

# Development and Comparison of Drying Tropical Herbal Strategies for *Annona muricata* Leaves: Integrating of Effective Moisture Diffusivity Using Antioxidant Activity, FTIR Ratios, and Color Attributes

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Received: 14 October 2025, Revised: 27 October 2025, Accepted: 3 November 2025, Published: 30 January 2026

## Abstract

Drying strategy employed exerts a significant influence on the kinetics and functional quality of *Annona muricata* (soursop) leaves. However, the current literature offers a paucity of practical, non-destructive indicators to inform process decisions. The present study sets out to compare sun drying (SD), room-temperature drying (RTD), convective tray drying (CTD; 40 - 60 °C), and microwave drying (MWD; 120 - 380 W), models thin-layer curves, and integrates effective moisture diffusivity ( $D_{\text{eff}}$ ) with quality metrics - antioxidant activity ( $IC_{50}$ , DPPH), FTIR ratios ( $R_Q$ ,  $R_1 - R_3$ ), and color attributes ( $\Delta E$ ,  $a^*$ ). Multi-parameter models have been shown to outperform simpler forms. The Midilli model provided the most precise global fit ( $R^2 > 0.95$ ;  $RMSE < 0.05$ ), while Jenna-Das performed well in specific convective subsets. As the temperature/power were increased,  $D_{\text{eff}}$  increased and reached a peak at an MWD of 380 W. This resulted in an approximate acceleration of  $\sim 225\times$  compared to the CTD 50 °C and a drying time of approximately  $\approx 4$  min. A clear trade-off emerged: CTD 40 °C exhibited a preserved appearance ( $\Delta E \approx 2.7$ ) but under-retained phenolics (weaker  $IC_{50}$ ), whereas CTD 60 °C and MWD 380 W produced higher  $\Delta E$  ( $> 8$ ) yet superior FTIR ratios ( $R_Q$ ,  $R_1 - R_3$ ) and stronger antioxidant activity; mid-power MWD (120 - 250 W) was detrimental. The colorimetric - spectroscopic linkages were found to be quantitative, with  $\Delta E$ - $IC_{50}$  exhibiting a weak-moderate relationship, and  $a^*$  demonstrating a strong colleration with  $R_Q$  ( $R^2 \approx 0.73$ ). Chemometrics (PLSR with VIP) identified  $R_Q/R_3$  as dominant predictors ( $R^2_{\text{LOOCV}} \approx 0.33$ ), thereby converting FTIR from descriptive readout to an actionable inline/at-line QC. Collectively, these results establish a predictive quality-control framework - using  $\Delta E$  and  $a^*$  with  $R_Q$  - for efficient selection, development, and implementation of tropical herbal drying technologies, and provide actionable set-points (optimal: MWD 380 W; convective alternative: CTD 60 °C) that balance speed and bioactive retention.

**Keywords:** Soursop leaves, Effective moisture diffusivity,  $IC_{50}$ , Thin-layer drying, FTIR ratio

## Introduction

The utilization of tropical herbal leaves in functional foods and phytopharmaceuticals has

increased in response to the demand for natural and sustainable products. While the drying process has been

demonstrated to extend shelf life, this stage can concomitantly result in a decline in phenolics and antioxidant capacity due to pigment conversion, oxidation, and structural degradation [1-6]. It has been demonstrated that microwave and freeze-drying techniques result in superior preservation of bioactivity when compared to conventional ovens. However, sun drying remains a viable option in tropical settings despite the high variability observed [7-12]. Consequently, there is a practical need for efficient drying strategies and non-destructive, predictive indicators that bridge laboratory analysis and industrial decision-making.

*Annona muricata* (soursop) leaves have been found to be rich in phenolics (e.g., gallic acid, catechin, quercetin, rutin) and flavonoids, supporting robust antioxidant activity and reported cytotoxic/anti-inflammatory effects that motivate their use in herbal teas and extracts [13-16]. However, post-harvest processing is fragmented: convective, solar, microwave, and other modalities have yielded variable outcomes for phenolics, antioxidant capacity, and color [17-19]. Consequently, a concise, integrative approach is imperative to balance process efficiency, speed, and bioactivity retention.

The quality of tropical herbal materials is commonly assessed via colorimetry ( $\Delta E$ ,  $a^*$ ) as markers of pigment/phenolic degradation, Fourier-transform infrared spectroscopy (FTIR) to track functional-group shifts associated with oxidation, and  $IC_{50}$  (DPPH) as a global indicator of radical-scavenging capacity [5,20-23]. Prior studies have frequently focused on a single indicator or a single drying method, thereby limiting comparative insight and rapid translation [24-27]. Emerging literature suggest that integrating color-FTIR-bioactivity may facilitate rapid, non-destructive prediction of herbal quality, particularly when combined with chemometrics [28,29,35,36]. However, such systematic integration has rarely been applied to *A. muricata* leaves. Three concise gaps emerge from this analysis: (i) limited cross-method evaluation of drying; (ii) insufficient integration of colorimetry and FTIR with bioactivity; and (iii) lack of rapid parameters suitable for real-time quality control.

In order to address these gaps, the present study introduces a multiscale integration of drying kinetics and modelling (sun/room drying; convective 40 - 60 °C;

microwave 120 - 380 W) using established thin-layer models (Midilli, Page, Henderson-Pabis), with effective moisture diffusivity ( $D_{eff}$ ) as a process-efficiency descriptor [30-34]. This study proposes a novel approach to predicting  $IC_{50}$  trends, offering a rapid and non-destructive surrogate for decision-making process. This study utilizes a combination of simple colorimetric indicators ( $\Delta E$ ,  $a^*$ ) with informative FTIR ratios ( $R_Q$ ,  $R_1 - R_3$ ) to achieve this objective, aligning with green processing principles and scalable quality control [28,29,35-39].

The objective of this research is 2-fold: (i) to evaluate the effects of temperature, power, and drying method on drying kinetics (thin-layer models,  $D_{eff}$ ) and (ii) to assess product quality ( $IC_{50}$ , FTIR ratios, and color). Additionally, the study aims to quantify the structure–function relationships linking colorimetric parameters ( $\Delta E$ ,  $a^*$ ) and FTIR indices ( $R_Q$ ,  $R_1 - R_3$ ) with antioxidant capacity ( $IC_{50}$ ). The results of this study are expected to (iii) enable a predictive quality control framework in which  $\Delta E$  and  $a^*$ , in combination with  $R_Q$ , serve as rapid and non-destructive indicators suitable for real-time process monitoring, and (iv) to provide actionable drying guidelines-comparing convective and microwave approaches - that balance process efficiency and phenolic retention for tropical herbal processing.

## Materials and methods

### Materials

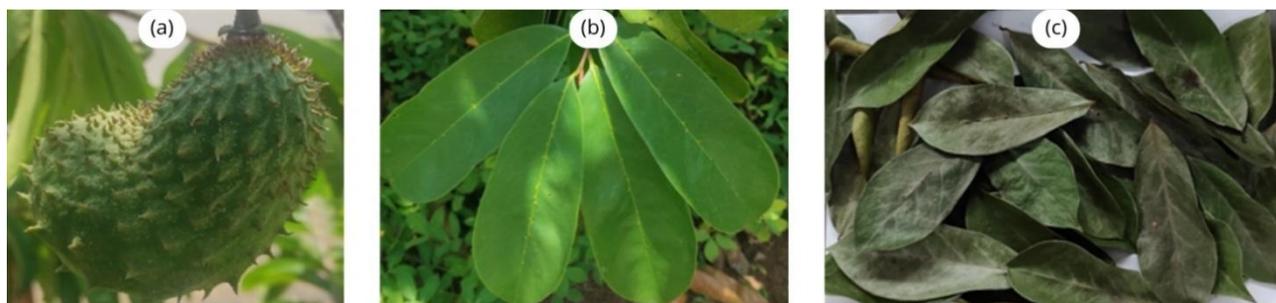
#### *Plant materials*

Fresh soursop leaves (*Annona muricata*; **Figure 1**) were obtained from the cultivation garden of Diponegoro University, Tembalang, Semarang, Indonesia. The identification process was conducted in the Process Laboratory, and voucher specimens were stored for the purpose of taxonomic authentication. Prior to the commencement of the treatment, the leaf stalks were meticulously separated, the leaves were thoroughly washed and then stored at a temperature of 4 °C for 24 h. The samples were weighed (0.7 - 1.6 g on average) and their thickness was measured by means of a micrometer screw gauge (Mitutoyo, Japan; accuracy 0.1 mm). In each drying treatment, 30 g of fresh leaves were utilized (based on wet weight) with 3 replicates ( $n = 3$ ).

### Chemicals

Analytical-grade solvents (ethanol, methanol, n-hexane) were obtained from Merck (Germany). The standards of gallic acid, quercetin, and DPPH (2,2-

diphenyl-1-picrylhydrazil) were obtained from Sigma-Aldrich (USA) and potassium bromide (KBr,  $\geq 99\%$ ) was utilized for the preparation of FTIR pellets.



**Figure 1** *Annona muricata*: (a) fruits, (b) fresh leaves, and (c) dry leaves.

### Drying process

The drying treatments (**Figure 2**) comprised sun drying (SD), room temperature drying (RTD), convective tray drying (CTD), and microwave drying (MWD).

- Sun drying (SD). It was conducted in an outdoor environment between 9:00 a.m. and 3:00 p.m. A total of 30 g of the sample was placed into a thin cloth bag (30×23 cm<sup>2</sup>) and placed on a stainless-steel tray (40×30 cm<sup>2</sup>).
- Room temperature drying (RTD): It was conducted at room temperature between 27.5 and 31.1 °C with a relative humidity of 57.4% - 68.5%, in an indoor environment. The experiment was conducted

from 08:00 a.m. to 16:00 p.m., and the temperature and humidity levels were monitored at regular intervals. The experiment was conducted in a way ascertaining protection from direct sunlight.

- Convective tray drying (CTD). It utilized a Sharp EO-35ST oven (Tokyo, Japan; 1000 W with maximum temperature at 250 °C). The oven was operated at temperatures of 40, 50, and 60 °C, with 75 mm between trays.
- Microwave drying (MWD). It was carried out using a Samsung MS20A3010AL microwave (Seoul, Korea; maximum 700 W) operated at 120, 250, and 380 W. A total of 30 g of the sample was spread evenly on a glass tray (205×205 mm<sup>2</sup>).



**Figure 2** Drying methods utilized: (a) SD, (b) RTD, (c) CTD, and (d) MWD.

During the drying process, the samples were weighed at regular intervals using electronic scales (D2B, PT Arta Joil Tappa, China; accuracy (0.01 g) until a constant weight was achieved (3 consecutive weighing with a variation of <0.1%). Time stamps were synchronized for subsequent MR-time analysis.

### The modelling of the drying kinetics

#### The moisture ratio (MR)

The drying characteristics of the samples were evaluated using the moisture ratio (MR; Eq. (1)):

$$MR = \frac{X_t - X_e}{X_0 - X_e} \quad (1)$$

with  $X_t$  = moisture at time  $t$ ,  $X_0$  = initial moisture and  $X_e$  = equilibrium moisture [40].

**Drying rate**

The instantaneous rate was computed as Eq. (2):

$$\text{Drying rate} = \frac{X_{t+\Delta t} - X_t}{\Delta t} \tag{2}$$

**Table 1** Thin-layer drying model.

| Model               | Equation                                |
|---------------------|-----------------------------------------|
| Page                | $MR = \exp(-kt^n)$                      |
| Henderson and Pabis | $MR = a \exp(-kt)$                      |
| Logarithmic         | $MR = a \exp(-kt) + b$                  |
| Midilli             | $MR = a \exp(-kt^n) + bt$               |
| 2-term exponential  | $MR = a \exp(-kt) + (1 - a) \exp(-kat)$ |
| Diffusion approach  | $MR = a \exp(-kt) + (1 - a) \exp(-kbt)$ |
| Jenna-Das           | $MR = a \exp(-kt) + b\sqrt{t} + c$      |

**Effective moisture diffusivity ( $D_{eff}$ )**

The falling-rate period was modeled using Fick’s Second Law for flat geometry (Eq. (3)):

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[ -\frac{(2n+1)^2 \pi^2 D_{eff} t}{4L^2} \right] \tag{3}$$

where  $L$  refers to the average thickness of leaves and  $t$  denotes time. In practice, the first-term approximation was applied where appropriate to obtain stable  $D_{eff}$  estimates, while full-series fitting was checked for sensitivity.

**Model adequacy and validation**

The suitability of the model was evaluated by means of the coefficient of determination ( $R^2$ ), root mean square error (RMSE), sum of squares error (SSE), and total sum of squares (TSS), with adequacy criteria of  $R^2 > 0.95$  and  $RMSE < 0.05$  [44,45].

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[ -\frac{(2n+1)^2 \pi^2 D_{eff} t}{4L^2} \right] \tag{4}$$

$$SSE = \sum_{i=1}^n (MR_{exp,i} - MR_{pre,i})^2 \tag{5}$$

$$TSS = \sum_{i=1}^n (MR_{exp,i} - \overline{MR_{exp,i}})^2 \tag{6}$$

**Thin-layer drying models**

This study involved the evaluation of seven models: Page, Henderson-Pabis, Logarithmic, Midilli, 2-term exponential, Diffusion approach, dan Jenna-Das [41-43]. The detailed equations are presented in **Table 1** with constants ( $a, b, c, k, k_0, k_1, n$ ) estimated using non-linear regression.

Residuals were screened for systematic deviation (runs/curvature). Models failing adequacy criteria were not advanced to comparative discussion.

**Analysis of quality**

**Antioxidant activity (DPPH assay)**

DPPH (2,2-diphenyl-1-picrylhydrazyl) is a stable radical used to assess radical-scavenging capacity.  $IC_{50}$  denotes the extract concentration ( $\mu\text{g/mL}$ ) required to reduce DPPH absorbance by 50% - a smaller  $IC_{50}$  indicates stronger activity. With minor modifications to [46,47], leaf powder (1 g) was extracted in 20 mL 80% methanol using a digital sonicator (Elmasonic S30H, Germany) for 30 min, then filtered (Whatman No.1, 0.45  $\mu\text{m}$ ). An aliquot (2 mL extract) was mixed with 2 mL 0.1 mM DPPH, incubated 30 min in the dark, and absorbance was recorded at 517 nm (UV-Vis Shimadzu UV-1900, Japan). Inhibition (%) was computed by Eq. (7):

$$SSE = \sum_{i=1}^n (MR_{exp,i} - MR_{pre,i})^2 \tag{7}$$

$$TSS = \sum_{i=1}^n (MR_{exp,i} - \overline{MR_{exp,i}})^2 \tag{8}$$

where  $A_0$  = control absorbance and  $A_1$  = sample absorbance. The value of  $IC_{50}$  was determined from the

regression curves between the percentage of inhibition towards concentration.

### FTIR spectroscopy

The spectrum was obtained using a PerkinElmer UATR Spectrum Two (US) in the range of 4,000 - 400  $\text{cm}^{-1}$ , with a resolution of 4  $\text{cm}^{-1}$ , scanned 32 times. The sample (in dry powder or fresh leaf pieces) was then placed directly on the ATR crystal. The main bands included O-H stretching, aliphatic C-H, ester/ $\gamma$ -lactone C=O, aromatic C=C, phenolic/ester C-O, and glycosidic C-O-C. Ratio indices  $R_1 - R_3$  and the composite  $R_Q$  were computed from integrated band areas, modified from the Tepe method [48]. A consistent baseline and pathlength normalization were employed across runs.

### Color analysis

The color coordinates (L,  $a^*$ , and  $b^*$ ) were measured using a HunterLab ColorFlex EZ Colorimeter (USA) in accordance with CIE standards [49,50]. Derived indices such as  $\Delta E$ , C, and  $h^\circ$  were calculated for further analysis.

$$SSE = \sum_{i=1}^n (MR_{exp,i} - MR_{pre,i})^2 \quad (9)$$

$$TSS = \sum_{i=1}^n (MR_{exp,i} - \overline{MR_{exp,i}})^2 \quad (10)$$

$$C = \sqrt{a^{*2} + b^{*2}}, h^\circ = \arctan\left(\frac{b^*}{a^*}\right) \quad (11)$$

### Statistical analysis and software

All experiments were performed in triplicate ( $n = 3$ ) with randomized drying order to minimize bias, and the results were presented as the mean  $\pm$  standard deviation. Nonlinear regression on the drying curve was performed using Polymath 6.10 (USA), while  $IC_{50}$  values and color parameters were analyzed by means of

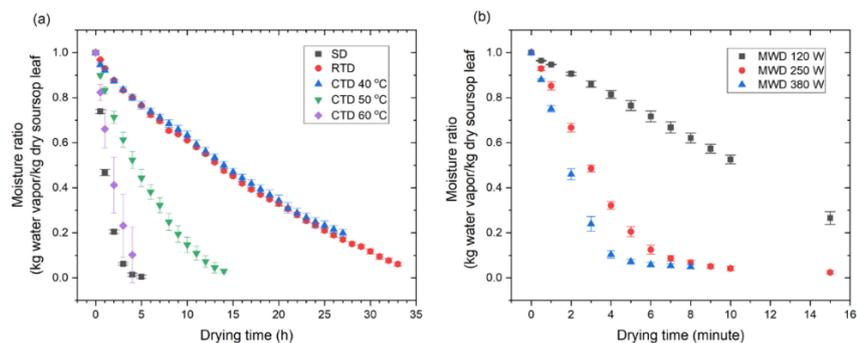
one-way ANOVA followed by Tukey's test at a significance level of  $p < 0.05$  (Minitab, Minitab LLC, USA). Treatment effects were also evaluated using Hedges'  $g$  effect size and linear and non-parametric relationships were tested with OLS regression and Spearman's correlation. To correlate rapid indicators with bioactivity, Partial Least Squares Regression (PLSR) (MATLAB R2024a, MathWorks, USA) was performed between FTIR indices ( $R_1 - R_3, R_Q$ ) and  $IC_{50}$ . Leave-one-out cross-validation (LOOCV) was used to estimate predictive ability (reporting  $R^2_{\{LOOCV\}}$  and RMSE), and Variable Importance in Projection (VIP) was computed to rank spectral predictors [51,52]. Chemometric adequacy was judged by stable VIP ranking ( $VIP \geq 1$  considered influential) and the absence of pathological leverage.

## Results and discussion

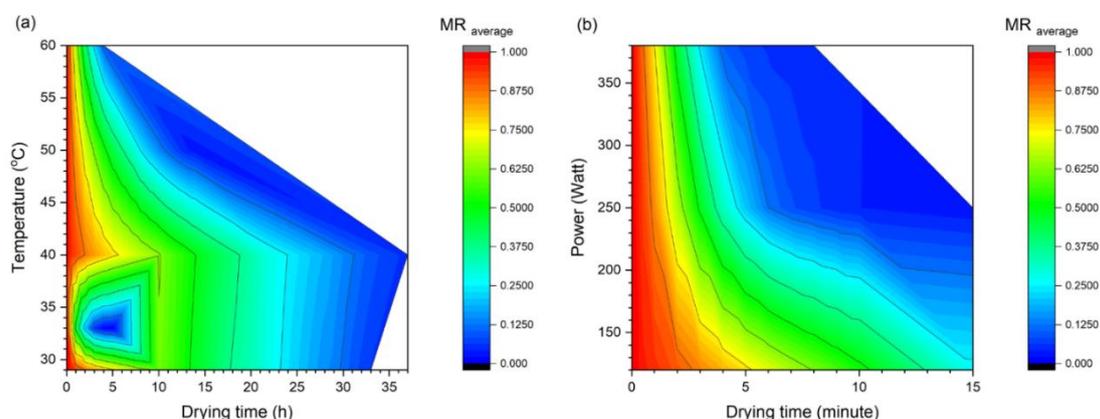
### Drying process

#### *Drying behavior and the factors of temperature/power*

The MR-time curve (**Figure 3**) demonstrated that an augmentation in convective temperature (40 - 60  $^\circ\text{C}$ ), microwave power (120 - 380 W), and sun-drying (SD) conditions significantly accelerated moisture release. RTD (29  $^\circ\text{C}$ ) and CTD 40  $^\circ\text{C}$  were found at the slowest, requiring  $\approx 30 - 33$  h to reach  $MR = 0.10$ , while CTD 60  $^\circ\text{C}$  reached it in  $\approx 4$  h ( $\approx 64\%$  faster than CTD 50  $^\circ\text{C}$ ). SD appeared faster ( $\approx 2.7$  h) yet highly variable due to sunlight fluctuation. MWD 380 W has been observed as the most significant intensification, reducing drying time to  $\approx 4$  min (97% - 99% shorter than CTD 50  $^\circ\text{C}$ ). The mean falling-rate slope ( $MR$  0.60 - 0.20) substantiated this gradient: 0.025  $MR \cdot h^{-1}$  (CTD 40  $^\circ\text{C}$ ) to 0.199  $MR \cdot h^{-1}$  (CTD 60  $^\circ\text{C}$ ), 0.313  $MR \cdot h^{-1}$  (SD), and 0.036 - 0.226  $MR \cdot \text{min}^{-1}$  (MWD 120 - 380 W) (**Figure 4**; **Table 2** summarizes statistics).



**Figure 3** The moisture ratio during the drying process of soursop leaf.



**Figure 4** The plot of mean moisture ratio: (a) SD, RTD, CTD, and (b) MWD.

It is evident that all drying conditions follow a dominant falling-rate phase without a constant rate stage, which is typical of leafy matrices with robust internal diffusion resistance [42,53,54]. The enhancement observed at elevated CTD temperatures is consistent with findings reported for *Moringa oleifera* and holy basil [55,56]. The unreliability of SD is analogous to that observed in *Plantago lanceolata* and *Alcea rosea* [57,58]. In contrast, MWD generates volumetric heating (dipole rotation/ionic conduction), creating steep internal vapor-pressure gradients and microstructural disruption, as evidenced by celery and coriander [59,60]. It is noteworthy that an acceleration of up to  $\sim 225\times$  (MWD 380 W vs. CTD 50 °C) is rarely reported for tropical medicinal leaves. However, it should be noted that each method has its limitations. The reliability of SD is compromised by the influence of

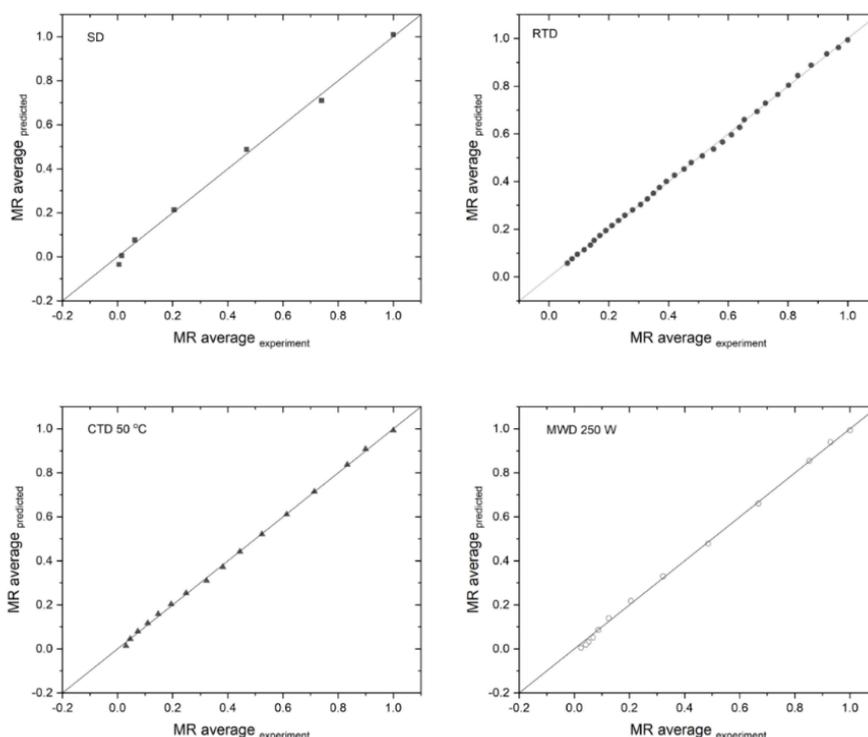
UV/wind resulting in low reproducibility. MWD is susceptible to local overheating when the load is inadequate. RTD/CTD necessitate extended exposure and are vulnerable to phenolic degradation.

#### ***Thin-layer modelling***

The multi-parameter nonlinear models (Midilli, Jenna-Das) demonstrated the highest level of accuracy ( $R^2 \geq 0.994$ ; RMSE 0.006 - 0.026; **Table 2**; **Figure 5**). Jenna-Das demonstrated proficiency at RTD and at CTD 50 °C, while Midilli exhibited superiority at 40 °C; both exhibited comparable performance at 60 °C. Across MWD 120 - 380 W, Midilli was consistently superior ( $R^2 > 0.995$ ). Drying constants increased with temperature/power (0.028 - 0.387 CTD; 0.020 - 0.311 MWD), while  $n$  decreased modestly (1.68 - 1.36).

**Table 2** Constants-coefficients for fitted model and its statistical parameter for drying of soursop leaves.

| Model                | Statistical Parameter | SD, 33 °C | RTD, 29 °C               | Temperature              |                         |                          | Power MWD                |                         |                         |
|----------------------|-----------------------|-----------|--------------------------|--------------------------|-------------------------|--------------------------|--------------------------|-------------------------|-------------------------|
|                      |                       |           |                          | CTD 40 °C                | CTD 50 °C               | CTD 60 °C                | 120 W                    | 250 W                   | 380 W                   |
| Page                 | k                     | 0.7252    | 0.0303                   | 0.0284                   | 0.1419                  | 0.3866                   | 0.0200                   | 0.1568                  | 0.3106                  |
|                      | n                     | 1.2223    | 1.2318                   | 1.2475                   | 1.1174                  | 1.1974                   | 1.5543                   | 1.4108                  | 1.3491                  |
|                      | R <sup>2</sup>        | 0.9081    | 0.9878                   | 0.9922                   | 0.9935                  | 0.9933                   | 0.9893                   | 0.9985                  | 0.9935                  |
|                      | RMSE                  | 0.1009    | 0.0322                   | 0.0265                   | 0.0250                  | 0.0295                   | 0.0429                   | 0.0133                  | 0.0260                  |
| Henderson and Pabis  | k                     | 0.8076    | 0.0614                   | 0.0613                   | 0.1785                  | 0.4762                   | 0.0815                   | 0.2907                  | 0.4523                  |
|                      | a                     | 1.0322    | 1.0333                   | 1.0370                   | 1.0099                  | 1.0324                   | 1.0708                   | 1.0697                  | 1.0619                  |
|                      | R <sup>2</sup>        | 0.9052    | 0.9767                   | 0.9799                   | 0.9904                  | 0.9847                   | 0.9703                   | 0.9875                  | 0.9868                  |
|                      | RMSE                  | 0.1024    | 0.0445                   | 0.0424                   | 0.0305                  | 0.0446                   | 0.0714                   | 0.0387                  | 0.0371                  |
| Logarithmic          | k                     | 0.5580    | 0.0288                   | 0.0353                   | 0.1239                  | 0.3570                   | 0.0690                   | 0.2792                  | 0.4666                  |
|                      | a                     | 1.1136    | 1.4875                   | 1.2934                   | 1.1620                  | 1.1429                   | 1.1569                   | 1.0861                  | 1.0539                  |
|                      | b                     | -0.1262   | -0.5140                  | -0.3100                  | -0.1833                 | -0.1347                  | -0.0980                  | -0.0195                 | 0.0115                  |
|                      | R <sup>2</sup>        | 0.9840    | 0.9991                   | 0.9989                   | 0.9991                  | 0.9962                   | 0.9860                   | 0.9877                  | 0.9874                  |
|                      | RMSE                  | 0.0421    | 8.54 × 10 <sup>-3</sup>  | 9.90 × 10 <sup>-3</sup>  | 9.51 × 10 <sup>-3</sup> | 0.0221                   | 0.0491                   | 0.0385                  | 0.0361                  |
| Midilli              | k                     | 0.7046    | 0.0511                   | 0.0314                   | 0.1577                  | 0.3789                   | 0.0135                   | 0.1511                  | 0.3103                  |
|                      | n                     | 1.0385    | 0.8637                   | 1.1290                   | 0.9206                  | 1.1018                   | 1.6776                   | 1.4358                  | 1.3647                  |
|                      | a                     | 1.0093    | 0.9940                   | 0.9660                   | 0.9928                  | 0.9980                   | 0.9612                   | 0.9934                  | 1.0017                  |
|                      | b                     | -0.0117   | -8.83 × 10 <sup>-3</sup> | -2.96 × 10 <sup>-3</sup> | -0.0109                 | -9.92 × 10 <sup>-3</sup> | -6.89 × 10 <sup>-4</sup> | 2.90 × 10 <sup>-4</sup> | 7.62 × 10 <sup>-4</sup> |
|                      | R <sup>2</sup>        | 0.9939    | 0.9995                   | 0.9991                   | 0.9993                  | 0.9965                   | 0.9979                   | 0.9988                  | 0.9952                  |
|                      | RMSE                  | 0.0260    | 6.56 × 10 <sup>-3</sup>  | 9.15 × 10 <sup>-3</sup>  | 8.11 × 10 <sup>-3</sup> | 0.0212                   | 0.0188                   | 0.0120                  | 0.0224                  |
| Two-term exponential | k                     | 1.0999    | 0.0584                   | 0.0814                   | 0.2232                  | 0.6227                   | 0.1256                   | 0.4201                  | 0.6491                  |
|                      | a                     | 1.7808    | 1.0000                   | 1.7631                   | 1.6088                  | 1.7233                   | 1.9691                   | 1.9411                  | 1.9176                  |
|                      | R <sup>2</sup>        | 0.9080    | 0.9747                   | 0.9919                   | 0.9943                  | 0.9929                   | 0.9861                   | 0.9986                  | 0.9943                  |
|                      | RMSE                  | 0.1009    | 0.0464                   | 0.0269                   | 0.0235                  | 0.0305                   | 0.0488                   | 0.0131                  | 0.0243                  |
| Diffusion approach   | k                     | 0.7672    | 0.0237                   | 0.0357                   | 0.1205                  | 0.3589                   | 0.0595                   | 0.2459                  | 0.4515                  |
|                      | a                     | -2.9799   | -2.9800                  | 2.1046                   | -2.6285                 | -4.9600                  | -6.3755                  | 0.9999                  | 1.0000                  |
|                      | b                     | 0.8263    | 0.8317                   | 0.4777                   | 1.2740                  | 0.9466                   | 0.8726                   | 0.9998                  | 0.9996                  |
|                      | R <sup>2</sup>        | 0.9080    | 0.9887                   | 0.9986                   | 0.9982                  | 0.9862                   | 0.9872                   | 0.9257                  | 0.9818                  |
|                      | RMSE                  | 0.1009    | 0.0311                   | 0.0112                   | 0.0133                  | 0.0424                   | 0.0468                   | 0.0945                  | 0.0435                  |
| Jenna-Das            | k                     | 0.7672    | 0.0237                   | 0.0357                   | 0.1205                  | 0.3589                   | 0.0595                   | 0.2459                  | 0.4515                  |
|                      | a                     | 0.8696    | 1.3176                   | 1.3543                   | 0.9498                  | 1.3192                   | 1.4092                   | 1.2501                  | 1.0795                  |
|                      | b                     | -0.1000   | -0.0301                  | 6.92 × 10 <sup>-3</sup>  | -0.0584                 | 0.0536                   | 0.0209                   | 0.0293                  | 6.30 × 10 <sup>-3</sup> |
|                      | c                     | 0.1567    | -0.3158                  | -0.3780                  | 0.0477                  | -0.3206                  | -0.3795                  | -0.2015                 | -0.0181                 |
|                      | R <sup>2</sup>        | 0.9940    | 0.9996                   | 0.9989                   | 0.9995                  | 0.9966                   | 0.9896                   | 0.9899                  | 0.9878                  |
|                      | RMSE                  | 0.0257    | 5.83 × 10 <sup>-3</sup>  | 9.79 × 10 <sup>-3</sup>  | 6.81 × 10 <sup>-3</sup> | 0.0211                   | 0.0423                   | 0.0348                  | 0.0355                  |



**Figure 5** Comparison of MR experiment and Midilli modelling in the drying process of soursop leaf.

These findings emphasized the Midilli model as a universal predictive tool suitable for scale-up, while the Jenna-Das model remains more appropriate for convective subsets, consistent with the behavior observed in *Cosmos caudatus* and *Vernonia amygdalina* [61,62]. However, as empirical constructs, these models do not explicitly account for morphology or porosity evolution during drying process. Consequently, cross-batch validation is imperative, with particular consideration for variations in leaf thickness and ambient relative humidity, is required to ensure the adequacy of the input range and maintain predictive

reliability.

**Effective moisture diffusivity ( $D_{eff}$ )**

As demonstrated in **Table 3**,  $D_{eff}$  exhibited a marked increase with both temperature and microwave power: From  $1.92 \times 10^{-15} \text{ m}^2/\text{s}$  (RTD) and  $2.34 \times 10^{-15} \text{ m}^2/\text{s}$  (CTD 40 °C) to  $1.26 \times 10^{-13} \text{ m}^2/\text{s}$  (CTD 60 °C); SD yielded  $2.14 \times 10^{-13} \text{ m}^2/\text{s}$ ; MWD 120 - 380 W reached  $2.07 \times 10^{-13}$  to  $3.40 \times 10^{-12} \text{ m}^2/\text{s}$  ( $\approx 8.1 - 225 \times$ CTD 50 °C). The increase is indicative of lower viscosity, higher vapor pressure, and micro-cracks formation under higher thermal/microwave energy.

**Table 3** Effective moisture diffusivity of soursop leaf.

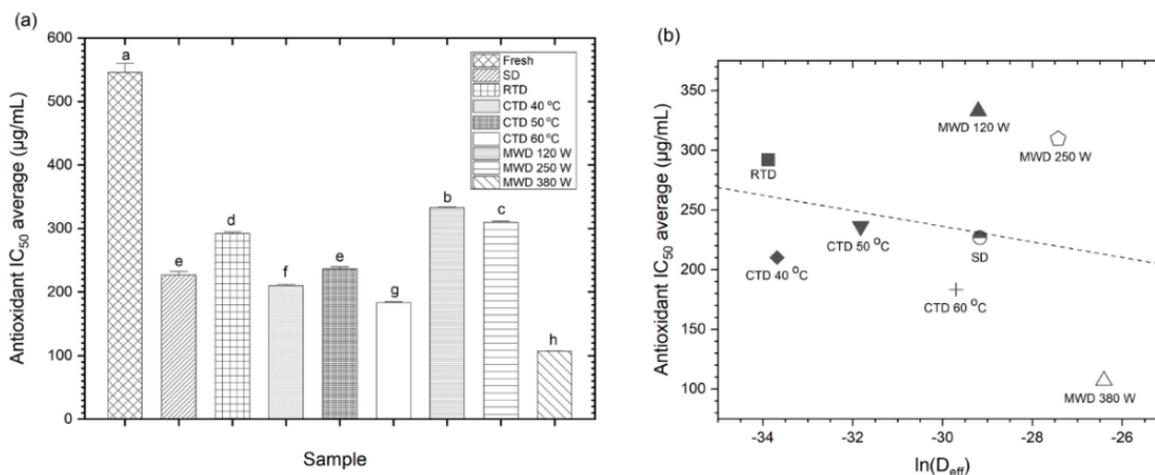
| Sample    | $D_{eff}, \text{m}^2/\text{s}$ | $R^2$ | Relative to CTD 50 °C (fold) |
|-----------|--------------------------------|-------|------------------------------|
| SD        | $2.142 \times 10^{-13}$        | 0.989 | 14.19                        |
| RTD       | $1.924 \times 10^{-15}$        | 0.951 | 0.13                         |
| CTD 40 °C | $2.343 \times 10^{-15}$        | 0.958 | 0.16                         |
| CTD 50 °C | $1.509 \times 10^{-14}$        | 0.959 | 1.00                         |
| CTD 60 °C | $1.263 \times 10^{-13}$        | 0.983 | 8.37                         |
| MWD 120 W | $2.067 \times 10^{-13}$        | 0.940 | 13.70                        |
| MWD 250 W | $1.225 \times 10^{-12}$        | 0.947 | 81.15                        |
| MWD 380 W | $3.400 \times 10^{-12}$        | 0.942 | 225.13                       |

At 380 W, the effective moisture diffusivity ( $D_{eff}$ ) exhibited a 2 to 3 orders of magnitude increase compared to the CTD, a finding consistent with observations reported for *C. nardus* and *C. roseus* [33,63]. Its strong correlation with the falling-rate slope and the time required to reach  $MR = 0.10$  highlights  $D_{eff}$  as a practical descriptor of drying efficiency. As demonstrated in relevant literature, elevated  $D_{eff}$  values under MWD conditions have been shown to be associated with enhanced phenolic retention and superior color preservation [64,65]. However, the accuracy of  $D_{eff}$  estimation is contingent upon the accuracy of geometry and thickness assumptions, and the reliability of the estimation is potentially compromised by the presence of uncontrolled external factors in the SD conditions.

**Antioxidant activity (the assay of DPPH,  $IC_{50}$ )**

$IC_{50}$  exhibited significant variation among treatments (Figure 6(a)), confirming a strong drying-method effect (ANOVA,  $F = 1,557.83$ ;  $p < 0.001$ ;  $\eta^2 = 0.999$ ; Table 4). Fresh leaves demonstrated the lowest

antioxidant activity ( $\approx 546 \mu\text{g/mL}$ ), whereas MWD 380 W exhibited the strongest potential ( $\approx 107 \mu\text{g/mL}$ ;  $\sim 5$ -fold improvement). The CTD 60 °C ( $\sim 183 \mu\text{g/mL}$ ) demonstrated superior performance in comparison to CTD 40 - 50 °C ( $\sim 210 - 236 \mu\text{g/mL}$ ), while RTD exhibited the weakest performance ( $\sim 292 \mu\text{g/mL}$ ). Tukey analysis categorized treatments into distinct groups, and Hedges'  $g$  revealed substantial contrasts, particularly between MWD 380 W and other methods (Table 5). A salient observation is that medium microwave power (120 - 250 W) yielded unanticipated  $IC_{50}$  values ( $\approx 310 - 333 \mu\text{g/mL}$ ), indicating that mid-level energy prolongs exposure and expedite oxidative degradation, resulting in a U-shaped response. Conversely, high microwave intensity (i.e.  $\geq 380 \text{ W}$ ) generates sufficient volumetric heating to reduce residence time and preserve bioactivity (Figure 6(a)). The  $IC_{50}$ - $\ln(D_{eff})$  relationship was weak and insignificant ( $R^2 = 0.06$ ;  $\rho = -0.14$ ; Figure 6(b); Table 6), indicating that kinetic intensification alone is inadequate to predict antioxidant preservation.



**Figure 6** Soursop leaf: (a) anti-radical activity value, and (b) correlation of  $IC_{50}$ - $\ln(D_{eff})$ .

**Table 4** ANOVA results regarding the effects of drying method on soursop leaf.

| Source of variation | SS           | df | MS           | F          | p-value    | F-crit     |
|---------------------|--------------|----|--------------|------------|------------|------------|
| Between Groups      | 368,170.4211 | 8  | 46,021.30263 | 1,557.8299 | 5.9164E-24 | 2.51015789 |
| Within Groups       | 531.7549217  | 18 | 29.54194009  |            |            |            |
| Total               | 368,702.176  | 26 |              |            |            |            |

**Table 5** Level of Hedges' effects  $g$  (CI 95%) for the contrast of main  $IC_{50}$  among treatments.

| Contrast               | Hedges' $g$ | 95% CI            | Interpretation               |
|------------------------|-------------|-------------------|------------------------------|
| MWD 380 W vs Fresh     | -35.09      | [-59.45, -10.74]  | Very large, significant      |
| MWD 380 W vs SD        | -23.28      | [-39.46, -7.10]   | Large, significant           |
| MWD 380 W vs RTD       | -85.42      | [-114.63, -26.21] | Very large, significant      |
| MWD 380 W vs CTD 40 °C | -55.51      | [-94.00, -17.02]  | Very large, significant      |
| MWD 380 W vs CTD 50 °C | -41.78      | [-70.76, -12.80]  | Very large, significant      |
| MWD 380 W vs CTD 60 °C | -48.58      | [-82.27, -14.89]  | Very large, significant      |
| MWD 380 W vs MWD 120 W | -198.36     | [-335.83, -60.90] | Extremely large, significant |
| MWD 380 W vs MWD 250 W | -97.35      | [-164.82, -29.88] | Extremely large, significant |
| CTD 60 °C vs Fresh     | -28.78      | [-48.77, -8.80]   | Very large, significant      |
| CTD 60 °C vs SD        | -8.12       | [-13.89, -2.35]   | Moderate, significant        |
| CTD 60 °C vs RTD       | -41.51      | [-70.31, -12.72]  | Very large, significant      |
| CTD 60 °C vs CTD 40 °C | -11.29      | [-19.21, -3.36]   | Large, significant           |
| CTD 60 °C vs CTD 50 °C | -15.47      | [-26.27, -4.67]   | Large, significant           |
| SD vs Fresh            | -23.60      | [-40.00, -7.20]   | Large, significant           |
| SD vs CTD 50 °C        | -1.60       | [-3.29, 0.10]     | Small, not significant       |

**Table 6** Correlation of  $IC_{50}$  to  $\ln(D_{eff})$  using OLS regression and Spearman correlation.

| Analysis             | Parameter               | Value  | Note                           |
|----------------------|-------------------------|--------|--------------------------------|
| OLS regression       | Intercept ( $\beta_0$ ) | 41.33  | $\pm 322.47$ (SE)              |
|                      | Slope ( $\beta_1$ )     | -6.50  | $\pm 10.65$ (SE)               |
|                      | $t(\beta_1)$            | -0.610 | df = 6                         |
|                      | p-value                 | 0.564  | Not significant                |
|                      | $R^2$                   | 0.058  | Weak                           |
|                      | $R^2_{adj}$             | -0.099 | Negative (small n or poor fit) |
| Spearman correlation | $\rho$                  | -0.142 | Weak negative                  |
|                      | $t$                     | -0.353 | df = 6                         |
|                      | p-value                 | 0.736  | Not significant                |

This nonlinear response has been documented in phenolic-rich matrices, where mid-level MWD induces oxidative stress, while higher microwave power enhances retention [66,67]. Similarly, moderate CTD temperatures (50 - 70 °C) often better preserve polyphenols compared to prolonged low-temperature drying or sun drying [5,68-70]. However, the DPPH assay only reflects overall antioxidant capacity and does not differentiate specific molecular contributors; thus, identifying individual compounds such as flavonoids or

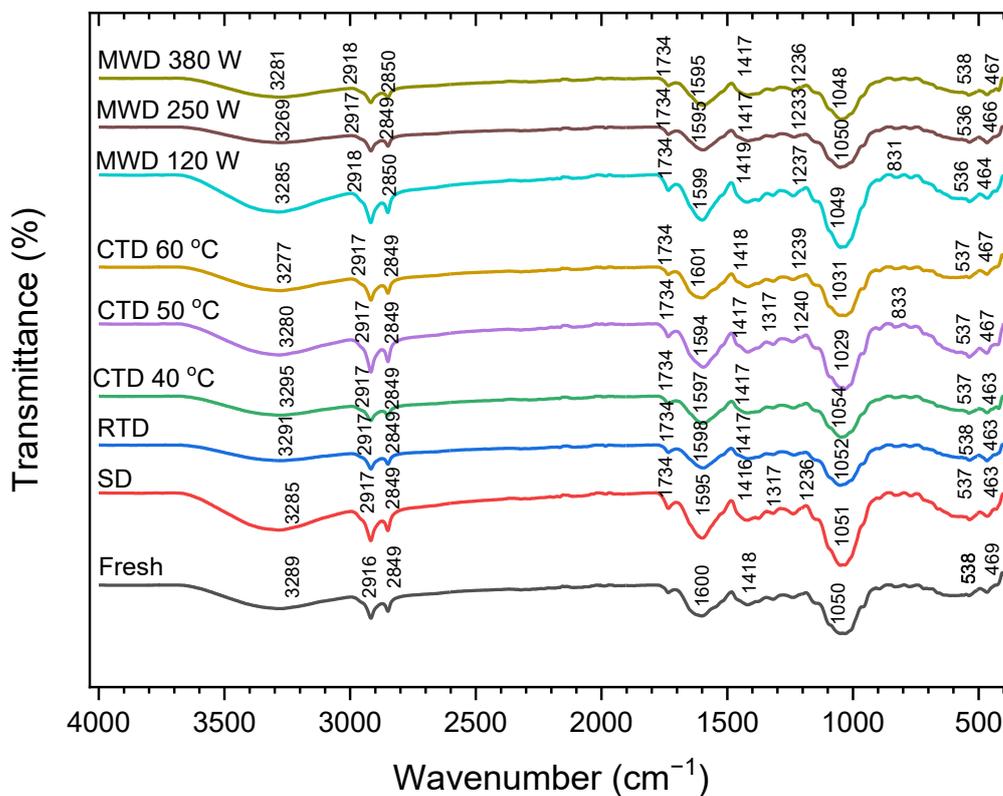
acetogenins requires further analysis using HPLC or LC-MS techniques.

#### FTIR analysis of phytochemical retention

FTIR spectra (**Figure 7**) displayed characteristic lignocellulosic-phenolic bands: O-H (3,280 - 3,300  $cm^{-1}$ ), aliphatic C-H (2,920/2,850  $cm^{-1}$ ), aromatic C=C (1,595 - 1,600  $cm^{-1}$ ), C-O (1,050 - 1,030  $cm^{-1}$ ), and glycosidic C-O-C (1,236 - 1,240  $cm^{-1}$ ). In comparison with fresh leaves (O-H at 3,289  $cm^{-1}$ ), dried samples exhibited a red shift (3,269 - 3,295  $cm^{-1}$ ) and diminished

O–H/C–O intensities, indicating weakened H-bonding and partial phenolic loss. A distinct C=O band ( $\sim 1,734$   $\text{cm}^{-1}$ ) emerged exclusively after drying (SD–MWD 380

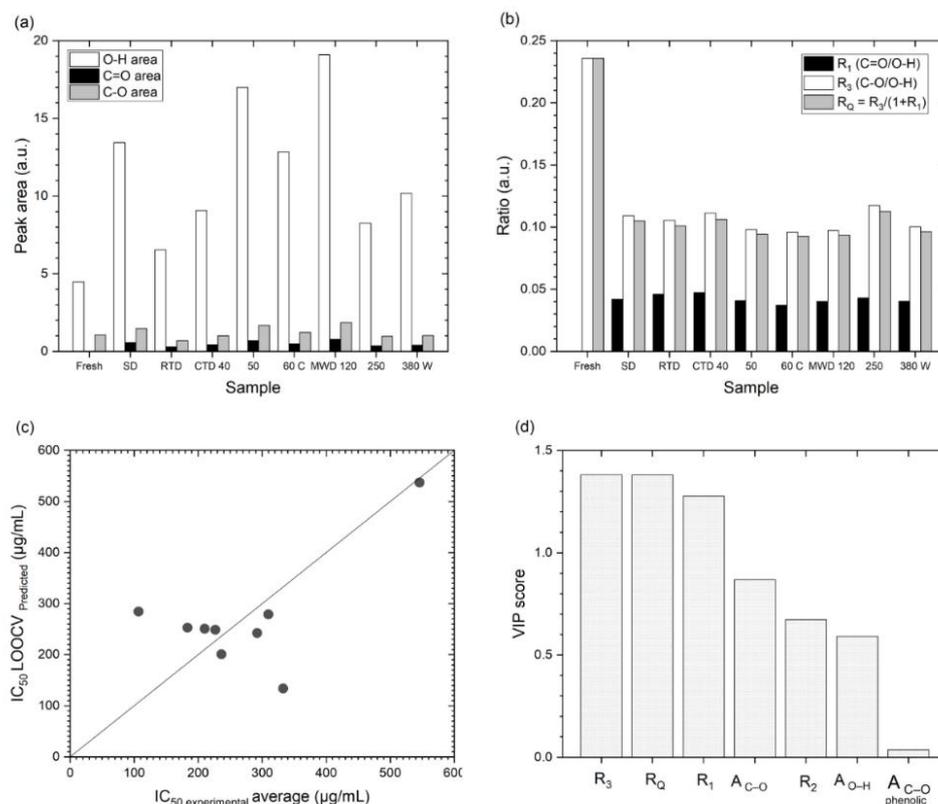
W), thereby indicating oxidation/esterification. At 380 W, the weakening at  $1,236 - 1,240$   $\text{cm}^{-1}$  signified partial polysaccharide depolymerization.



**Figure 7** FTIR spectra of soursop leaf.

To capture these shifts quantitatively, ratios  $R_1$ – $R_4$  and a composite  $R_Q$  were proposed (**Figures 8(a)** and **8(b)**).  $R_Q$  demonstrated the strongest consistency with  $IC_{50}$  and outperformed single-band indicators, thereby transforming FTIR analysis from a purely descriptive tool into an in-line quality control (QC) approach. Analogous correlations between FTIR indices and antioxidant capacity have been reported for *Centella asiatica*, *Moringa oleifera*, black carrot, dates, and apples [48,71-76]. Integration with chemometric modeling further reinforced this framework: PLS

regression analysis between FTIR predictors and  $IC_{50}$  yielded  $R^2_{\{LOOCV\}} = 0.325$  and  $RMSE = 95.9$   $\mu\text{g/mL}$ . VIP scores identified  $R_3$  and  $R_Q$  as dominant predictors (**Figures 8(c) - 8(d)**), thereby confirming that the phenolic-carbonyl equilibrium plays a pivotal role in antioxidant retention [77-82]. However, FTIR provides only spectral proxies; LC-MS or HPLC remains necessary to identify specific phenolic subclasses, and PLSR is sensitive to preprocessing steps such as baseline correction and normalization, warranting multi-batch replication for validation.



**Figure 8** Quantitative FTIR analysis: (a) peak areas of O–H, C=O, and C–O; (b) ratio of  $R_1$  to  $R_0$ ; (c) regression of PLS FTIR vs. antioxidant activity; and (d) FTIR ratio vs. VIP score.

#### Color attributes ( $L$ , $a^*$ , $b^*$ , $\Delta E$ , $C$ and $h^\circ$ )

Fresh leaves exhibited low brightness and strong greenness (Table 7 for details;  $L = 24.05 \pm 0.04$ ;  $a^* = -6.72 \pm 0.04$ ;  $b^* = 7.01 \pm 0.03$ ). Drying resulted in significant alterations across coordinates:  $L$  increased (highest at CTD 60 °C and MWD 250 W),  $a^*$  shifted

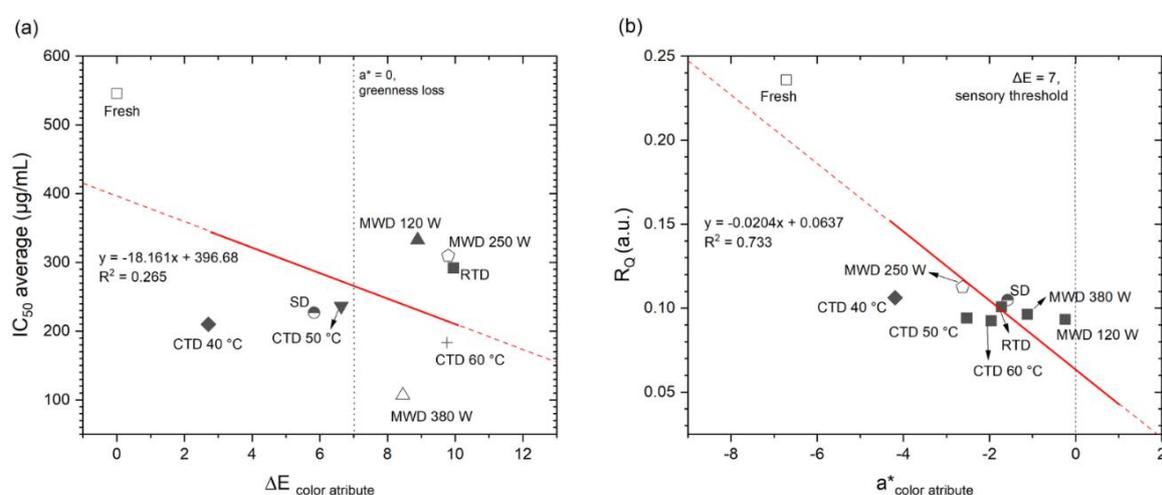
towards 0 (smallest loss at CTD 40 °C), and  $b^*$  increased (maximum at CTD 60 °C).  $\Delta E$  was smallest for CTD 40 °C ( $\approx 2.71$ ; below the  $\sim 3$  perceptual threshold) and highest for RTD; MWD was generally high.

**Table 7** Changes in the color attributes of soursop leaves using various drying methods.

| Sample    | $L$                | $a^*$              | $b^*$             | $\Delta E$        | $C$ (Chroma)      | $h^\circ$ (hue angle) |
|-----------|--------------------|--------------------|-------------------|-------------------|-------------------|-----------------------|
| Fresh     | $24.05 \pm 0.04^d$ | $-6.72 \pm 0.04^h$ | $7.01 \pm 0.03^d$ | -                 | $9.71 \pm 0.05^a$ | $-46.22 \pm 0.06^a$   |
| SD        | $25.84 \pm 0.02^c$ | $-1.58 \pm 0.03^c$ | $4.93 \pm 0.02^g$ | $5.83 \pm 0.07^f$ | $5.17 \pm 0.04^g$ | $-72.22 \pm 0.18^c$   |
| RTD       | $15.61 \pm 0.02^f$ | $-1.72 \pm 0.04^d$ | $5.34 \pm 0.02^e$ | $9.95 \pm 0.04^a$ | $5.61 \pm 0.03^f$ | $-72.13 \pm 0.28^c$   |
| CTD 40 °C | $24.08 \pm 0.59^d$ | $-4.19 \pm 0.02^g$ | $7.86 \pm 0.05^b$ | $2.71 \pm 0.04^g$ | $8.91 \pm 0.05^c$ | $-61.91 \pm 0.09^b$   |
| CTD 50 °C | $19.35 \pm 0.01^e$ | $-2.53 \pm 0.06^f$ | $4.94 \pm 0.05^g$ | $6.63 \pm 0.05^e$ | $5.56 \pm 0.07^f$ | $-62.87 \pm 0.27^c$   |
| CTD 60 °C | $32.37 \pm 0.03^a$ | $-1.96 \pm 0.02^e$ | $8.87 \pm 0.02^a$ | $9.76 \pm 0.03^b$ | $9.08 \pm 0.02^b$ | $-77.52 \pm 0.14^f$   |
| MWD 120 W | $29.82 \pm 0.03^b$ | $-0.24 \pm 0.03^a$ | $5.05 \pm 0.04^f$ | $8.89 \pm 0.02^c$ | $5.06 \pm 0.04^g$ | $-87.24 \pm 0.34^h$   |
| MWD 250 W | $32.94 \pm 0.05^a$ | $-2.62 \pm 0.04^f$ | $7.50 \pm 0.02^c$ | $9.79 \pm 0.09^b$ | $7.94 \pm 0.03^d$ | $-70.71 \pm 0.23^d$   |
| MWD 380 W | $30.39 \pm 0.03^b$ | $-1.12 \pm 0.06^b$ | $6.95 \pm 0.04^d$ | $8.45 \pm 0.02^d$ | $7.04 \pm 0.03^e$ | $-80.82 \pm 0.53^g$   |

Note: Fresh samples were excluded from ANOVA  $\Delta E$  due to 0 variance. Values are expressed as mean  $\pm$  SD with  $n = 3$ ;  $p < 0.05$ , Tukey's test.

The  $\Delta E$ - $IC_{50}$  plot demonstrated a weak-to-moderate correlation ( $R^2 = 0.265$ ; **Figure 9**), indicating that visual color changes alone cannot fully explain antioxidant variation [83-85]. In contrast, the  $a^*$  parameter exhibited a robust correlation with the FTIR  $R_Q$  ratio ( $R^2 = 0.733$ ), where the loss of greenness ( $a^*$  approaching 0) corresponded to a decrease in C–O/O–H ( $R_Q$ ) and an increase in C=O ( $\sim 1,734\text{ cm}^{-1}$ ), reflecting phenolic depletion and carbonyl accumulation. These findings are consistent with those reported for *Strobilanthes crispus* and *Allium ursinum* [5,7,86-88].



**Figure 9** Correlation of color and function in soursop leaf.

### Synthesis and practical implications

Overall, MWD 380 W represents an optimal process-product compromise:  $\approx 4$  min drying, low  $IC_{50}$ , and FTIR signatures consistent with phenolic retention; CTD 60 °C is a viable convective compromise.  $D_{\text{eff}}$  emerges as a kinetic surrogate, whereas bioactive quality should be predicted via FTIR ratios ( $R_Q/R_3$ ) and, when required,  $a^*$  as a rapid indicator. The integration of FTIR analysis with PLSR and VIP framework demonstrated potential for in-line quality control in tropical herbal processing.

### Conclusions

This present study systematically integrated drying kinetics, spectroscopic indicators, and antioxidant response to establish a predictive framework for optimizing *Annona muricata* leaf drying. The findings can be summarized as follows: The first point to consider is that of kinetics and quality (scientific).

In practice, CTD 40 °C was found to be capable of maintaining visual appearance ( $\Delta E \approx 2.7$ ) but did not optimize phenolic preservation. Conversely, CTD 60 °C and MWD 380 W produced higher  $\Delta E$  values (a noticeable sensory shift [84,89,90]) yet achieved superior phenolic retention and  $IC_{50}$  performance. However, it should be noted that color is determined by illumination conditions and instrument calibration, and it is unable to differentiate chlorophyll from phenolic degradation pathways. Therefore, cross-device color-space standardization is recommended.

The drying methods employed had a significant impact on both the kinetics and the quality of the product; the Midilli model yielded the optimal thin-layer fit ( $R^2 > 0.95$ ;  $\text{RMSE} < 0.05$ ), while the Jenna-Das model demonstrated efficacy under specific convective conditions. The effective moisture diffusivity ( $D_{\text{eff}}$ ) exhibited an increase in accordance with both temperature and power, reaching a maximum at MWD 380 W, which resulted in an acceleration of up to  $\sim 225\times$  in comparison to CTD 50 °C. A trade-off was observed, where CTD 40 °C maintained appearance ( $\Delta E \approx 2.7$ ) but reduced phenolic retention, whereas CTD 60 °C and MWD 380 W yielded higher  $\Delta E$  ( $> 8$ ) yet superior FTIR ratios ( $R_Q$ ,  $R_1 - R_3$ ) and  $IC_{50}$  values; mid-power MWD (120 - 250 W) was detrimental. (ii) Structure-function relationships (scientific): The  $\Delta E$ - $IC_{50}$  correlation was weak to moderate, but  $a^*$  exhibited a strong correlation with  $R_Q$  ( $R^2 \approx 0.73$ ). Furthermore, PLSR between FTIR indices and  $IC_{50}$  yielded  $R^2_{\{\text{LOCV}\}} \approx 0.33$  with VIP

emphasizing  $R_3$  and  $R_Q$ . This validates FTIR ratios as non-destructive predictors of antioxidant preservation. In the third instance, the practical implementation of predictive quality control was demonstrated. The combination of the parameters  $\Delta E$  and  $a^*$  in conjunction with  $R_Q$ , functioned as expeditious indicators for real-time guidance. The integration of FTIR analysis with Partial Least Squares Regression (PLSR) and Variable Importance in Projection (VIP) transformed FTIR from a descriptive technique into an actionable inline or at-line quality control tool, thereby minimizing reliance on laborious assays and supporting green processing. The provision of actionable guidance on the practical implementation of drying processes is of paramount importance. MWD 380 W represented the optimal process-product compromise ( $\approx 4$  min, low  $IC_{50}$ , favorable FTIR ratios), while CTD 60 °C was a reliable convective alternative, SD was rapid but inconsistent, and mid-power MWD should be avoided. In practice,  $D_{eff}$  can function as a kinetic gate, with  $R_Q$  and  $a^*$  providing rapid bioactivity verification. It is recommended that future research endeavours extend the validation process to pilot and industrial scales. Additionally, there is a need to explore hybrid drying configurations, and integrate advanced chemometrics for real-time control.

#### Acknowledgements

The authors acknowledged the financial support from Universitas Diponegoro, Indonesia, 2025.

#### Declaration of Generative AI in Scientific Writing

The authors declare we have not used Artificial Intelligence (AI) tools in the creation of this.

#### CRedit Author Statement

**Dessy Agustina Sari:** Methodology, Investigation, Data curation, Writing - original draft, Visualization, Writing - review & editing; **Moh Djaeni:** Conceptualization, Supervision, Validation, Writing-review & editing; **Devi Yuni Susanti:** Formal analysis; **Joko Nugroho Wahyu Karyadi:** Visualization; **Olly Sanny Hutabarat:** Software; **Setia Budi Sasongko:** Software, Visualization; **Aji Prasetyaningrum:** Formal analysis; **Ching Lik Hii:** Methodology, Writing - review & Editing.

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