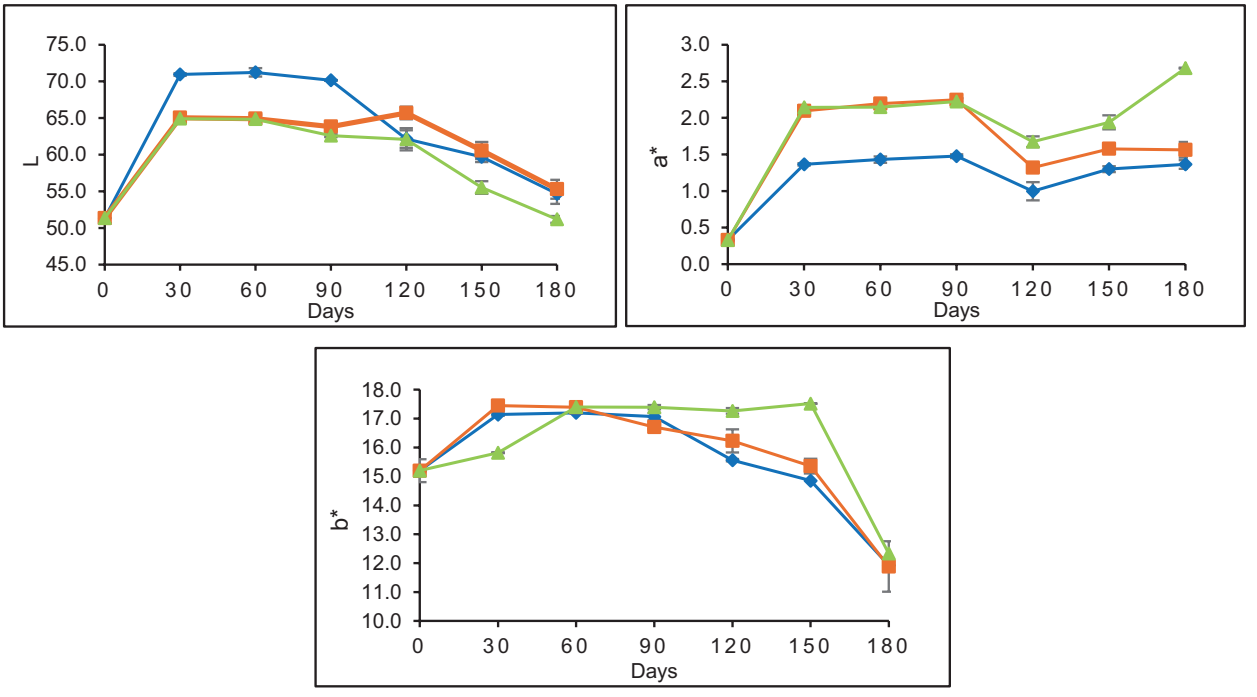

Figure 4. Colour (L, a*, b*) of the APGPM during storage.

values remained stable for approximately 90–120 days before declining to levels similar to those found at Day 0 (L = 51.3 ± 0.7) (Figure 4).

The behaviour observed in the initial phase remains unclear and is also evident in chromaticity parameters a and b. The increase in surface water absorption in APGPM during this period (as indicated by rising X_w and a_w) would theoretically result in changes in surface refractive indices and increased light absorption, potentially leading to a less clear appearance of the samples. However, the opposite trend was observed. This suggests that, during the early storage phase, water diffuses into the deeper layers, where it preferentially adsorbs, leaving the porous and dry surface of APGPM exposed to the spectrophotometer's light beam, thereby increasing L values. After 30 days, APGPM may undergo simultaneous degradation processes within the matrix, including phenolic oxidation and polymerisation, as well as non-enzymatic browning reactions (Maillard reaction), among others (de Carvalho *et al*., 2020). Additionally, Δa and Δb values remained low (≤5.0), indicating minimal variations (a: 0.3 ± 0.0–2.6 ± 0.0, b: 15.2 ± 0.4–11.9 ± 0.7), which are considered non-critical and imperceptible to the human eye (Lucas-Aguirre *et al*., 2019). These or similar behaviours have been reported by several authors during the storage of other fruit powders, such as pineapple (Lima *et al*., 2019), banana (Barooah *et al*., 2018), jambolan (de Carvalho *et al*., 2020) and soursop (Chang *et al*., 2018a), among others.

Active compounds

Based on the initial content of active compounds and antioxidant activity of APGPM, favourable nutritional conditions are emphasised, particularly as a source of Vit. C. However, during storage, a decreasing trend was observed in antioxidant activity and bioactive compounds (Vit. C, β-Car, TF and TP) with storage time, making these variables the most critical during storage. The ANOVA analysis revealed that bioactive compounds and antioxidant activity were significantly influenced by storage time, temperature and their interaction.

During storage, the decrease in antioxidant activity (DPPH and ABTS) and TP and TF concentrations is associated with the increase in X_w and a_w over time and rising temperature (Figures 5A and 5B). This phenomenon affects molecular mobility, leading to the activation of hydrolytic or oxidative enzymes such as polyphenol oxidase, thereby inducing chemical oxidation reactions (Udomkun *et al*., 2016). The effect of temperature on phenolic compounds such as TP and TF is related to the changes they can undergo in their structure, by breaking the carbon–oxygen bond that causes the loss of hydroxyl (OH) groups responsible for their biological activities and antioxidant properties.

On the other hand, both temperature and storage time can impact β-Car contents through two primary degradation mechanisms: oxidation and isomerisation. Oxidation may occur through self-activation (autooxidation), light exposure (photooxidation) or enzymatic catalysis (enzymatic oxidation). Conversely, isomerisation, primarily influenced by temperature, involves the transformation of trans-β-Car into the cis-β-Car form, which exhibits lower biological activity.

During storage, the degradation of Vit. C is primarily driven by temperature, alongside other factors such as oxygen availability, pH, transition metals (iron and copper) and light exposure. Additionally, salts, a_w and the presence of other vitamins, among other factors, also play significant roles.

It is important to note that the Vit. C content in APGPM results from both its natural presence in pineapple and ginger and its addition in the binding solution during agglomeration. This distinction is relevant when interpreting degradation kinetics, as the stability of an added compound can differ from that of a naturally occurring one due to potential matrix effects. The food matrix may provide structural or chemical protection that influences degradation rates, either by limiting oxygen exposure or interacting with other bioactive compounds. Therefore, the observed degradation reflects both the loss of added Vit. C and the naturally occurring fraction, which may degrade at different rates depending on the storage conditions.

This degradation process involves oxidation, wherein ascorbic acid undergoes a reversible reaction to form dehydroascorbic acid, establishing an oxidation–reduction system. Subsequently, dehydroascorbic acid undergoes further oxidation, transforming into 2,3-diketogulonic acid, which lacks biological activity. Through Strecker degradation, 2,3-diketogulonic acid is further broken down, producing carbon dioxide and furfural (Badui, 2006).

Table 2 presents the kinetic fits of the active compounds and the antioxidant activity of APGPM during storage. It describes the most suitable reaction order (n), degradation rate constant (k), regression coefficients (R^2) and Ea. The optimal degradation kinetics for the quality attributes are as follows: zero-order (for TF and DPPH), first-order (for TP and Vit. C) and second-order (for ABTS and β-car).

In general, the degradation rate constant (k) was higher at elevated temperatures, attributed to the

Table 2. Kinetic adjustments of active compounds and antioxidant activity of APGPM during storage.

T	n	ABTS	DPPH	TP	TF	Vit. C	β-car
		2	0	0	1	1	2
15°C	*k* (mg day^{-1})	2×10^{-5}	25.9	0.7	4×10^{-3}	4.6×10^{-3}	9×10^{-6}
	R^2	0.91	0.93	0.93	0.77	0.96	0.98
25°C	*k* (mg day^{-1})	5×10^{-4}	33.9	0.8	5.4×10^{-3}	6.7×10^{-3}	$1x10^{-5}$
	R^2	0.96	0.88	0.96	0.96	0.98	0.95
35°C	*k* (mg day^{-1})	5×10^{-3}	27.2	0.8	6×10^{-3}	$\times 10^{-2}$	3×10^{-5}
	R^2	0.96	0.94	0.91	0.92	0.96	0.95
	Ea (kJ/mol)	34.0	2.3	5.6	20.6	0.17	44.0

APGPM, agglomerated pineapple–ginger powder mixture; β-Car, β-carotenoids; Ea, activation energy; k, degradation rate constant; n, reaction order; R^2, coefficient of determination; TF, total flavonoids; TP, total phenols; Vit. C, vitamin C.

thermosensitive nature of the active compounds analysed. This relationship serves as an indicator of their thermal degradation, where lower values indicate greater stability of the compound (de Carvalho *et al*., 2020). The degradation mechanisms of the active compounds may be affected by their physical state, as well as the presence of O_2 and high water activity, influencing the degradation rate due to increased molecular mobility (Cruz-Tirado *et al*., 2021).

On the other hand, Ea represents the minimum energy required for the degradation process to occur, meaning the energy needed for molecules to transition from the initial state to the formation of degradation products. In this context, a higher Ea is preferable, as it indicates greater stability of the active compounds during storage.

The Ea values obtained for the different bioactive compounds and antioxidant activity indicators varied, reflecting differences in their susceptibility to temperature-induced degradation. Higher Ea values were associated with compounds exhibiting greater resistance to thermal degradation, whereas lower values indicated increased vulnerability to storage conditions.

The reaction order observed for each parameter aligns with previous studies on the degradation kinetics of bioactive compounds in fruit powders (de Carvalho *et al*., 2020; Cruz-Tirado *et al*., 2021). The zero-order kinetics observed for TF and DPPH suggest a constant degradation rate, independent of the remaining concentration. In contrast, the first-order kinetics identified for total phenolics (TP) and Vit. C indicate that the degradation rate is proportional to the concentration of the compound, meaning that higher initial concentrations lead to faster degradation. The second-order kinetics for ABTS and β-Car suggest a more complex degradation mechanism, potentially influenced by interactions with other matrix components or secondary oxidative reactions.

Furthermore, the degradation rate constants increased with storage temperature, highlighting the thermosensitive nature of these compounds. This behaviour aligns with Arrhenius kinetics, reinforcing the importance of controlling temperature during storage to preserve the functional properties of APGPM. Additionally, the combined effects of high water activity and oxygen availability may accelerate oxidative reactions, further compromising the stability of bioactive compounds over time.

In summary, the degradation of active compounds in APGPM follows distinct kinetic models depending on the compound type, with temperature playing a key role in determining the rate of deterioration. Strategies such as minimising oxygen exposure, optimising moisture content, and implementing appropriate packaging solutions could help mitigate degradation and extend the shelf life of APGPM while maintaining its nutritional and antioxidant properties.

Figure 5 illustrates a gradual decrease in the ABTS and DPPH variables during 180 days of storage. Both exhibit similar retention levels at storage temperatures of 15, 25 and 35°C: 50.7, 51.9 and 33.2% for ABTS, and 53.6, 61.2 and 34.0% for DPPH, respectively. ABTS fluctuates from 287.4 to 145.8, 149.2, and 96.5 mg TE/100 g dry basis, while DPPH ranges from 324.1 to 173.6, 198.6, and 110.2 mg TE/100 g dry basis, respectively. Both antioxidant properties undergo greater degradation under the highest thermal stress (35°C), as previously discussed.

The kinetic models that provided the best regression fit for the ABTS and DPPH variables were of second and zero order, respectively. The k values were (2×10^{-5}, 50×10^{-5} and 500×10^{-5} mg/day^1) for ABTS and (25.9, 33.9 and 27.2 mg/day^1) for DPPH at 15, 25, and 35°C, respectively. A low activation energy requirement (Ea = 2.3 kJ/mol) for DPPH degradation was observed. Additionally, the regression coefficients obtained, ranging from a

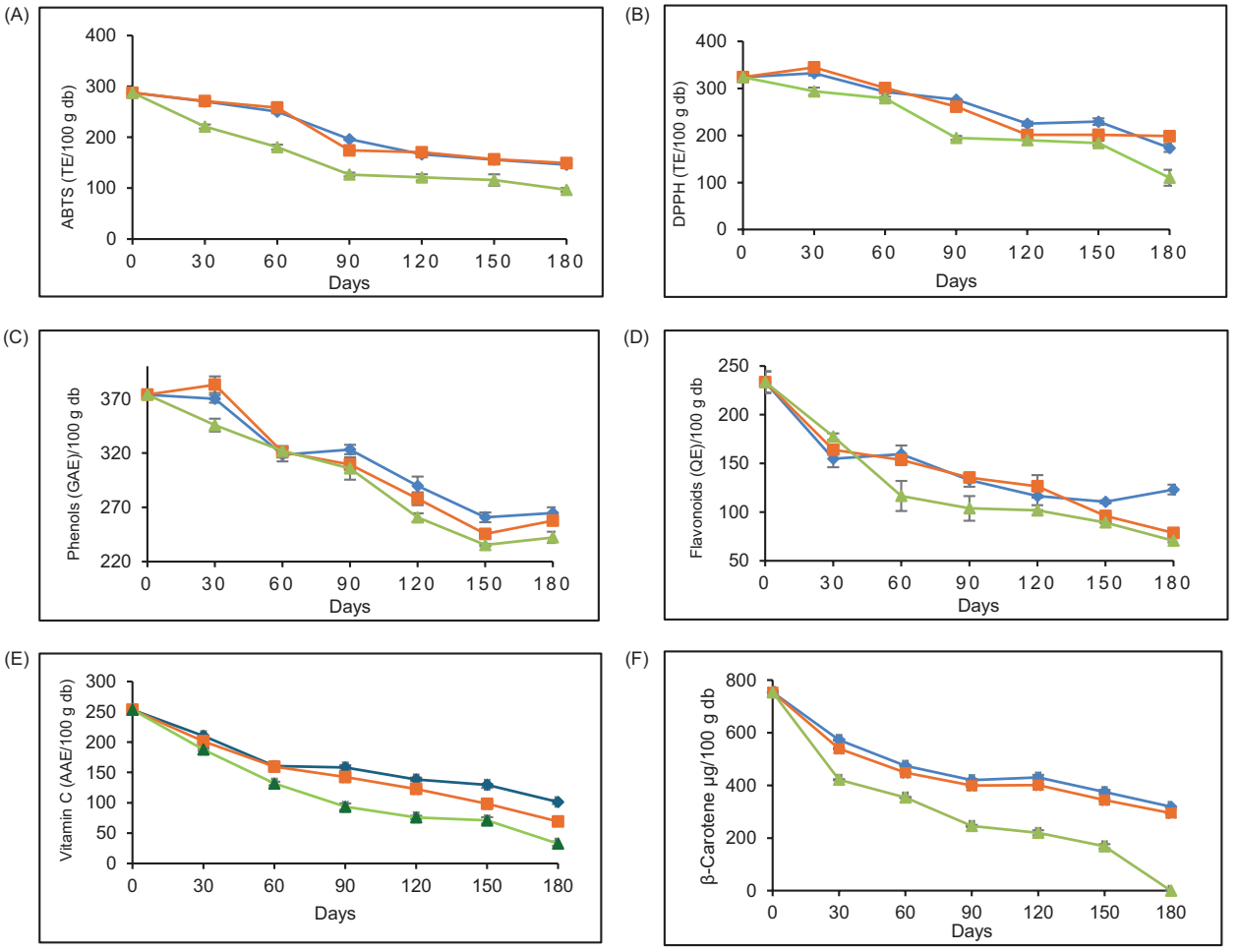

Figure 5. Evolution of antioxidant activity and active compounds of APGPM during storage (blue: 15°C, orange: 25°C and green: 35°C). (A) ABTS, (B) DPPH, (C) TP, (D) TF, (E) Vit. C, (F) β-Car.

minimum of 0.88 to a maximum of 0.96, are considered acceptable. Similar results were reported for dehydrated papaya stored at 30°C, with ABTS and DPPH retention levels of 66 and 42%, respectively (Udomkun *et al*., 2016). Conversely, Mishra *et al*. (2014) reported 83.7% retention levels in plums stored at 4°C.

The APGPM TP exhibited the lowest retention levels after 150 days of storage at 15, 25 and 35°C: 69.7, 65.6 and 62.9%, respectively. These values were very similar to those recorded at 180 days and, as expected, were lower at higher temperatures (35°C). Regarding reaction kinetics, TP followed a zero-order model with R^2 values of 0.93, 0.96 and 0.91 and k values of 0.7, 0.8 and 0.8 mg/day^1 at 15, 25 and 35°C, respectively, with an Ea of 5.6 kJ/mol.

Regarding the TF in APGPM, at 15, 25 and 35°C, their retention levels after 180 days of storage were 52.7, 33.6, and 30.1%, respectively. Figure 5D illustrates a greater TF loss at 15 and 25°C during the first 30 days (29.8 and 33.7%, respectively), whereas at 35°C, the highest loss occurred during the first 60 days (50.1%). The kinetic fit of TF followed a first-order model with R^2 values of 0.77, 0.96 and 0.92, and k values of 4×10^{-3}, 5.4×10^{-3} and 6×10^{-3} mg/day at 15, 25 and 35°C, respectively, with an Ea of 20.6 kJ/mol.

Udomkun *et al*. (2016) reported zero-order kinetics for dehydrated papaya stored in aluminium-laminated polyethylene, with a k value of 1.1/day (R^2 = 0.99). However, when polyamide/polyethylene packaging was used, the k value increased to 1.3/day^1 (R^2 = 0.97). Similarly, Daza *et al*. (2016) observed TP content losses exceeding 25% in all samples after 120 days of storage. Varastegani *et al*. (2019) reported retention values for spray-dried *N. sativa* TF after 12 months of storage, ranging between 58 and 68%. Conversely, de Carvalho *et al*. (2020) found that TF in jambolan powder stored at 4, 25 and 35°C remained highly stable, showing no significant statistical differences with respect to temperature or storage time. The decrease in TP and TF concentrations during

storage could be attributed to the activation of oxidative enzymes, such as polyphenol oxidase, or chemical oxidation triggered by molecular mobility, temperature, oxygen exposure or light (Zorić *et al*., 2017).

The Vit. C content in APGPM after 180 days of storage showed retention levels of 40.0, 27.2 and 12.8% at 15, 25 and 35°C, respectively. Figure 5E illustrates a progressive degradation over time, with higher degradation occurring at higher temperatures. The kinetic fit of Vit. C in APGPM followed a first-order mathematical model, with R^2 values of 0.96, 0.98 and 0.96, k values of 4.6×10^{-3}, 6.7×10^{-3} and 1×10^{-2} mg/day, and an Ea of 0.17 kJ/mol. This suggests that vitamin C is the least stable of the active components studied, as it requires less energy for its oxidative processes to occur, leading to the formation of dehydroascorbic acid—which is still bioavailable but less stable—and ultimately to its further oxidation.

Based on the results obtained, a reference portion of 30 g of APGPM is sufficient to prepare 250 g of a pineapple and ginger drink with approximately 11.0 °Brix. After 180 days of storage, this portion would provide 27.8, 19.0 and 8.8% of the daily Vit. C requirement, according to the World Health Organisation (WHO) recommendation of 100 mg of Vit. C per day, at 15, 25 and 35°C, respectively.

In Colombia, Resolution 821 of 2021 from the Ministry of Health and Social Protection establishes the daily reference nutrient requirement value (NRV-N) for Vit. C at 83 mg. Accordingly, a 30 g portion of APGPM provides 33.5, 22.9 and 10.7% of the NRV-N at storage temperatures of 15, 25 and 35°C, respectively.

Finally, the β-carotene (β-Car) content in APGPM after 180 days of storage showed retention levels of 42.3, 39.1 and 0% at 15, 25 and 35°C, respectively. It is important to note that, at 35°C, β-Car was not quantifiable using the HPLC calibration curve applied; therefore, its value was considered as 0. The evolution of TF over time revealed a progressive decrease throughout storage (Figure 2F), and ANOVA identified two homogeneous groups: one at 15 and 25°C, and another at 35°C, with significantly lower retention at 35°C across all time points. The kinetic fit of β-Car in APGPM followed a second-order mathematical model, with R^2 values of 0.95 and 0.95 and k values of 9×10^{-6}, 10×10^{-6} and 30×10^{-6} mg/day^1 for 15, 25 and 35°C, respectively, with an Ea of 44.0 kJ/mol.

Several studies have reported different reaction orders for the degradation of active compounds during storage in powdered food products: Zorić *et al*. (2017) reported zero and first-order kinetics for the degradation of phenolic compounds in sour cherry powder stored at 4, 20 and 37°C for 365 days. Ferrari *et al*. (2021) reported zero-order kinetics for Vit. C degradation in mango peel powder stored at 10, 15, and 25°C for 180 days; Shiby *et al*. (2017) reported first-order kinetics for Vit. C degradation in pineapple powder; Syamila *et al*. (2019) reported first-order kinetics for β-car, lutein and α-tocopherol in spinach powder stored at 4, 20 and 40°C. On the other hand, Daza *et al*. (2016) reported phenol losses under first-order kinetics in cagaita powder stored at 25°C for 150 days.

Conclusions

At the end of storage, X_w and a_w values confirmed the physicochemical stability of APGPM, with flowability classified as good to very good and cohesiveness ranging from low to intermediate. Among the colour parameters, luminance (L) was the most critical, while chromaticity values a and b remained stable (Δa^* and $\Delta b^* < 5$), making them imperceptible to the human eye. The degradation kinetics exhibited strong model fits (R^2 between 91 and 99%), except for TP at 15°C ($R^2 = 0.77$). Retention levels of active compounds and antioxidant capacity after 180 days of storage at 15, 25, and 35°C were: ABTS (50.7–33.2%), DPPH (53.6–34.0%), TP (69.7–62.9%), TF (52.7–30.1%), Vit. C (40.0–12.8%) and β-car (42.3–0%), confirming higher degradation rates at elevated temperatures.

The findings highlight the relevance of storage conditions in preserving the nutritional and functional quality of APGPM, with 15 and 25°C providing optimal stability. This study offers valuable insights into the shelf life prediction of APGPM, supporting its potential for commercial applications in functional food formulations. Furthermore, future research on sorption isotherms and phase transitions is recommended to refine shelf life estimations, ensuring better stability and product performance under different storage environments.

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Data Availability

Data will be made available on request.

Authors Contribution

Lina María Cardona contributed to the conceptualisation, methodology, validation, formal analysis, writing the

original draft, and visualisation. Misael Cortes-Rodríguez was involved in the study conceptualisation, methodology, formal analysis, writing – review & editing of the manuscript, supervision, project administration, and funding acquisition. Francisco Javier Castellanos Galeano and Jesús Humberto Gil G contributed to the conceptualisation, methodology, formal analysis, and supervision. Rodrigo Ortega-Toro contributed to the conceptualisation, validation and writing – review & editing of the manuscript and study visualisation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Conflicts of Interest

None.

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