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ORIGINAL ARTICLE

Physicochemical properties and thermal stability of *Hibiscus rosa-sinensis* flower dye with potential as a functional ingredient in foods

Propiedades fisicoquímicas y estabilidad térmica del colorante de flores de *Hibiscus rosa-sinensis* con potencialidades como ingrediente funcional en alimentos

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Abstract Natural plant-based dyes offer sustainable and functional alternatives to synthetic dyes. This study evaluated the stability and functional properties of a powdered dye derived from *Hibiscus rosa-sinensis* L., obtained by spray drying, using maltodextrin and xanthan gum. Flow properties and thermal stability were determined. The powder demonstrated efficient retention of bioactive compounds, with 672,6 mg GAE/100 g of total polyphenols, 400 mg QE/100 g of flavonoids, 380,9 mg C3G/100 g of anthocyanins, and an antioxidant capacity of 837,5 mM AAE/g. It had a moisture content of 7,64 % and high hygroscopicity (22,9 %), solubility of 87,8 %, and a fast dissolution time (47,3 s). The porosity (74,3 %) and the Carr (24 %) and Hausner (1,33) indices indicated acceptable flowability and low cohesiveness, suitable for industrial applications. The thermal degradation of anthocyanins followed first-order kinetics, with a rate constant ranging from 0,0143 d^{-1} at 25 °C to 0,0788 d^{-1} at 55 °C and an activation energy of 50,01 kJ/mol. The half-lives ($t_{1/2}$) and t_{90}% decreased with temperature, and the Q_{10} coefficient showed greater sensitivity between 25–35 °C. The combination of encapsulants stabilized the anthocyanins, preserving color and antioxidant activity. The powder has high technological and nutraceutical value as a functional natural colorant in foods, requiring controlled temperature and humidity during storage.

Keywords *Hibiscus rosa-sinensis* L., natural dye, antioxidant capacity, spray drying, thermal stability.

Resumen Los colorantes naturales de origen vegetal constituyen alternativas sostenibles y funcionales frente a los sintéticos. Este estudio evaluó la estabilidad y propiedades funcionales del colorante en polvo de *Hibiscus rosa-sinensis* L., obtenido por secado por atomización, utilizando maltodextrina y goma xantana. Se determinaron las propiedades de flujo y la estabilidad térmica. El polvo mostró retención eficiente de bioactivos, con 672,6 mg EAG/100 g de polifenoles totales, 400 mg EQ/100 g de flavonoides, 380,9 mg C3G/100 g de antocianinas, y capacidad antioxidante de 837,5 mM EAA/g. Presentó humedad de 7,64 % y elevada higroscopicidad (22,9 %), solubilidad de 87,8 % y rápido tiempo de disolución (47,3 s). La porosidad (74,3 %) y los índices de Carr (24 %) y Hausner (1,33) indicaron fluidez aceptable y baja cohesividad, adecuada para aplicaciones industriales. La degradación térmica de antocianinas siguió cinética de primer orden, con constante de velocidad de 0,0143 d^{-1} a 25 °C a 0,0788 d^{-1} a 55 °C y energía de activación de 50,01 kJ/mol. Los tiempos de vida media ($t_{1/2}$) y t_{90}% disminuyeron con la temperatura, y el coeficiente Q_{10} evidenció mayor sensibilidad entre 25–35 °C. La combinación de encapsulantes estabilizó las antocianinas, preservando color y actividad antioxidante. El polvo presenta alto valor tecnológico y nutracéutico como colorante natural funcional en alimentos, requiriendo control de temperatura y humedad en almacenamiento.

Palabras clave *Hibiscus rosa-sinensis* L., colorante natural, capacidad antioxidante, secado por atomización, estabilidad térmica.

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PUBLIS EDITORIAL

Introduction

The food industry is promoting "clean label" products, which requires replacing synthetic colorants with natural ingredients that offer added functionality. Plant-based colorants, rich in anthocyanins and polyphenols, not only provide color but also antioxidant activity and health benefits for consumers (Castañeda-Ovando et al., 2009; He & Giusti, 2010). This dual function makes them ideal for functional foods, but their industrial application requires ensuring stability against factors such as temperature and humidity.

Among the various production techniques, spray drying allows for the preservation of heat-sensitive compounds and the production of soluble, homogeneous powders that are easy to dose (Fang & Bhandari, 2011). This method is particularly valuable for sensitive pigments such as anthocyanins, whose degradation can compromise their functionality. The choice of coating agents and drying conditions directly influence the quality of the final powder, underscoring the importance of optimizing the process for food applications. Therefore, it is essential to evaluate parameters such as moisture content, solubility, flowability, hygroscopicity, and porosity, as these factors determine the shelf life and technological acceptance of the colorant.

Thermal stability must be quantified through accelerated degradation studies and kinetic models, which make it possible to predict shelf life, estimate critical parameters such as half-life and partial degradation times, the Q_{10} factor, and activation energy, and optimize storage conditions (Liao et al., 2024).

H. rosa-sinensis L. is a promising source of natural pigments and bioactive compounds. The production of a powdered dye from this species, along with its physicochemical characterization and an assessment of its thermal stability, is essential for validating its use as a functional ingredient and substitute for artificial dyes in "clean label" products, thereby maximizing its added value and bioactive functionality.

Methodology

H. rosa-sinensis (HRS) flowers were selected for uniformity; the petals and pistils were separated, then dried in an oven (YLD-6000, AISET, China) at 40°C for 72 hours, ground (Fumar, Italy), sieved (0.5 mm), and stored in amber bottles in a desiccator until use (Arencibia et al., 2026).

The extract was obtained by maceration with occasional stirring for 24 h at 60 °C in 50% (v/v) ethanol with 0.5% citric acid, at a ratio of 1 g HRS/20 mL. It was filtered under vacuum (ULVAC DTC-21), and the filtrate was stored at −32°C (Domestic, MF 110, Luxembourg) (Arencibia et al., 2023). The hydroalcoholic extract was concentrated in an RV 10 Basic rotary evaporator (IKA, Argentina) at 40°C and 85 s^{-1} under vacuum (ULVAC DTC-21) to 7% solids (Arencibia et al., 2026).

The concentrated extract was prepared for spray drying by adding DE 20 maltodextrin and xanthan gum as coating and stabilizing agents, adjusting the final total solids content to 20–25%. For 35 mL of extract, 10 g of maltodextrin and 0.073 g of xanthan gum were added, and the mixture was homogenized at 750 rpm. Subsequently, drying was performed in a mini spray dryer (B-290, BÜCHI, Switzerland) under controlled conditions: inlet temperature of 140 °C, outlet temperature of 56–58 °C, feed flow rate of 9 mL/min, atomization pressure of 742 mbar, and air flow rate of 667 L/h. The drying yield (DY) was calculated as the ratio of the mass recovered in the collector (excluding adhered dye) to the total mass of solids in the feed, expressed as a percentage of the initial processed solids (Felicó-Romero et al., 2026). This procedure yielded a stable, homogeneous, and easily handled powder, suitable for the evaluation of its physicochemical and functional properties.

The total polyphenol content was determined using the Folin-Ciocalteu method, with a reading at 765 nm (Slinkard & Singleton, 1977), and expressed as milligrams of gallic acid equivalents (GAE) per 100 g of powder. Total flavonoids were quantified using the $AlCl_3$ colorimetric method, with absorbance measured at 415 nm (Chang et al., 2002; Pękal & Pyrzynska, 2014), and expressed as mg of quercetin equivalents (QE) per 100 g of powder. Total anthocyanins were assessed using the differential pH method, with readings at 510 and 700 nm (Lee et al., 2005), and expressed as mg of cyanidin-3-glucoside (C3G) equivalents per 100 g of powder. Quantification was performed using external calibration curves, and all determinations were carried out in triplicate using a Rayleigh UV-1601 spectrophotometer (Beijing, China).

Antioxidant capacity was determined using the $ABTS^{+\bullet}$ method, following the procedure described by Apak et al. (2022), with slight modifications. The radical was generated from solutions of ABTS (19.4 mM) and ammonium persulfate (438 mM), incubated at 68 °C for 13 min, and diluted to an absorbance of 0.70 ± 0.02 at 734 nm. For the assay, 980 µL of the radical was mixed with 20 µL of the extract, incubated at 37 °C for 10 min, and the absorbance was measured at 734 nm. Quantification was performed using an external calibration curve with ascorbic acid (100–700 µM), using a UV–Vis spectrophotometer (Rayleigh UV-1601, Beijing, China). The results were expressed in millimoles of ascorbic acid equivalent (AAE) per gram of powder.

Moisture content was determined by drying in an oven at 105 °C (YLD-6000 AISET, China) and calculated by weight difference (da Rosa et al., 2019). Solubility was evaluated by quantifying the percentage of powder dissolved in distilled water through agitation, centrifugation, and drying of the recovered solids. It was calculated by comparing the initial weight with that of the dry solids (Cano-Chauca et al., 2005). Furthermore, hygroscopicity was determined after equilibrating the samples in a desiccator with a saturated NaCl solution (RH 75.29%, 25 °C) for 7 days (Tonon et al., 2008). Dissolution time was measured as the time required for the complete dissolution of 1 g of sample in 25 mL of distilled water under magnetic stirring at 900 rpm (Goula & Adamopoulos, 2010).

The flow properties of the powder were characterized by determining its bulk density, tapped density, and particle density. Based on these parameters, the Carr Index and the Hausner Index were calculated; these indices are used as indicators of the flowability and compressibility of the particulate material (Barbosa-Cánovas et al., 2005). Likewise, the porosity of the powder was estimated based on the ratio of bulk density to true density (Qiu et al., 2015). The angle of repose (θ) was determined using the static funnel method (Jinapong et al., 2008).

The powdered dye (0.20–0.25 g) was placed in Eppendorf tubes and stored at 25, 35, 45, and 55 °C in a YLD-6000 (AISET, China) to evaluate its thermal stability and AT degradation kinetics, following methodologies reported for extracts and powders of Hibiscus sabdariffa (Castañeda-Ovando et al., 2017; Cassol et al., 2020). Over a 21-day period, samples were collected in triplicate at the start and at varying intervals (4, 3, and 2 days) depending on the storage temperature.

The residual concentration of total anthocyanins over time was fitted to zero-order, first-order, and second-order kinetic models, with the most appropriate model selected based on the coefficient of determination (R^2). Based on the rate constant obtained, the half-life ($t_{1/2}$) and the time required for 10% degradation (t_{90}%) were calculated as indicators of stability. The effect of temperature on the degradation rate was evaluated using the Q_{10} coefficient, and the activation energy (E_a) was estimated using the Arrhenius model, following procedures reported for aqueous and powdered systems of Hibiscus sabdariffa anthocyanins (Castañeda-Ovando et al., 2017; Cassol et al., 2020).

All experiments were carried out in triplicate to ensure accuracy (n=3). Data are expressed as the mean (standard deviation). Analysis of variance (ANOVA) and Duncan's multiple range test ($p \leq 0.05$) were performed using IBM SPSS Statistics (V22.0, 2013, IBM, Armonk) software.

Results and discussion

The final product was obtained with a DY of 58.4%, which falls within the expected range for spray-dried botanical extracts (Fang & Bhandari, 2011). The results of the characterization of the HRS powdered dye are presented in Table 1.

Table 1. Bioactive compound content and antioxidant capacity of the powdered dye from *H. rosa-sinensis* L.

Indicator	Mean (standard deviation)
Total polyphenols (mg GAE/100 g)	672,6 (24,8)
Total flavonoids (mg QE/100 g)	400,0 (46,0)
Total anthocyanins (mg C3G/100 g)	380,9 (39,1)
Antioxidant capacity (mM AAE/g)	837,5 (4,83)

Note: GAE: gallic acid equivalents; QE: quercetin equivalents; C3G: cyanidin-3-glucoside equivalents; AAE: ascorbic acid equivalent.

Table 1 shows that HRS powder effectively retains bioactive compounds: total polyphenols (672.6 mg GAE/100 g), total flavonoids (400 mg EQ/100 g), and total anthocyanins (380.9 mg C3G/100 g). Compared to microencapsulated powders of *Hibiscus sabdariffa*, the polyphenol and anthocyanin contents fall within or exceed the ranges reported by Cid-Ortega and Guerrero-Beltrán (2020) and Leyva-López et al. (2024), while total flavonoids were somewhat lower than those reported by Leyva-López et al. (2024).

This confirms that, despite differences between species and microencapsulation conditions, anthocyanins constitute the predominant phenolic subgroup, contributing significantly to the total polyphenol fraction and antioxidant activity. The powder exhibited exceptional antioxidant capacity of 837.5 mM AAE/g, reflecting the preservation and concentration of bioactive compounds. These results highlight that the powder functions not only as a natural colorant but also as a high-value-added functional ingredient for the food and nutraceutical industries.

Table 2. Physicochemical parameters and flow properties of HRS powdered dye

Indicator	Mean (standard deviation)
Moisture (%)	7,64 (1,02)
Hygroscopicity (%)	22,9 (1,5)
Solubility (%)	87,8 (0,08)
Dissolution time (s)	47,33 (3,51)
Bulk density (g/mL)	0,258 (0,008)
Tapped density (g/mL)	0,339 (0,008)
Particle density (g/mL)	1,013 (0,001)
Porosity (%)	74,3 (0,02)
Carr index (%)	24 (4)
Hausner index	1,33 (0,14)
Angle of repose (°)	16,5 (1,8)

The moisture content of the dye obtained (Table 2) via spray drying (7.64 ± 1.02%) was lower than that of the freeze-dried powder (9.48 ± 0.02%) reported by Arencibia et al. (2026), indicating greater efficiency in water removal and promoting physicochemical and microbiological stability. The hygroscopicity (22.9 ± 1.5%) exceeded that of the freeze-dried powder (17.98 ± 0.14%), reflecting a greater affinity for water attributable to the amorphous structure and high surface area; this value is comparable to that of black cherry dye (25.2%) reported by Gaibor et al. (2022), underscoring the need for packaging under low-humidity conditions to prevent caking. Solubility was high (87.8 ± 0.08%), close to that of the freeze-dried powder (90.23 ± 0.36%) reported by Arencibia et al. (2026), and the dissolution time was fast (47.33 s), confirming that spray drying produces easily dispersible powders suitable for industrial applications.

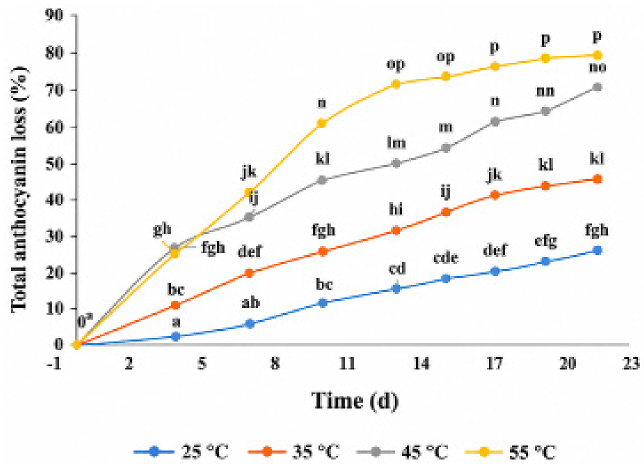
Regarding flow properties (Table 2), the powdered dye obtained had a bulk density of 0.258 ± 0.008 g/mL and a tapped density of 0.339 ± 0.008 g/mL, indicating good flowability and a low tendency to caking. The particle density (1.013 ± 0.001 g/mL) and high porosity (74.3 ± 0.02%) reflect amorphous and highly porous particles, characteristics typical of spray drying, which favor rapid reconstitution and high solubility. The Carr Index (24%) and the Hausner Index (1.33) classify the powder as having "acceptable flowability" and "low cohesiveness" (Jinapong et al., 2008), while the angle of repose of 16.5° indicates excellent flow. These properties suggest that the powder would not present operational problems in industrial dosing, mixing, or packaging.

Figure 1 shows that the degradation kinetics of the anthocyanin content in the powdered dye are strongly temperature dependent. Pigment loss increased significantly with time and temperature ($p \leq 0.05$), reaching final retention levels of 68% at 25 °C, 45% at 35 °C, 28% at 45 °C, and 22% at 55 °C. At elevated temperatures, the rapid initial decline suggests the activation of multiple degradation pathways, including thermally induced oxidation, hydrolysis of glycosidic bonds, and breakdown of the flavanone ring. The observed nonlinear kinetics reflect the structural complexity of anthocyanins and their high thermal susceptibility. These results highlight the need to control storage temperature to preserve the color intensity and antioxidant functionality of the pigment.

The kinetic parameters obtained from fitting the experimental data to zero-order, first-order, and second-order models are presented in Table 3, where the first-order model showed the highest coefficients of determination (R^2 = 0.9544–0.9961), indicating that it adequately describes the degradation of anthocyanins in HRS powder.

The first-order rate constant increased significantly with temperature, from 0.0143 d^{-1} at 25 °C to 0.0788 d^{-1} at 55 °C, confirming the pigment's thermal sensitivity and consistent with the degradation profile (Figure 2).

Figure 1. Loss of anthocyanin content in powdered dye



during storage under different temperature and time conditions (n = 3). Values with different letters indicate significant differences ($p \leq 0.05$).

This increase reflects the endothermic nature of the degradation reactions, in which the rise in thermal energy favors the breakdown of the flavanone ring, promoting the formation of colorless chalcone-type structures and, subsequently, brown-colored degradation products (Cavalcanti et al., 2011).

This behavior is consistent with that reported by Idham et al. (2012) for anthocyanins from *H. sabdariffa*, encapsulated with DE 20 maltodextrin and gum arabic, where the degradation constants of the powders also increased with temperature, exhibiting lower values than those in this study, but showing a similar thermal pattern and a similar stabilizing effect provided by encapsulation. Taken together, these results confirm that the thermal degradation of anthocyanins follows first-order kinetics and that temperature is the main factor modulating their stability, while the combination of maltodextrin with an additional polysaccharide effectively stabilizes the pigments and prolongs their shelf life in powder form.

Table 3. Kinetic parameters for the degradation of total anthocyanins in the powdered dye

Temperature (°C)	Order 0		Order 1		Order 2	
	k (mg g^{-1} d^{-1})	R^2	k (d^{-1})	R^2	k (mg^{-1} g d^{-1})	R^2
25	4,7047	0,9780	0,0143	0,9814	0,00005	0,9744
35	8,2812	0,9903	0,0294	0,9961	0,0001	0,9882
45	11,424	0,9562	0,0528	0,9859	0,0003	0,9287
55	14,362	0,9033	0,0788	0,9544	0,0006	0,9518

The parameters derived from the first-order rate constant (Table 3) show a marked dependence of anthocyanin stability on temperature. The half-life ($t_{1/2}$) and the time required for 10% degradation (t_{90}%) decreased dramatically as temperature increased, confirming the strong thermal effect on degradation kinetics. $t_{1/2}$ decreased from 48.47 days at 25 °C to 8.05 days at 55 °C, representing a reduction of approximately sixfold, while t_{90}% decreased from 7.34 to 1.21 days, respectively. These results are consistent with the degradation profile (Figure 2) and with the high k values reported in Table 3. The t_{90}% parameter, widely used to estimate the shelf life of sensitive products, indicates that the dye exhibits limited stability at elevated temperatures, which restricts its prolonged storage under uncontrolled conditions.

Table 4. Stability parameters for anthocyanin degradation

Temperature (°C)	$t_{1/2}$ (days)	t_{90}% (days)	Q_{10}
25	48,47	7,34	-
35	23,57	3,57	2,05
45	13,00	1,96	1,81
55	8,05	1,21	1,61

A comparison with *H. sabdariffa* powders encapsulated with DE 20 maltodextrin and gum arabic, as reported by Idham et al. (2012), shows that, although the half-lives are shorter in the system studied, both exhibit a consistent trend: $t_{1/2}$ decreases as temperature increases (Table 4). This confirms that encapsulation with maltodextrin combined with an additional polysaccharide provides a protective effect on anthocyanins, improving the thermal stability of the pigment and extending its shelf life in powder form.

Table 4 shows that the Q_{10} coefficient decreased as the temperature increased (from 2.05 to 1.61), a typical behavior in food degradation kinetics, indicating greater thermal sensitivity in the lower temperature range (25–35 °C) compared to the higher range (45–55 °C). A Q_{10} value of 2.05 implies that the degradation rate doubles with a 10 °C increase (Rasmusson et al., 2019), confirming the strong impact of temperature on pigment stability.

The activation energy (Ea) is a key parameter for predicting how temperature affects the rate of degradation (Table 4). The Ea value obtained in this study (50.01 kJ/mol) indicates that the anthocyanins present in the powdered dye are highly sensitive to temperature changes, such that even moderate increases can significantly accelerate the degradation of the pigment. This value is consistent with that reported for other anthocyanin systems fitted to a first-order model, such as black rice bran powder (Loypimai et al., 2016).

In the study of *H. sabdariffa* extracts, similar values have been observed, with Ea in the range of 47–61 kJ/mol (Cisse et al., 2009), suggesting that the thermal dependence of the anthocyanins in the powdered dye falls within the expected range for this type of pigment. These results highlight the need to carefully control storage conditions (temperature, light, and humidity) to preserve the stability and functionality of the pigment.

Conclusions

A natural powdered dye was obtained from *Hibiscus rosa-sinensis* L. flowers via spray drying; it had a high content of bioactive compounds, notably polyphenols, flavonoids, and anthocyanins, as well as high antioxidant capacity. The product exhibited high solubility, rapid reconstitution, and flow properties suitable for industrial operations, although its marked hygroscopicity requires moisture-barrier packaging. The degradation of anthocyanins followed first-order kinetics and showed strong temperature dependence, evidenced by the reduction in $t_{1/2}$ and t_{90}% as thermal storage increased. The activation energy confirmed the pigment's sensitivity to heat. Overall, the combination of maltodextrin and xanthan gum allowed for the stabilization of phenolic compounds and the preservation of the colorant's functionality, positioning it as a promising ingredient for "clean label" foods, with storage recommended below 25 °C.

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Conflicts of interest

The authors declare that they have no conflicts of interest.

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Data availability statement

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Statement on the use of AI

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