Drying and Degradation Kinetics of the Physicochemical Characteristics of

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DRYING AND DEGRADATION KINETICS OF THE PHYSICOCHEMICAL CHARACTERISTICS OF PARIJOTO FRUIT (MEDINILLA SPECIOSA) WITH CALCIUM CHLORIDE PRE-TREATMENT

ABSTRACT

Parijoto (*Medinilla speciosa*) is an Indonesian local plant with high levels of bioactive compounds crucial in improving overall health. However, these bioactive compounds are susceptible to high temperatures from prolonged heating processes and environmental factors such as oxygen, light, and pH. Therefore, a significant decline in the parijoto fruit quality may occur during drying, which prompts a need for a solution to prevent damage to the bioactive compounds in the fruits. As a food additive, calcium chloride (CaCl2) can help maintain cell wall strength and prevent damage from enzymatic, mechanical, and microbial activities in food products. The study aimed to investigate the impact of soaking with $CaCl_2$ (10 minutes) and drying temperatures (60, 70, and 80ºC) for 8 hours on physicochemical characteristics such as antioxidant activity, total anthocyanin content, and colour. The moisture ratio, colour intensity, antioxidant activity and total anthocyanin content at hourly intervals during drying were measured. The results indicated that soaking in CaCl2 can lead to osmotic dehydration, accelerating the drying rates and preserving the anthocyanin content. The kinetics of the degradation of anthocyanins and antioxidant activity were established, as well as the drying kinetic model for parijoto fruits. The Page model was found to be the most relevant and suitable drying kinetics model based on the drying design in this study compared to the other two models.

Keywords: Parijoto fruit; Degradation kinetic, Drying kinetic, Calcium chloride

ABSTRAK

Parijoto (*Medinilla speciosa*) adalah tanaman lokal Indonesia yang mengandung senyawa bioaktif tinggi yang penting untuk meningkatkan kesehatan secara keseluruhan. Namun, senyawa bioaktif ini rentan terhadap suhu tinggi dari proses pemanasan yang panjang dan faktor lingkungan seperti oksigen, cahaya, dan pH. Oleh karena itu, penurunan kualitas buah parijoto yang signifikan dapat terjadi selama pengeringan, sehingga diperlukan solusi untuk mencegah kerusakan pada senyawa bioaktif di buah tersebut. Sebagai bahan tambahan makanan, kalsium klorida ($CaCl_2$) dapat membantu menjaga kekuatan dinding sel dan mencegah kerusakan akibat aktivitas enzimatik dan mikroba pada produk pangan. Penelitian ini bertujuan untuk menginvestigasi dampak perendaman dengan $CaCl_2$ (10 menit) dan suhu pengeringan (60, 70, dan 80ºC) selama 8 jam terhadap karakteristik fisiko-kimia seperti aktivitas antioksidan, total kandungan antosianin, dan warna. Perbandingan rasio kadar air, intensitas warna, aktivitas antioksidan, dan kandungan total antosianin diuji setiap jam selama proses pengeringan. Hasil penelitian menunjukkan bahwa perendaman dalam $CaCl_2$ dapat menyebabkan dehidrasi osmotic sehingga mempercepat laju pengeringan, dan menjaga kandungan antosianin. Pada studi ini, dilakukan pula pemodelan kinetika degradasi antosianin dan aktivitas antioksidan, serta model kinetika pengeringan untuk buah parijoto. Model *Page* terbukti menjadi model kinetika pengeringan yang paling relevan dan sesuai berdasarkan desain pengeringan dalam studi ini.

Kata kunci: Buah parijoto; Kinetika degradasi, Kinetika pengeringan, Kalsium klorida

1. INTRODUCTION

Parijoto (*Medinilla speciosa*) is a local Indonesian plant that grows, often uncultivated, in Kudus, Central Java. Parijoto is currently often cultivated as a decorative plant. However, the fruit of parijoto contains a high amount of bioactive compounds such as ascorbic acid, carotenoids, flavonoids, vitamin E, flavonol glycoside and phenolic compounds which may act as antioxidants (Angriani, 2019). Antioxidant compounds play an essential role in the health of the body, as they can protect the body from oxidative damage, inhibit oxidative stress, reduce inflammation, and boost the immune system (Haeraniet al., 2018).

Previous research has shown that anthocyanin compound in parijoto fruit can be used as a natural blue colourant (Priska et al., 2018). Anthocyanin can also act as antioxidant, anticancer, antidiabetics, and antiinflammation (Basri, 2021; Tan et al., 2021). However, the bioactive compounds in the parijoto fruit are very vulnerable to damage, especially the anthocyanin compound and the antioxidant components such as flavonoids and phenolics (Wachidah, 2013). The damage to such compounds can be caused by high-temperature processes and environmental conditions such as oxygen, light, and pH (Feng et al., 2015). Drying, on the other hand, is a standard preservation method because it can increase the storage life and facilitate the distribution, supply, and ease-of-use. Therefore, it is necessary to prevent the damage of bioactive compounds due to the drying temperature of the parijoto fruit, e.g by pre-treatments.

Using organic acid solutions (citric acid, acetic acid) and salt solutions (Na^{+}, Ca^{2+}) with specific concentrations as a pre-drying treatment can retain bioactive compounds in food materials. Calcium chloride ($CaCl_2$) is a salt classified as a food additive. According to a study by Guo et al. (2023), the lifespan of lychee fruit increased because $CaCl_2$ increased the strength of the cell wall and prevented the activity of polyphenol oxidase (PPO) enzymes and microbes. Looking at the potential of parijoto fruit as a novel health-promoting food ingredient, this study aims to firstly examine the effect of $CaCl_2$ and temperature in the drying process of parijoto fruit. Secondly, this study also aims to establish the drying and degradation kinetics, which will be useful in developing parijoto fruit products that are shelf-stable with optimum bioactive compound activities.

2. MATERIALS AND METHOD

2.1. Materials

Fresh parijoto fruits were obtained from Kudus, Central Java. Other materials used in this study are $CaCl_2$, KCl, CH_3COONa, 2-diphenyl-1-picrylhydrazyl (DPPH), and metanol 99.98%. All the chemicals used are of analytical grade unless specified.

2.2. Methods

2.2.1. Parijoto fruit preparation and pre-treatment

Parijoto fruits were separated from the branch, sorted and then washed under a running tap water. Half of the cleaned parijoto fruits were submerged in $CaCl_2$ 2% solution for 10 min. (sample code : Ca) while the other half were not submerged as a control (sample code : K).

2.2.2. Drying process

Drying was done using a dryer cabinet HetoPowerDry LL1500 . Parijoto fruits were placed on a tray and were spread evenly. The control and pre-treated samples were dried at 60, 70, and 80ºC for 8 hours. During the drying process, the mass of the parijoto fruits was weighed every 1 hour. After drying, the samples were grinded with mortar and pestle for further chemical analysis of the antioxidant activity and total anthocyanin.

2.2.3. Ultrasound-assisted methanol extraction for chemical analysis

Five grams of the grinded dried parijoto fruit was suspended in 50 ml methanol. The mixture was subjected to ultrasound in a sonication bath (BioBase, China) at frequency 40 kHz for 30 min and then was let to sit for another 1 h. The mixture was filtered and the filtrate were diluted into 100 ml using methanol. The extract was stored until further analysis for anthocyanin and antioxidant activity analysis.

2.2.4. Total anthocyanin analysis

Anthocyanin analysis was done using pH differential method described in Turmanidze *et al.* (2016). The methanol extract obtained was further diluted 2x using methanol. Two milliliters of the diluted samples were mixed with 2 ml of KCl buffer solution pH 1 and CH_3COONa buffer solution pH 4.5. The mixture was incubated in a dark room for 15 min. The absorbance of the mixture was measured using UV-Vis spectrophotometer (UV1280, Shimadzu, Japan) at wavelength 520 and 700 nm. Total anthocyanin in the extract were measured using the equations below:

$$A = (A_{520} - A_{700})_{pH\,1} - (A_{520} - A_{700})_{pH\,4,5} \quad (1)$$

$$Total\ Anthocyanin\ (\mathrm{mg/L}) = \frac{A \times MW \times DF \times 1000}{\varepsilon \times L} \quad (2)$$

where A is the absorbance value at different wavelength, MW is the molecular weight of cyanidine-3-glucoside (449.2 g/mol), DF is the dilution factor (20), ε is the molar absorptivity of cyanidine-3-glucoside (26900 L/mol.cm) and L is the cuvet width (1 cm).

2.2.5. Antioxidant activity analysis

Antioxidant activity was measured using the method described in Ahmed *et al*. (2015). The methanol extract was diluted into 1500 ppm using methanol. Afterwards, 0.3 ml of the diluted sample were reacted with 9 ml of DPPH solution (Merck, Germany) in the dark room for 30 min. Blank solution were prepared using 0.3 ml methanol and 9 ml DPPH solution. After 30 min, the absorbance of the sample (A_{sample}) and blank solution (A_{blank}) was measured using UV-Vis spectrophotometer (UV1280, Shimadzu, Japan) at 517 nm. The antioxidant activity is calculated using the equation below.

$$Antioxidant\ activity\ (\%) = \left[\frac{(A_{blank} - A_{sample})}{A_{blank}}\right] \times 100 \qquad (3)$$

2.2.6. Degradation kinetics

The degradation kinetic of the total anthocyanin content and antioxidant activity was fitted into the first order kinetic equation (eq. 4). The degradation kinetic coefficient (k) was obtained from the regression of the experimental data (Fogler, 2006 in Peron *et al*., 2017).

$$\ln(C_t) = \ln(C_0) - kt \qquad (4)$$

C_t = Concentration of total anthocyanin or Antioxidant activity at time *t*
C_0 = Initial concentration of total anthocyanin or Antioxidant activity
k = degradation kinetics coefficient
t = time (h)

Furthermore, half-life time ($t_{1/2}$), the time in which the component's degradation reached half of its initial value, was calculated using eq. 6 below (Peron *et al*., 2017).

$$t_{1/2} = Ln(\frac{0.5}{k}) \qquad (6)$$

$t_{1/2}$ = half-life time
k = degradation kinetic coefficient

2.2.7. Drying kinetics

Water content analysis was done using gravimetric method, which 2.5 g sampel was dried in a porcelain dish at 100°C. Water content analysis was carried out throughout the drying process and the drying kinetic model was done through the moisture ratio (MR) calculation in eq 7 below.

$$MR = \frac{M_t}{M_0} \qquad (7)$$

M_t = moisture content (d.b) at time t
M_0 = initial moisture content (d.b)

The MR data obtained will be used to determine the drying kinetic based on the three types of semi-empirical models (Turan & Firatligil, 2019), which can be seen in Table 1. Mathematical modelling was done using nonlinear regression. Increasing R^2 values and increasing RMSE values are factors in determining the relevant kinetic drying model (Vardin & Yilmaz, 2018). RMSE determination was done following eq 8.

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{exp.i} - MR_{pre.i}\right)^2\right]^{\frac{1}{2}} \qquad (8)$$

N	= number of observation
$MR_{exp.i}$	= MR experimental
$MR_{pre.i}$	= MR prediction

Table 1. Drying kinetic models

Model	Equation
Lewis	$MR = \exp(-kt)$
Henderson & Pabis	$MR = a \cdot \exp(-kt)$
Page	$MR = \exp(-kt^n)$

2.2.8. Effective moisture diffusivity

Effective moisture diffusivity coefficient (D_{eff}) describes the effectiveness of water diffusion processes in a drying process (Chen et al., 2016). The D_{eff} was calculated based on the value of k (slope) of the linear regression of eq. 9 below.

$$\ln(MR) = \ln\left(\frac{6}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff}}{r^2}\right)(\mathrm{t}) \quad (9)$$

$$k = -\frac{\pi^2 D_{eff}}{r^2} \quad (10)$$

MR	= *moisture ratio*
r	= material's radius
t	= time

Activation energy (E_a) is the minimum energy needed to start the reaction (Syah *et al*., 2020). The value of E_a of the moisture diffusion process was obtained through a regression of eq 11 below.

$$D_{eff} = D_0 \, . \, e^{\left(-\frac{E_a}{R}\right)\left(\frac{1}{T}\right)} \quad (11)$$

T	= temperature (K)
R	= ideal gas constant (8.314 J mol^{-1} K^{-1})
D_0	= exponential equation constant

2.2.9. Color intensity

Colour intensity measurement was done through digital imaging analysis. The digital images of the parijoto fruits during drying was captured using a smartphone (Infinix Note 11 Pro, Infinix Mobile, China). The digital images of parijoto fruit were taken every hour during drying inside a modified mini photo studio box. Colour intensity measurements of the digital images based on L*, a*, and b* colours are conducted using the eyedropper tool in Adobe Photoshop CS3 software (Adobe, USA). Measurements were taken three times at different points.

2.2.10. Data analysis

Data analysis and model fitting were carried out using Microsoft Excel and SPSS statistical software analysis v.23. Analysis of variance was carried out to measure statistical significant difference at α 0.5.

3. RESULTS AND DISCUSSION

1.1. Antioxidant activity

Figure 1 shows the antioxidant activity of the parijoto fruit before and after drying. The $CaCl_2$ submersion pre-treatment did not significantly influence the antioxidant activity of parijoto fruit, while higher drying temperature significantly decrease the antioxidant activity of parijoto fruits.

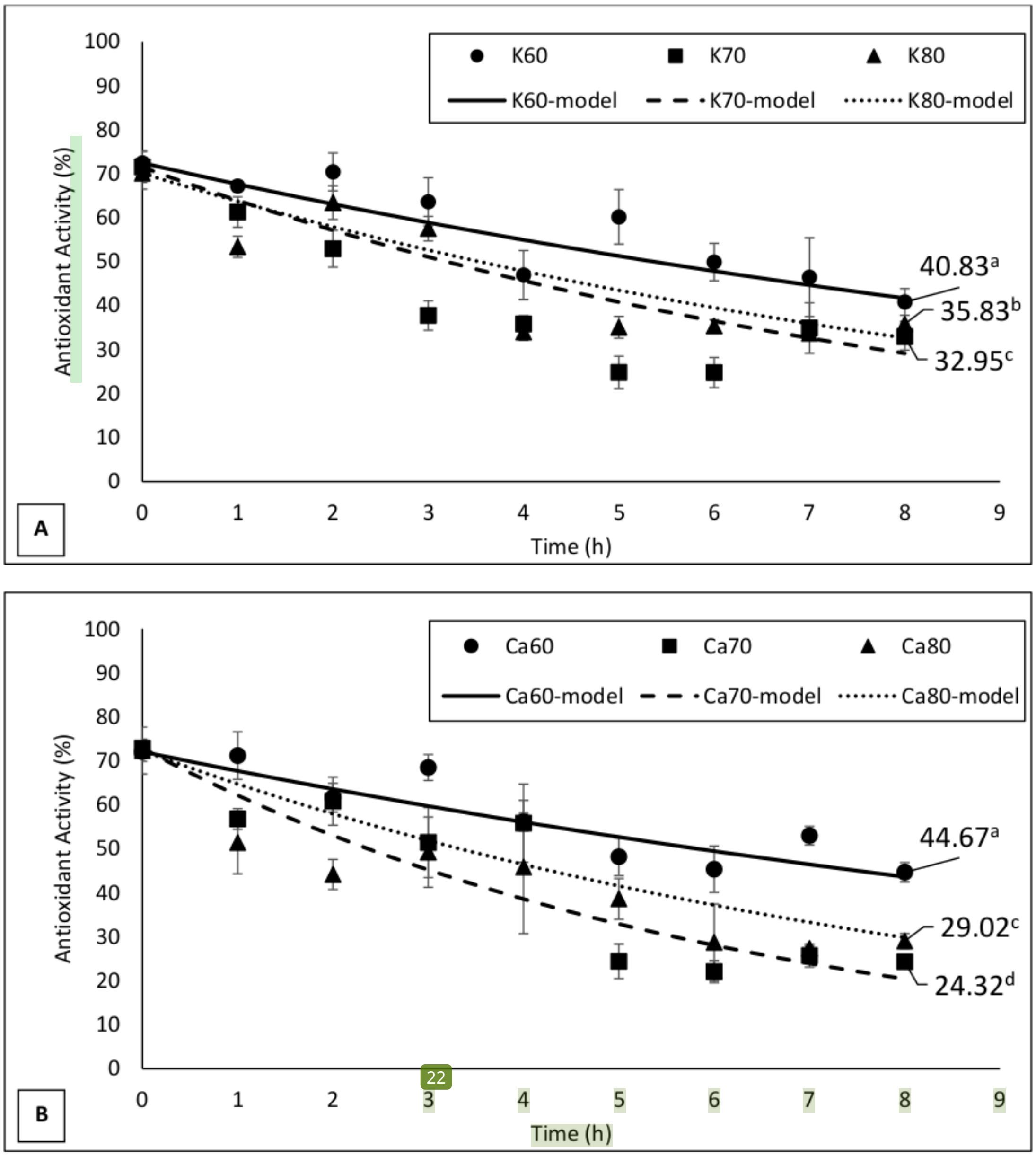
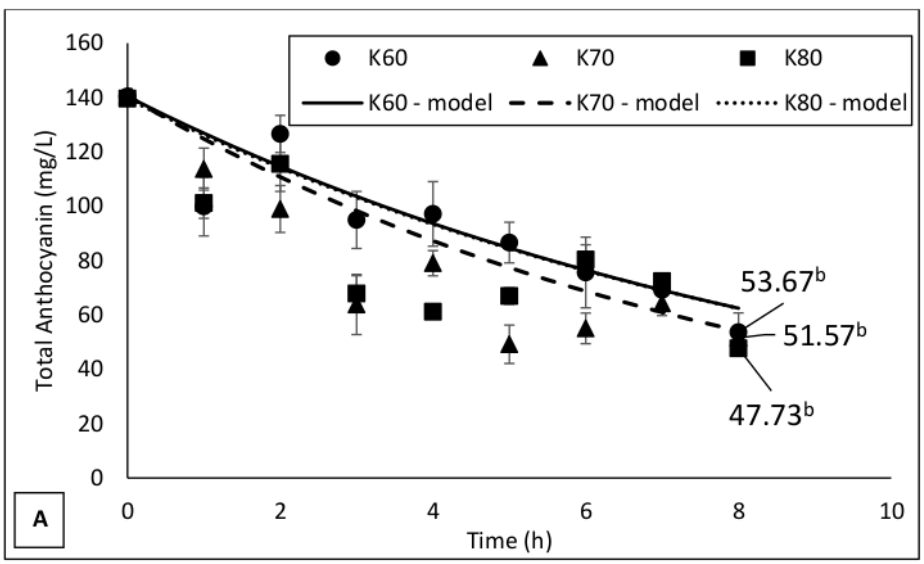


Figure 1. Antioxidant activity of parijoto fruit dried at different temperature without pre-treatment (A) and with $CaCl_2$ submersion (B)

High temperatures can damage antioxidant compounds in materials, leading to decreased antioxidant activity (Hwang & Do Thi, 2014). According to research by Aloo *et al*. (2022), $CaCl_2$ soaking treatment can maintain the ascorbic acid content and antioxidant compounds in bell peppers after 16 days of storage at room temperature. Similar findings can be observed in this study for parijoto fruits dried at 60°C treatment, which shows higher results in the soaked fruit than the control. Calcium ions in $CaCl_2$ can form calcium pectate cross-links with pectin molecules in food materials. This can enhance mechanical properties in parijoto fruit, thereby preserving intracellular antioxidant compounds. Goutam et al. (2010) in Aloo et al. (2022) also mentioned that calcium ions could decrease oxidative enzyme activity, thus maintaining antioxidant activity stability against oxidative degradation in parijoto fruit. However, the positive effect of the CaCl2 soaking was not observed for drying at 70 and 80℃, indicating that the high temperature's destructive effect affects the antioxidant activity more than the protection of the $CaCl_2$ pre-treatment.

1.2. Total Anthocyanin Content

Figure 2 shows the total anthocyanin content of the dried parijoto fruits. Drying caused parijoto fruits to lose its anthocyanin content significantly. However, the results show that $CaCl_2$ pre-treatment significantly preserve the anthocyanin content of parijoto fruits. On the other hand, the drying temperature did not significantly affect the anthocyanin content of the fruit.



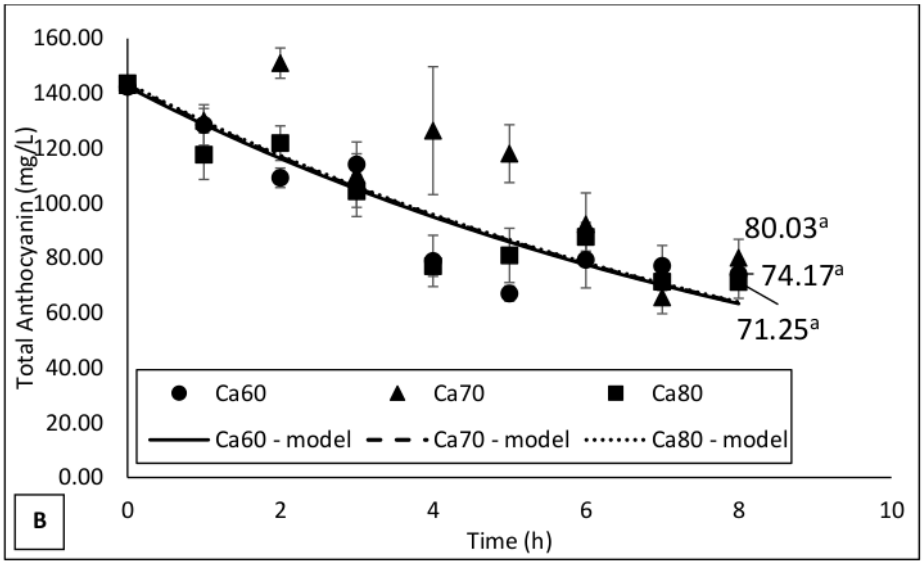


Figure 2. Antioxidant activity of parijoto fruit dried at different temperature without pre-treatment (A) and with $CaCl_2$ submersion (B)

Research by Feng et al. (2022) showed that the utilization of $CaCl_2$ solution can preserve the phenolic compounds and stability of antioxidant compounds in luffa (*Luffa cylindrica*). Calcium pectate cross-links may form during the $CaCl_2$ pre-treatment and they can strengthen the interaction between pectin and anthocyanin (Lin et al., 2016) which may protect the anthocyanin content from the heat treatment during drying. Furthermore, the formation of calcium pectate cross-links enhances the integrity of the cell and prevents cellular damage which encourage of enzymatic browning in food materials due to the release of the polyphenol oxidase (PPO). Since anthocyanins are natural compounds in parijoto fruit belonging to the phenolic group, damage to anthocyanin compounds from the PPO activity can be prevented. This could explain the higher total anthocyanin content in $CaCl_2$-soaked samples compared to the control.

1.3. Degradation kinetic coefficient of antioxidant activity and total anthocyanin content

3

The values of k, $t_{1/2}$ and E_a obtained from the first order kinetic regression from the antioxidant activity and total anthocyanin content during drying are presented in Table 2. These values are useful in describing the properties degradation kinetics during drying and to compare the susceptibility of the properties to heat degradation. Higher k value indicates faster degradation and thus, a more susceptible material. On the other hand, higher $t_{1/2}$ showed a slower and more difficult degradation, which indicate a more stable material (Peron et al., 2017).

The results of the antioxidant activity analysis show that the degradation rate constant (k) increases with higher drying temperatures. This indicates a faster decline in antioxidant activity with increasing drying temperature, affecting the time for antioxidant activity to reach half its initial value ($t_{1/2}$). Thus, it can be concluded that the antioxidant activity of parijoto fruits is very vulnerable to increase in temperature during drying.

Table 2. Values of k and $t_{1/2}$

Parameter	Pre-treatment	Temp (°C)	k (h^{-1})	$t_{1/2}$ (h)
Antioxidant activity	Control	60	0.0691	10.03
		70	0.1121	6.18
		80	0.0955	7.26
	$CaCl_2$	60	0.0628	11.04
		70	0.1590	4.36
		80	0.1109	6.25
Total Anthocyanin content	Control	60	0.1012	6.85
		70	0.1193	5.81
		80	0.1006	6.89
	$CaCl_2$	60	0.0885	7.83
		70	0.0882	7.86
		80	0.0869	7.98

On the contrary, the k value of the total anthocyanin degradation kinetic remained the same with higher drying temperature. This indicate that the temperature difference in this study did not affect the kinetics of the anthocyanin degradation. Interestingly, $CaCl_2$ treatment caused significant reduction in the k value and increase in the $t_{1/2}$ value. This may be due to calcium pectate interactions with anthocyanins as previously discussed (Lin et al., 2016), which can slow down anthocyanin degradation. However, the $CaCl_2$ submersion did not slow down the degradation of antioxidant activity of parijoto fruits during drying.

1.4. *Moisture diffusion properties of parijoto fruits during drying*

The values of D_{eff} and E_a of parijoto fruits dried with different conditions are presented at Table 3. Higher value of D_{eff} indicates that moisture could diffuse out of the fruit tissue more effectively during drying (Chen *et al*., 2016). On the other hand, higher E_a indicates that more energy is required to start moisture diffusion out of the tissue.

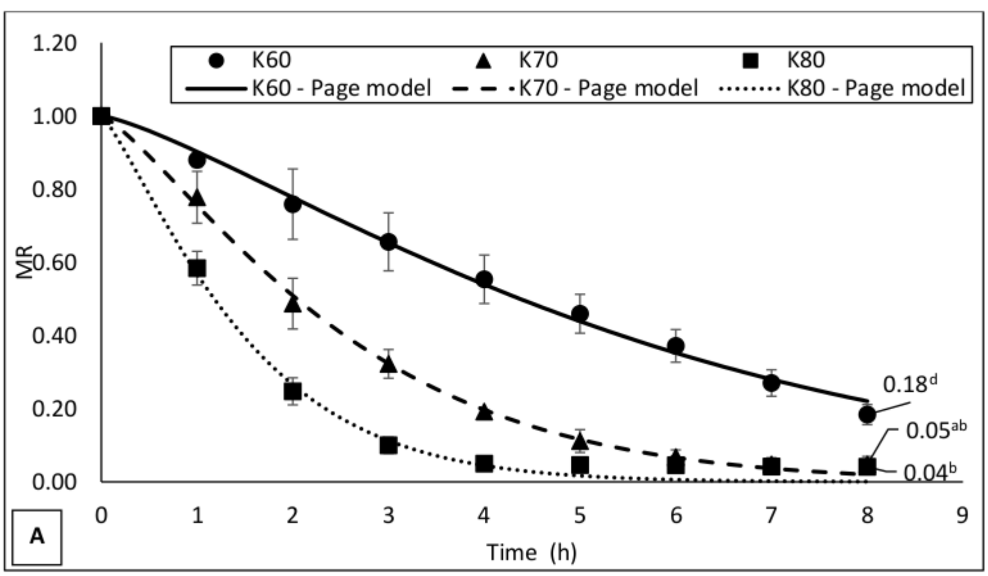
With higher drying temperatures, a higher diffusion coefficient could be achieved. $CaCl_2$ submersion as pre-treatment also significantly increased the diffusion coefficient and lowered the activation energy. This indicates that moisture more easily escaped from the tissue and cells of parijoto fruits. Thus, a more efficient and faster drying occurred for parijoto fruits dried with pre-treatment and at higher temperatures. The results correlate well with the drying kinetics in Figure 3, discussed below. The presence of salts such as $CaCl_2$ could induce osmotic dehydration in fruit cells (Udomkun *et al*., 2014). Osmotic dehydration occurred due to the difference in the osmotic pressure between the materials and the salt solutions used to submerge them. Osmotic dehydration can only partially remove water from the materials and usually uses a pre-treatment as the materials require further processing to be shelf-stable (Berk, 2018).

Table 3. *Effective Moisture Diffusivity* dan Energi Aktivasi

Pre-treatment	Temp (°C)	D_{eff} (m^2s^{-1})	E_a (kJ/mol)
Control	60	3.27×10^{-3}	35.53
	70	6.91×10^{-3}	
	80	6.71×10^{-3}	
$CaCl_2$	60	4.49×10^{-3}	29.48
	70	9.00×10^{-3}	
	80	8.13×10^{-3}	

1.5. Drying kinetics of parijoto fruits

The change in the moisture ratio during drying for all the different treatments is shown in Figure 3. Based on the drying kinetics, a moisture ratio plateau (which indicate no further moisture reduction) was already reached at approximately 7 hours and 4 hours for 70 and 80°C, respectively, with a final moisture ratio of about 0.05 for the control sample and about 0.02 for pre-treated samples. On the other hand, parijoto fruits dried at 60°C, both with or without pre-treatment, did not reach the same level of moisture ratio after 8 hours. Parijoto fruits with $CaCl_2$ submersion reached a lower final moisture ratio than the control samples at all temperature levels, indicating a more effective drying due to the pre-treatment before drying. As discussed, $CaCl_2$ pre-treatment caused osmotic dehydration, significantly increasing moisture diffusivity out of parijoto fruits (Table 3).



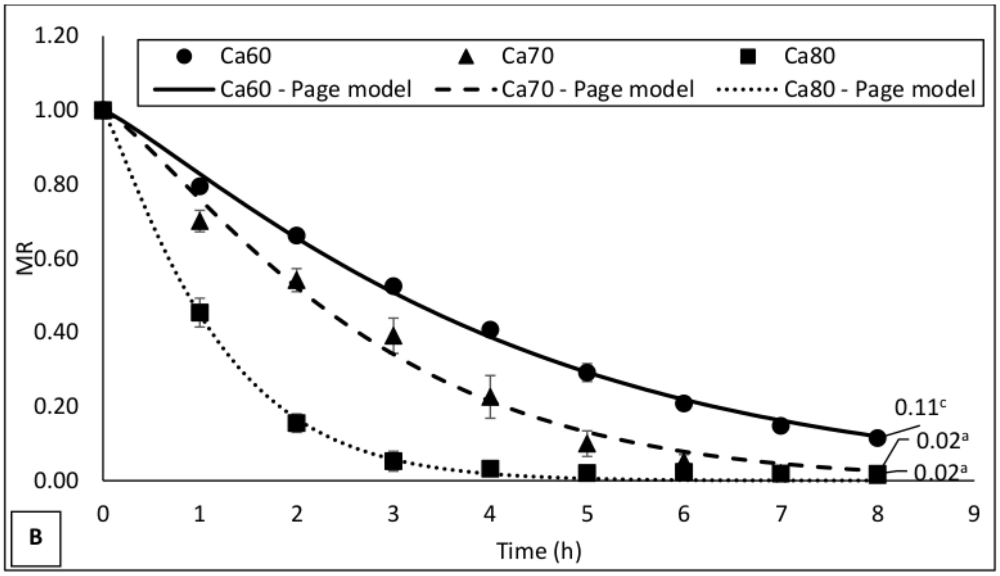


Figure 3. Moisture ratio of parijoto fruit dried at different temperature without pre-treatment (A) and with $CaCl_2$ submersion (B)

Three models were fitted into the drying kinetics, i.e. Lewis, Henderson & Pabis and Page model. The coefficients obtained from the model fitting are presented at Table 3. Based on the R^2 and RSME values, Page model best describe the drying kinetics of parijoto fruits using cabiner dryer. Similar model has been used to describe the drying of gilaburu berries (Dönmez & Kadakal, 2024) and aryl of pomegranate (Vardin & Yilmaz, 2018). The value of k increased significantly with higher temperature and with $CaCl_2$ submersion pre-treatment, which indicate faster drying.

Table 4. Coefficients of drying kinetics with different models

Pre-treatment	Suhu (°C)	Model	k	n	a	R^2	RMSE
Control	60	Lewis	0.165			0.947	0.099
		Henderson & Pabis	0.174		1.041	0.952	0.119
		Page	0.103	1.292		**0.966**	**0.019**
	70	Lewis	0.379			0.977	0.143
		Henderson & Pabis	0.394		1.045	0.980	0.143
		Page	0.281	1.266		**0.988**	**0.015**
	80	Lewis	0.648			0.986	0.195
		Henderson & Pabis	0.657		1.016	0.986	0.185
		Page	0.566	1.229		**0.989**	**0.027**
$CaCl_2$	60	Lewis	0.239			0.989	0.112
		Henderson & Pabis	0.246		1.023	0.990	0.117
		Page	0.189	1.163		**0.995**	**0.016**
	70	Lewis	0.369			0.974	0.141

		Henderson & Pabis	0.377		1.027	0.975	0.133
		Page	0.275	1.242		**0.984**	**0.032**
	80	Lewis	0.857			0.994	0.223
		Henderson & Pabis	0.864		1.006	0.994	0.210
		Page	0.802	1.158		**0.995**	**0.014**

1.6. Color changes of parijoto fruits during drying

Digital image analysis was carried out to the parijoto fruits during drying. The visual representations of the color change are shown in Table 5. The results of the analysis (L*, a* and b* values) are shown in Figure 4-6. Heat from the drying immediately caused a change in the color profile of parijoto fruits from initially dark purple to reddish color. Slight increase of the L* values were observed after drying and a significant increase of the a* value was observed which indicates the increased intensity of the red color after drying. On the other hand, the value of b* changed from negative to positive, which indicate a change of color hue from dominant blue to yellow after drying.

$CaCl_2$ pre-treatment seems to have insignificant impact on the color of parijoto fruits after drying. The change of the color from purple to reddish color due to drying may be caused by the increase in the acidity level of the fruits, due to the change of the proportion after moisture removal. Anthocyanin color changed at different acidity level, in which it becomes redder at acidic environment.

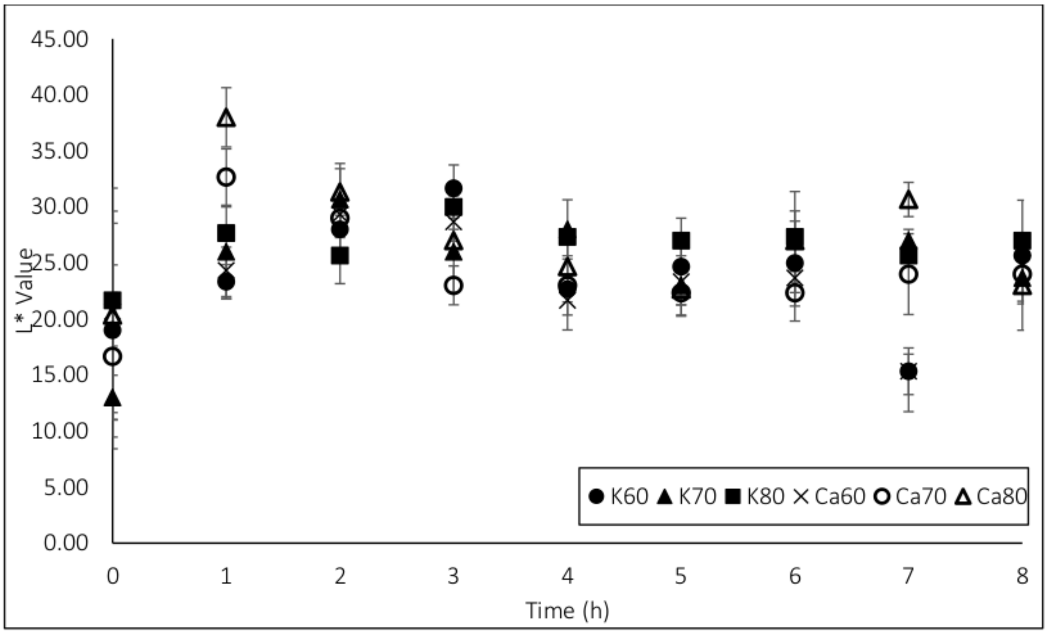


Figure 4. L* values from the digital image analysis of the parijoto fruits during drying with different condition

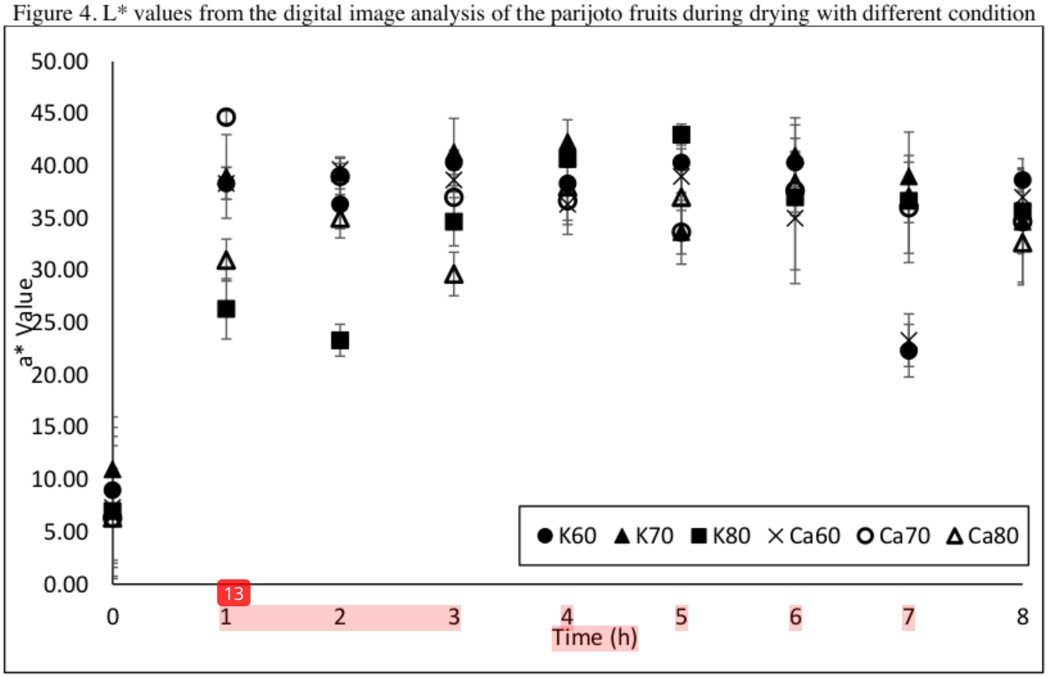


Figure 5. a* values from the digital image analysis of the parijoto fruits during drying with different condition

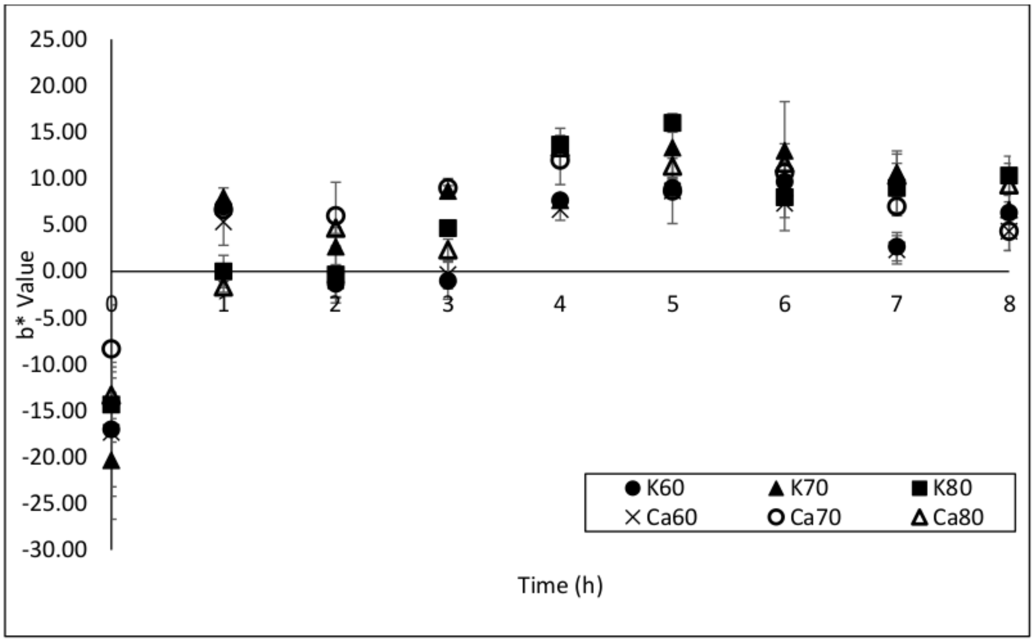
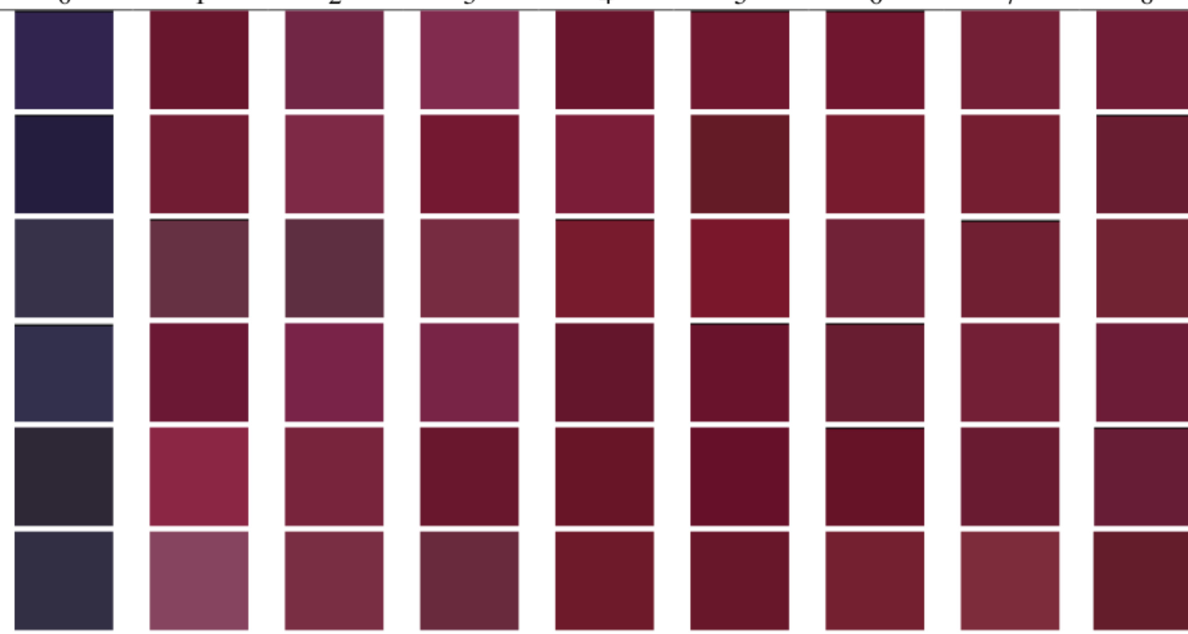


Figure 6. b* values from the digital image analysis of the parijoto fruits during drying with different condition

Table 5. Digital color profile of parijoto fruits throughout drying with different treatments

Treatment	Drying time (h)								
	0	1	2	3	4	5	6	7	8
K60									
K70									
K80									
Ca60									
Ca70									
Ca80									



2. CONCLUSIONS

Drying of parijoto fruit at 60-80ºC may cause significant reduction on its antioxidant activity and total anthocyanin content. The antioxidant activity of parijoto fruits are especially susceptible to an increase in temperature during drying. However, with $CaCl_2$ submersion as pre-drying treatment, the degradation of anthocyanin content can be reduced. $CaCl_2$ submersion and higher drying temperature can also increase the drying rate of parijoto fruit, which make it possible to dry at a shorter time to prevent further degradation of the anthocyanin content. Higher drying rate correlates to a higher effective diffusion coeeficient and the drying kinetics of parijoto fruits can best be described by the Page model.

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