



# Influence of experimental temperatures on drying kinetics of *Theobroma speciosum* tea flowers

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## Abstract

Drying is a crucial step in ensuring the quality of teas, as it can alter their chemical composition and organoleptic characteristics. In the present study, we examined the effects of different drying temperatures (ranging from 60 to 90°C) using hot air drying (HAD) on *Theobroma speciosum* flowers concerning drying kinetics, color, digestibility, and antioxidant capacity. From the obtained curves, we analyzed the physical aspects of *T. speciosum*, along with the moisture loss throughout the process, and the impact of different temperatures. This revealed that the Midilli mathematical model was the most suitable, where a set of equations was used to predict the analysis method's behavior. The results demonstrated that drying at 60°C was more effective in terms of drying rates, diffusivity, and bioactive compound content, making it a promising option for the pre-treatment of *T. speciosum* flower tea.

**Keywords:** *T. speciosum*; drying; antioxidants; Midilli mathematical model.

**Practical Application:** Drying at 60°C preserves bioactive compounds and optimizes the quality properties of tea.

## 1 INTRODUCTION

Edible flowers have been used in traditional medicine and cuisine worldwide, adding delightful flavors and providing valuable bioactive compounds such as polyphenols and anthocyanins when used in salads, desserts, beverages, and tea blends (Takahashi et al., 2020). Bioactive compounds in edible flowers are potent antioxidants that enhance nutritional value, improve flavor and health, protect against chronic diseases, and prevent oxidative deterioration of food (Zawiślak et al., 2022).

Numerous studies have extensively documented the antioxidant activity and phenolic composition of *Theobroma* species (*T. cacao*, *T. bicolor*, and *T. grandiflorum*) in their pulp juice, along with cupuassu pulp and seeds, as well as cupuassu and cocoa liquors and cocoa powder (Mar et al., 2024). *Theobroma speciosum*, known as “cacaui” in Brazil, is considered a prominent unconventional food plant. The aqueous extract of *T. speciosum* flowers exhibit fractions rich in phenolic compounds, which are directly related to its antioxidant capacity (Moreira Mar et al., 2021).

Optimal drying conditions are essential in food processing to preserve bioactive compounds by preventing enzymatic degradation and microbial growth (Zawiślak et al., 2022). Hot

air drying (HAD) is a widely used and more affordable method for industrial drying due to its faster process (Shi et al., 2019).

Dehydration with hot air is common for preserving plant materials. However, high temperatures can degrade flavor, color, and bioactive compounds. Lower drying temperatures help retain nutritional and sensory quality (Marcel et al., 2016). The choice of drying techniques depends on factors such as the type of product, the availability of a dryer, cost considerations, and desired time and energy consumption (Selvakumar, 2023).

Antioxidant properties of tea infusions vary with brewing conditions. Phytochemical release depends on time and temperature, and these factors influence the tea's health benefits. (Zhang et al., 2020). Moreover, the excellence of the tea infusion hinges on a range of factors. These encompass the type of tea, duration of brewing, temperature of the water, ratio of water to tea, and brewing time (Safdar et al., 2016).

This study analyzed moisture removal (MR) in *T. speciosum* flowers using drying models, determined diffusion coefficients, and evaluated the impact of drying air temperature on physico-chemical properties, color, phenolic content, and antioxidant capacity before and after digestion.

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## Relevance of the work

This study is relevant as it investigates the impact of different drying temperatures on the quality of *T. speciosum* flowers, with potential use in functional teas. Drying directly affects the preservation of bioactive compounds and sensory characteristics. The research analyzed drying kinetics, mathematical models, and parameters such as antioxidant activity, color, and bioaccessibility. Results showed that drying at 60°C was the most efficient, preserving high levels of functional compounds and ensuring better final product quality. This contributes to the development of a more efficient drying process, with reduced energy consumption and enhanced nutritional and functional value of the product.

## 2 MATERIALS AND METHOD

### 2.1 Collection and preparation of flower teas

*T. speciosum* flowers were collected from Manaus (3°6'26"S, 60°1'34"W; SisGen A26CD5E) and dried in a MYLABOR oven at 60, 70, 80, and 90°C with an air velocity of 850 m<sup>3</sup>·h<sup>-1</sup>. For each treatment, 5 g of flowers was sampled every 10 min to monitor moisture content over drying times from 0 to 240 min. The samples were then stored in a desiccator for chemical analysis. The final dried samples (0.5 g, < 0.15 mm) were used to prepare infusions, following specific mass-to-volume ratios. All procedures were performed in triplicate.

### 2.2 Drying kinetics and effective diffusivity

In order to establish the drying kinetics of *T. speciosum* flowers and analyze the mathematical dehydration models, the moisture ratio was calculated from the experimental data using Equation 1.

$$MR = \frac{(U(t) - U_{eq})}{(U_i - U_{eq})} \quad (1)$$

The experimentally obtained drying curves were fitted using the following models: the Midilli model (Equation 2) (Midilli et al., 2002), the Henderson and Pabis model (Equation 3), the Page model (Equation 4), and the Lewis model (Equation 5). Parameter estimation was carried out using RStudio<sup>®</sup> software version 2023.06.1+524 for Windows, where a non-linear regression analysis was conducted using the Non-Linear Regression package.

$$MR = a \cdot \exp(-k \cdot t^N) + b \cdot t \quad (2)$$

$$MR = a \cdot \exp(-k \cdot t) \quad (3)$$

$$MR = \exp(-k \cdot t^N) \quad (4)$$

$$MR = \exp(-k \cdot t) \quad (5)$$

To identify the best-fitting model for the experimental data, performance indices described by Ross (1996) were applied: root mean square error (RMSE), percentage prediction error (%SEP), bias factor ( $B_f$ ), and accuracy factor ( $A_f$ ) (Equations 6, 7, 8 and 9). Data processing was conducted using RStudio<sup>®</sup> version 2023.06.1+524 for Windows.

$$REQM = \sqrt{\frac{\sum(\text{obs} - \text{pred})^2}{n}} t \quad (6)$$

$$\%SP = \frac{100}{\text{mean obs}} \sqrt{\frac{\sum(\text{obs} - \text{pred})^2}{n}} t \quad (7)$$

$$Bf = 10^{\frac{\sum \log \frac{\text{pred}}{\text{obs}}}{n}} \quad (8)$$

$$Af = 10^{\frac{\sum |\log \frac{\text{pred}}{\text{obs}}|}{n}} \quad (9)$$

The temperature dependence of effective diffusivity can be described by the Arrhenius equation. The activation energy ( $E_a$ ) and pre-exponential factor ( $D_o$ ) are determined from the slope and intercept of the  $\ln(D_{\text{eff}})$  versus  $1/T$  plot (Uribe et al., 2014) (Equation 10).

$$D_{\text{eff}} = D_o \exp\left(\frac{-E_a}{RT}\right) \quad (10)$$

where:

$R$ : the universal gas constant (8.314 J mol<sup>-1</sup> K<sup>-1</sup>);

$E_a$ : the activation energy (kJ mol<sup>-1</sup> and  $D_o$  the Arrhenius factor (m<sup>2</sup> s<sup>-1</sup>))

$T$ : the absolute temperature (K).

### 2.3 Digestibility assay

The effect of drying on bioaccessibility in *T. speciosum* was evaluated through in vitro digestion (INFOGEST, without oral phase), assessing antioxidant potential before and after gastric and intestinal phases (de Souza Carvalho et al., 2020).

### 2.4 Antioxidant capacity assessment and total phenolic contents

For the pre- and post-digestibility samples, antioxidant activities were evaluated using the 2,2-Diphenyl-1-picrylhydrazyl (DPPH<sup>•</sup>) and 2,2'-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS<sup>•+</sup>) radical scavenging methods, as well as the ferric-reducing antioxidant power (FRAP) and total phenolic content (TPC) (Mar et al., 2021). The TPC was determined using the Folin-Ciocalteu colorimetric method. Results were expressed as mg of gallic acid equivalents (GAE) for 1 mL of tea. These analyses were performed with slight modifications from a previous report using the Epoch 2 Biotek microplate reader (Moreira Mar et al., 2021).

## 2.5 Color analysis

Color analysis of the samples was conducted using a Delta-Vista spectrophotometer (450G, DeltaColor, Brazil) with LED light, D65 illuminant, and a 10° observer, evaluating the  $L^*$ ,  $a^*$ , and  $b^*$  coordinates. A tea sample without temperature influence was used as the white reference (Mar et al., 2020).

## 2.6 Statistical analysis

Analysis of variance (ANOVA) was conducted in R (v3.5.1), and treatment means were compared using Tukey's test at a 5% significance level ( $p \leq .05$ ).

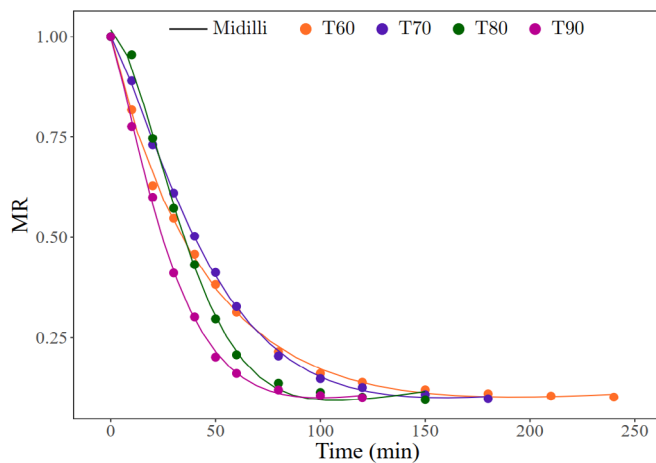
# 3 RESULTS AND DISCUSSION

## 3.1 Drying kinetics modeling

Drying curves for *T. speciosum* flowers at four temperatures, displayed in Figure 1, show that MR changes over drying time. All curves followed an exponential pattern. Equilibrium moisture content, approximately 0.02 g of water per gram of dry matter, was achieved at all temperatures, with quicker attainment at higher temperatures. At 80 and 90°C, the equilibrium moisture content was reached between 150 and 120 min, respectively, while at 60 and 70°C, drying took over 180 min under constant airflow.

Drying *Camellia sinensis* flowers to 0.08 g H<sub>2</sub>O/g dry matter took 180 min at 60°C and 60 min at 120°C. Higher temperatures increased vapor pressure, speeding up moisture migration. Thus, drying was faster at elevated temperatures (Shi et al., 2019). Drying *Cynara cardunculus* L. flowers at 35°C took 15 h. Each 10°C increase significantly reduced the drying time, reaching 2.5 h at 65°C; it can be said that higher temperatures greatly accelerated MR (Guiné et al., 2019).

Table 1 shows the constants, coefficient of determination ( $R^2$ ), and mean estimated standard errors (SEs) for each model obtained via nonlinear regression. The drying rate constant "k" was higher at 60°C for all models, except for the Midilli model,



**Figure 1.** Experimental drying curves modeled by the Midilli equation for samples of *T. speciosum* flowers at different drying temperatures.

where the elevated drying air temperature led to a higher drying rate, explaining the observed divergence.

Among the four models tested, the Midilli model best fit the drying data of *T. speciosum* flowers, showing the lowest mean errors and highest  $R^2$  values across all temperatures, with relative errors below 10%, indicating a good fit (Guiné et al., 2019).

The Midilli model was found to be the most suitable for describing the drying process in various fruit studies, such as pineapple drying at temperatures of 40, 50, and 60°C. To determine the best-fitting model, performance indicators from Ross (1996) were used, as outlined in Table 2. According to Ross (1996), the Midilli model showed the lowest RMSE and %SEP values, indicating a better fit to the experimental data, followed closely by the Page model. It also had the most favorable bias factor ( $B_f$ ), closest to the ideal value of 1, reinforcing its predictive accuracy.

Finally, the accuracy factor ( $A_f$ ) represents the average difference between predicted and observed data. Enhanced model accuracy is indicated by a reduced  $A_f$  value. For the temperatures under study, the Midilli model exhibited minimized values.

The Midilli model demonstrated the best fit to the experimental data at all four temperatures studied. Notably, the Page model yielded results comparable to the Midilli model within these temperature ranges.

**Table 1.** Kinetic and statistical parameters obtained for *T. speciosum* samples.

Model	Temp.	Model constants	$R^2$	SE
Midilli	60	$n = 9.617 \times 10^{-1}$ $k = 2.430 \times 10^{-2}$	0.9985	$3.25 \times 10^{-2}$ $3.19 \times 10^{-3}$
	70	$n = 1.240$ $k = 7.626 \times 10^{-3}$	0.9995	$2.96 \times 10^{-2}$ $8.99 \times 10^{-4}$
	80	$n = 1.583$ $k = 2.761 \times 10^{-3}$	0.9978	$8.39 \times 10^{-2}$ $8.86 \times 10^{-4}$
	90	$n = 1.227$ $k = 1.441 \times 10^{-2}$	0.9990	$4.40 \times 10^{-2}$ $2.27 \times 10^{-3}$
Henderson and Padis	60	$a = 0.95911$ $k = 0.01768$	0.9836	$3.60 \times 10^{-2}$ $1.24 \times 10^{-3}$
	70	$a = 1.027959$ $k = 0.018017$	0.9920	$2.31 \times 10^{-2}$ $7.61 \times 10^{-4}$
	80	$a = 1.085153$ $k = 0.022798$	0.9725	$4.84 \times 10^{-2}$ $1.84 \times 10^{-3}$
	90	$a = 1.010499$ $k = 0.028638$	0.9891	$3.15 \times 10^{-2}$ $1.59 \times 10^{-2}$
Page	60	$n = 0.791108$ $k = 0.042596$	0.9877	$4.69 \times 10^{-2}$ $8.24 \times 10^{-3}$
	70	$n = 1.060223$ $k = 0.013702$	0.992	$5.82 \times 10^{-2}$ $3.24 \times 10^{-3}$
	80	$n = 1.345806$ $k = 0.005645$	0.9836	$1.37 \times 10^{-1}$ $2.96 \times 10^{-3}$
	90	$n = 0.999999$ $k = 0.028303$	0.9885	$7.78 \times 10^{-2}$ $8.14 \times 10^{-3}$
Lewis	60	$k = 0.0187170$	0.9884	$9.66 \times 10^{-4}$
	70	$k = 0.0174053$	0.9909	$5.77 \times 10^{-4}$
	80	$k = 0.020768$	0.9702	$1.52 \times 10^{-3}$
	90	$k = 0.02830$	0.9885	$1.18 \times 10^{-3}$

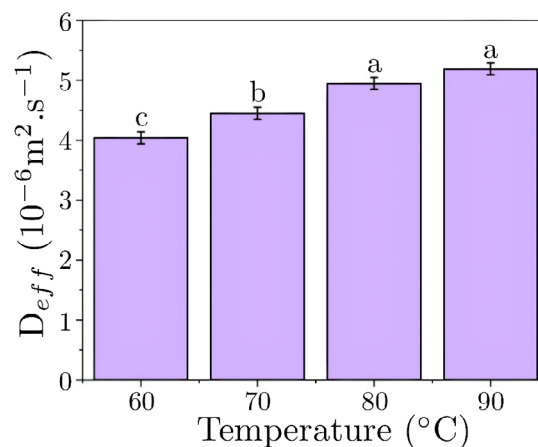
**Table 2.** Performance indicators for each model.

Model	Temp.	Performance indicators	Model	Temp.	Performance indicators
Midilli	60	REQM = 0.0480	Page	60	REQM = 0.0511
		%SEP = 13.1800			%SEP = 14.0329
		$B_f = 1.0038$ $A_f = 1.0041$			$B_f = 1.0132$ $A_f = 1.0010$
	70	REQM = 0.0470		70	REQM = 0.0340
		%SEP = 10.9160			%SEP = 7.9146
		$B_f = 1.0392$ $A_f = 1.0374$			$B_f = 1.0017$ $A_f = 1.0153$
	80	REQM = 0.0921		80	REQM = 0.0792
		%SEP = 21.7566			%SEP = 18.7104
		$B_f = 1.0493$ $A_f = 1.0368$			$B_f = 0.9382$ $A_f = 1.1152$
	90	REQM = 0.1401		90	REQM = 0.0935
		%SEP = 37.0891			%SEP = 24.7549
		$B_f = 1.0718$ $A_f = 1.0580$			$B_f = 0.9403$ $A_f = 1.1158$
Henderson and Padis	60	REQM = 0.1401	Lewis	60	REQM = 0.0582
		%SEP = 37.0891			%SEP = 15.9910
		$B_f = 1.0718$ $A_f = 1.0580$			$B_f = 0.9953$ $A_f = 1.0283$
	70	REQM = 0.0563		70	REQM = 0.0333
		%SEP = 15.4749			%SEP = 7.7470
		$B_f = 0.9998$ $A_f = 1.0218$			$B_f = 1.0080$ $A_f = 1.0059$
	80	REQM = 0.0688		80	REQM = 0.0685
		%SEP = 16.2672			%SEP = 16.1879
		$B_f = 0.9923$ $A_f = 1.0307$			$B_f = 1.0038$ $A_f = 1.0160$
	90	REQM = 0.0952		90	REQM = 0.0935
		%SEP = 25.2171			%SEP = 24.7549
		$B_f = 0.9381$ $A_f = 1.1192$			$B_f = 0.9403$ $A_f = 1.1159$

It can be observed that, for this model, the average adjusted coefficient of determination was 0.999, and according to Silva et al. (2020), values above 98% confirm a strong fit of the models to the dehydration process. Among them, the Midilli model best described the mass loss behavior during the drying of *Viola tricolor* × *Wittrockiana* (*Viola wittrockiana*) flowers (Midilli et al., 2002).

The variation in *T. speciosum* flowers can be explained by the fact that higher temperatures increase the vapor pressure inside the flowers, resulting in faster removal of moisture from the interior to the surface of the flowers. The  $D_{eff}$  values were  $4.15 \times 10^{-6}$ ,  $4.52 \times 10^{-6}$ ,  $4.96 \times 10^{-6}$ , and  $5.27 \times 10^{-6} \text{ m}^2 \cdot \text{s}^{-1}$  in the range of 60–90°C (Figure 2). The  $D_{eff}$  values were reported to be  $2.68 \times 10^{-9}$ ,  $3.86 \times 10^{-9}$ ,  $5.27 \times 10^{-9}$ , and  $8.66 \times 10^{-9} \text{ m}^2 \cdot \text{s}^{-1}$  in flowers of *C. sinensis* (Shi et al., 2019).

Higher drying temperatures increased  $D_{eff}$  values, as the added heat enhanced water molecule kinetic energy and moisture diffusivity. This behavior followed an Arrhenius-type relationship, with a strong linear correlation ( $R^2 = 0.996$ ) between the natural logarithm of  $D_{eff}$  and the inverse of absolute temperature.


**Figure 2.** Effective diffusivity in *T. speciosum* flowers.

### 3.2 Color analysis of fruits

Color parameters of *T. speciosum* flowers under different temperatures are shown in Table 3. As the drying air temperature and air velocity decreased, leading to longer drying times, the  $L^*$  and  $b^*$  values decreased, while the  $a^*$  value increased.

### 3.3 Antioxidant activity and bioaccessibility

The TPC of dried *T. speciosum* samples decreased with higher drying temperatures, ranging from 515.07 to 130.45  $\mu\text{molGA/mL}$  before digestion and from 81.10 to 8.92  $\mu\text{molGA/mL}$  after digestion. Despite this reduction, no significant differences were observed across temperatures ( $p < .05$ ), with the lowest TPC retention at 80°C. Polyphenolics are sensitive to heat, and extended exposure to high temperatures leads to permanent chemical alterations in these compounds (Vashisth et al., 2011) (Table 4).

Higher drying temperatures reduce drying time and may enhance bioactive compound levels. However, optimizing time and temperature is essential to preserve and recover phenolics based on each product's specific composition (Vashisth et al., 2011).

The means of antioxidant activities for *T. speciosum* samples, measured by the DPPH (1,384.75  $\mu\text{M Trolox/mL}$ ), ABTS (1,842.11  $\mu\text{M Trolox/mL}$ ), and FRAP (1,964.51  $\mu\text{M Fe (II)/mL}$ ) assays, showed the highest antioxidant capacity for T60 samples, followed by TP samples (1,199.75  $\mu\text{M Trolox/mL}$  for DPPH, 1,842.10  $\mu\text{M Trolox/mL}$  for ABTS, and 362.63  $\mu\text{M Fe(II)/mL}$  for FRAP), with no significant differences ( $p > .05$ ) compared to the other samples.

The health benefits of tea consumption are primarily based on its antioxidant activity (Liu et al., 2018). The antioxidant capacities of T60 and TP infusions were influenced by infusion conditions, showing significantly higher ABTS and DPPH scavenging activity than T70, T80, and T90. Controlled drying at lower temperatures enhanced the antioxidant capacity, whereas higher drying temperatures reduced it, aligning with previous findings (Kowalska et al., 2021).

The post-digestion bioavailability of antioxidants is highly significant. Many researchers use *in vitro* digestion methods to

**Table 3.** Color parameters for *T. speciosum* flowers.

Sample	L*	a*	b*	ΔE*
T60°C	21.767 ± 2.157 <sup>a</sup>	8.313 ± 1.432 <sup>a</sup>	4.522 ± 1.005 <sup>ab</sup>	10.589 ± 4.162 <sup>a</sup>
70°C	23.032 ± 1.391 <sup>a</sup>	8.072 ± 1.979 <sup>a</sup>	3.743 ± 1.120 <sup>b</sup>	13.031 ± 2.685 <sup>a</sup>
80°C	23.239 ± 2.229 <sup>a</sup>	9.393 ± 1.473 <sup>a</sup>	5.313 ± 1.317 <sup>a</sup>	13.431 ± 4.302 <sup>a</sup>
90°C	21.911 ± 2.037 <sup>a</sup>	6.734 ± 1.821 <sup>b</sup>	4.539 ± 1.453 <sup>ab</sup>	10.868 ± 3.931 <sup>a</sup>

L\*: lightness; a\*: the red-green coordinate; b\*: the yellow-blue coordinate; ΔE\*: the color variation. Means that do not share the same letters in the column (a-b) are significantly different.

**Table 4.** Values of DPPH, ABTS, and FRAP before and after *in vitro* digestion of *T. speciosum* flower teas.

Samples	DPPH (μM TE/mL)	ABTS (μM TE/mL)	TPC (mg GAE/mL)	FRAP (μM Fe(II)/mL)
TP	1,199.7 ± 10.9 <sup>b</sup>	1,842.1 ± 13.5 <sup>a</sup>	362.6 ± 1.0 <sup>b</sup>	1,509.7 ± 3.6 <sup>b</sup>
T60	1,384.7 ± 9.0 <sup>a</sup>	1,608.8 ± 6.9 <sup>b</sup>	515.1 ± 0.7 <sup>a</sup>	1,964.5 ± 2.3 <sup>a</sup>
T70	831.4 ± 5.7 <sup>d</sup>	1,422.1 ± 10.2 <sup>c</sup>	235.3 ± 1.2 <sup>d</sup>	1,170.7 ± 2.8 <sup>d</sup>
T80	674.7 ± 10.0 <sup>e</sup>	1,072.9 ± 7.5 <sup>d</sup>	130.4 ± 1.4 <sup>e</sup>	790.1 ± 3.9 <sup>e</sup>
T90	876.4 ± 8.8 <sup>c</sup>	785.4 ± 10.2 <sup>e</sup>	262.6 ± 0.8 <sup>c</sup>	1,295.6 ± 4.6 <sup>c</sup>
TPD	443.1 ± 8.0 <sup>i</sup>	243.2 ± 8.4 <sup>j</sup>	8.9 ± 0.8 <sup>j</sup>	644.1 ± 4.6 <sup>j</sup>
T60D	578.1 ± 7.6 <sup>g</sup>	604.3 ± 6.7 <sup>f</sup>	65.8 ± 1.0 <sup>g</sup>	713.0 ± 3.4 <sup>g</sup>
T70D	450.6 ± 8.8 <sup>i</sup>	485.4 ± 3.8 <sup>g</sup>	16.3 ± 1.2 <sup>i</sup>	657.5 ± 2.3 <sup>i</sup>
T80D	626.4 ± 7.6 <sup>f</sup>	340.9 ± 12.0 <sup>h</sup>	81.1 ± 1.3 <sup>f</sup>	749.7 ± 4.5 <sup>f</sup>
T90D	523.9 ± 7.6 <sup>h</sup>	546.5 ± 11.7 <sup>i</sup>	54.4 ± 1.3 <sup>h</sup>	678.6 ± 3.9 <sup>h</sup>

Results are expressed as mean ± standard deviation (n = 3); <sup>a-i</sup>Different letters in the same column are significant (p < .05); μM TE/mL: micromoles of Trolox equivalent per millimeters of sample; μM Fe(II)/mL: micromolar of ferrous sulfate per millimeters of sample; mg GAE/mL: milligram of gallic acid equivalent per millimeters of sample.

evaluate the stability of antioxidants found in foods, meals, and supplements (Liu et al., 2018). During digestion, the antioxidant potential of T60 and TP samples decreased significantly (58.25–97.54%) due to pH and enzyme conditions, reducing the bioactive compound levels in *T. speciosum* flowers.

Antioxidant capacity is affected by polyphenol levels and pH changes during digestion. As digestion progresses from the gastric to the intestinal phase, structural changes in polyphenols occur, altering their activity. In the intestine's alkaline environment (pH 6–8), polyphenols may degrade or transform into undetected forms with different properties and bioactivities. These changes are consistent with known polyphenol metabolism patterns, as reported in previous studies (Liu et al., 2020).

The antioxidant properties of grapefruit and pineapple juices increased before digestion but declined after pancreatic digestion due to alkaline pH. This suggests antioxidant instability. Structural changes in polyphenols may have affected their detectability, as noted by José Jara-Palacios et al. (2018).

## 4 CONCLUSIONS

This study evaluated the drying kinetics of *T. speciosum* flowers and compared mathematical models using performance indicators. The Midilli model best fit the data but required four parameters, while the Page model showed similar accuracy with

only two. Drying at 60°C led to faster equilibrium, and diffusion coefficients and activation energies followed the Arrhenius equation, indicating increased moisture diffusion with higher activation energy. Samples dried at 60°C retained high levels of bioactive compounds, antioxidant activity, and color quality. Drying for over 180 min at 60°C proved optimal for efficiency and quality, offering valuable insights for processing tea flowers.

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