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(RESEARCH ARTICLE)

Hygroscopicity of butterfly-pea under room temperature: Effect on sorption and physicochemical properties

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Abstract

This study aimed to determine the phenomenon during sorption on the whole dried and powder butterfly pea under room temperature storage. In addition, identified thermodynamics, hygroscopicity, degree of caking, and physicochemical properties to determine the quality of dried butterfly pea. Experiments applied the static gravimetric method by saturated salt at 13-27°C. The study results found that the best model was the Peleg Model. The increase in aw affects the increase in EMC during sorption. The comparison results indicated that the EMC of whole dried butterfly pea was smaller than that of powder. The net isosteric heat and differential entropy indicate a decrease with increasing EMC. However, spreading pressure indicates an increase with increasing a_w . Dried butterfly pea has a hygroscopicity characteristic in the extremely hygroscopic category, and it was again identified that the degree of caking of butterflypea powder. Recommendations for drying and storage are carried out until the moisture content is 21.57% kg H₂O/kg butterfly-pea solid (whole); 22.45% kg H₂O/kg butterfly-pea solid (powder). The whole and powder butterfly-pea storage increased in L* and ΔE attributes and decreased antioxidant activity with increasing time and temperature.

Keywords: Degree of Caking; Hygroscopicity; Moisture Sorption Isotherms; Physicochemical properties

1. Introduction

Butterfly-pea has the scientific name *Clitoria ternatea L.* and other names, i.e., butterfly pea and telang. Butterfly-pea has the potential as a high antioxidant. Butterfly-pea can be used to maintain health from several diseases caused by free radicals [35]. Related to the content of anthocyanins such as coumarins, flavonoids, tannins, and anthocyanins [7, 24, 35]. In addition, it contains water content, ash content, fat, protein, crude fiber, carbohydrates, and minerals [19]. The several essential ingredients in butterfly pea make processed butterfly pea into products widely traded on a massive scale. In addition to its high antioxidant activity, butterfly pea is often used as a natural colorant and functional drink because it has an exotic and attractive blue color [15].

In actual conditions, foodstuffs experience changes in temperature and relative humidity [20], which cause desorption and adsorption events to occur. If it occurs continuously for an extended period, it causes characteristic damage and decreases the quality of butterfly-pea products. Thus, affecting consumer interest in the consumption of butterfly-pea products. It affects storage conditions, drying, and other engineering aspects, i.e., process design, optimization, and final product packaging [9, 18, 35].

This problem is a serious problem that must be solved by several approaches, i.e., moisture sorption isotherms (MSI) related to the hygroscopic of the product. In addition, hygroscopic identification and degree of caking of dried whole and powdered butterfly pea have been identified in this manuscript. Hygroscopicity behavior for foodstuffs can be used as a mathematical model approach Mathlouthi and Roge [16] and Oliveira et al. [20] to explain the relationship of

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hygroscopicity with increasing EMC (Equilibrium Moisture Content) during sorption. As a result, the hygroscopicity of butterfly pea affects changes in physicochemical properties during storage. Determining the quality of butterfly pea in whole dry products and powder is extremely important in the development of food technology and as a determinant of the quality of butterfly-pea products in the modern era. In addition, many researchers have not carried out product development in terms of drying and storing butterfly pea. So this study is the basis from the initial processing to the final product of butterfly pea. However, this study aims to identify the hygroscopic correlation of butterfly-pea products, correlated with the degree of caking and thermodynamic properties, and predict the sorption behavior under room temperature. In addition, it further identified color changes and antioxidant activity during storage.

2. Material and methods

2.1. Sample preparation

Butterfly-pea flower samples were taken from Jember district, Indonesia. Butterfly pea was harvested at 3 pm (local time), and distribution was conditioned at 27°C for 15 hours. The harvested butterfly-pea (dark blue-dark purple, double flower) flowers were then tray dried for 12 hours, 50°C. After drying, the moisture content of the sample is 0.28 kg H_2O/kg butterfly-pea solid. The samples were divided into the whole dried and the powdered butterfly-pea sample. Whole dried butterfly-pea samples were immediately used as samples after drying. The butterfly-pea powder sample was made by immediately reducing the size using a blender and sieved with a 400 μ m sieve after drying. All samples were conditioned in airtight containers to avoid environmental exposure until samples were used as MSI experiment samples.

2.2. MSI determination

The MSI experiment used the static gravimetric method with five saturated salts. Saturated salt using MgCl₂, K₂CO₃, NaCl, KCl, and BaCl₂ (Merck, Germany). The sample used 0.500 grams. The experiment refers to the reference by Hawa et al. (2021) with slight modifications. The incubation temperature for MSI testing is below room temperature, i.e., 13, 18, and 27°C. To reach EMC, conditioning using a commercial refrigerator (Polytron, Indonesia) for 5-10 days. Determination of the mass of the sample solids refers to the reference by Hawa et al. [8] for 4-5 hours at 105°C.

2.3. Mathematical sorption model

The models used in this study are the GAB Model (Eq. 1) and Peleg (Eq. 2). This model is the best predicting EMC during sorption at the a_w (water activity) level that has been determined in the study. The GAB model has three constants: M_o (monolayer moisture content, %db), C, and K. M_o is the monolayer moisture content, and C and K are model constants (Peleg, 2020). The Peleg model has four constants, i.e., a, b, C, and n dimensionless [7].

$$\left[EMC_{(GAB)}\right] = \left[\frac{M_o CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}\right] \tag{1}$$

$$\left[EMC_{(Peleg)}\right] = \left[aa_w^b + Ca_w^n\right]$$
⁽²⁾

2.4. Net isosteric heat

The net isosteric heat can be used as the equation approximation in many references, i.e., the Clausius-Clapeyron as in Eq. 3 [6]. The symbol Q_{st} is the net isosteric heat of sorption, J/mol and R is the gas constant, 8.314 J/mol.K. The Q_{st} value was determined by plotting the ln aw versus 1/T curve at the best model EMC condition.

$$-R\left[\frac{d\ln(a_w)}{d\frac{1}{T}}\right] = \left[-Q_{st}\right] \tag{3}$$

2.5. Differential entropy heat

The reference of Fakhfakh et al. [6] provides a measure of determining the heat of differential entropy. The heat of differential entropy is determined by combining the Gibbs-Helmholtz equation with the Gibbs free energy equation. The result of merging is obtained by equation (Eq. 4). The intercept value is obtained with In a_w versus 1/T curve plot at the EMC level. This intercept value is then multiplied by the gas constant to obtain the value of differential entropy (ΔS , J/mol.K).

$$\left[-\ln a_{w}\right]_{Me} = \left[\frac{-Q_{st}}{RT} - \frac{\Delta S}{R}\right]$$
(4)

2.6. Spreading pressure

Spreading pressure is known using the GAB model approach as in Eq. 5 [13, 31]. The symbol of ϕ is the spreading pressure (J/m²); C_z is Boltzmann's constant, 1.38x10⁻²³ J/K; T is the absolute temperature, K; A_z is a single area of a water molecule, 1.06x10⁻¹⁹ m²; C and K are constants of the GAB model that have been fitted with experimental data.

$$\phi = \frac{C_z T}{A_z} ln \left[\frac{1 - (K.a_w) + (C.K.a_w)}{1 - (K.a_w)} \right]$$
(5)

2.7. Hygroscopicity

The hygroscopicity research procedure refers to the study conducted by Caparino et al. [3] with slight modifications. The sample of dry butterfly-pea powder (0.28 kg H₂O/kg butterfly-pea solid, 400 μ m) was taken at 0.500 gram. The sample was placed into an airtight container in which there was a saturated salt of NaCl (75.74% RH). Samples were incubated at 27°C for five days. Hygroscopicity (kg H₂O/kg butterfly-pea solid) is 1 kg of water vapor adsorbed per 100 kg butterfly-pea solid. Hygroscopicity was determined by Eq. 6 [27, 32]. H is hygroscopicity (%); Mci is the free moisture content of the sample (before the experiment, H₂O/kg butterfly-pea solid); Δ m is the increase in sample mass (after the experiment, gram); mt is the initial mass (gram).

$$H = \frac{Mci + (\Delta m/mt)}{1 + (\Delta m/mt)} x 100\%$$
(6)

2.8. Degree of caking

The degree of caking procedure was carried out on powdered dried butterfly-pea samples. The research procedure of degree of caking refers to [20, 32] with slight modifications. The butterfly-pea powder tested for hygroscopicity is then determined using an oven at a temperature of $105 \circ C$ for 1 hour. Then it was sifted ($400 \mu m$) until nothing was separated from the sieve. Determination of calculating the degree of caking can be used in Eq. 7 [32]. DC is the degree of caking, %; Q₁ is the initial powdered butterfly-pea mass (gram); Q₂ is the mass of butterfly-pea powder left in the sifter (gram).

$$DC(\%) = \left[\frac{Q^2}{Q_1}\right] x 100\% \tag{7}$$

2.9. Color measurement

The initial sample of butterfly-pea with a water content of 0.28 kg H_2O/kg butterfly-pea solid was conditioned using the static gravimetric method (saturated salt solution of NaCl). The samples were then incubated at a temperature of 13-27°C. Measurement of butterfly-pea color using the image approach method. Images were captured using a Vario-Tessar ZEISS 3.3-6.3/4.5-36 20.1 MP lens (1x optical magnification). The picture was taken at 10 cm from the top of the box. The cube box has 22x22x22 cm with an LED light on the top. LED light is conditioned constant at a light intensity of 25.5±0.1 W/m². The background of the image uses a white woven fabric. Analysis of L*, a*, and b* attributes using the Matlab 2019a application. Determination of the total color difference using Eq. 8 [23].

$$\Delta E = \sqrt{(\Delta a *)^2 + (\Delta b *)^2 + (\Delta L *)^2}$$
(8)

2.10. DPPH radical scavenging activity

Measurement of antioxidant activity was carried out according to the procedure reported Mir *et al.* [17] with the DPPH free radical scavenging method. 80 μ l of butterfly-pea methanol extract was mixed with 1000 μ l of DPPH (0.1 mM). The sample was then incubated for 30 minutes, and the absorbance value was measured at a wavelength of 515 nm. The percent inhibition of butterfly pea during storage can be determined in Eq. 9 [17].

$$\% inhibition = \frac{control \ absorbance - sampel \ absorbance}{control \ absorbance} x100 \tag{9}$$

2.11. Statistical analysis

The research was completed in three repetitions for all data. The experimental EMC data plot and model predictions were determined the best based on the RMSE, P, and R² values [7, 14, 33]. The research data (hygroscopicity, L* & ΔE , and %inhibition) were analyzed using two-way analysis of variance (ANOVA) and presented in the form of mean±SD. The significant difference using Turkey's with a 95% confidence level (Microsoft Excel 2016, Microsoft Corp., USA).

3. Results and discussion

3.1. Moisture sorption isotherms

Figure 1 shows the behavior pattern of butterfly-pea at each level of a_w. EMC increases with increasing a_w. Each period the temperature increases, the EMC decrease. In addition, the results of the comparison identification of whole dry butterfly pea have lower EMC than powdered dried butterfly pea. The increase in EMC with increasing temperature was correlated with less hygroscopic properties at higher incubation temperatures [28]. They reported that the active sites in foodstuffs decreased to bind water molecules at high temperatures.

Forms Models T (°C)		T (ºC)	Constants	R ²	Р	RMSE
	GAB	13	C=179309; M₀=0.126; K=0.857	0.9867	0.0461	0.0240
		18	C=179304; M ₀ =0.103; K=0.919	0.9845	0.0572	0.0221
1471 I		27	C=51988.4; M₀=0.075; K=0.961	0.9922	0.0501	0.0140
Whola	Peleg	13	a=0.49;b=0.954;C=15.8;n=49.82	0.9912	0.0207	0.0068
		18	a=0.44;b=1.00;C=-1.63; n=19.8	0.9982	0.0354	0.0138
		27	a=0.41;b=1.25; C=2.404; n=23.52	0.9999	0.0030	0.0006
		13	C=1206558; M _o =0.136; K=0.854	0.9732	0.0443	0.0243
	GAB	18	C=1206551; M _o =0.117; K=0.902	0.9848	0.0488	0.0219
		27	C=1206547; M _o =0.076; 0.97129	0.9907	0.0537	0.0174
Powder		13	a=3.49;b=31.94;C=0.490;n=0.881	0.9998	0.0061	0.0022
	Peleg	18	a=2.19;b=23.80;C=0.498;n=1.03	0.9991	0.0202	0.0134
		27	a=2.56;b=21.79;C=0.390;n=1.112	0.9999	0.0041	0.0009

Table 1 Constants and models fit test values

Figure 1 shows an area crossing the line at $a_w 0.00-0.45$ with $a_w>0.45-1.00$. The dividing line shows two sorption phenomena (desorption and adsorption) on the initial sample of 0.28 kg H₂O/kg butterfly-pea solid during the sorption study. The sample with saturated salt MgCl₂ and K₂CO₃ showed desorption phenomena, which indicated a reduction in the sample amount to the equilibrium limit. The EMC in the saturated salt condition is 0.10-0.21 kg H₂O/kg butterfly-pea solid (whole) and 0.11-0.24 kg H₂O/kg butterfly-pea solid (powder). In addition, the samples with saturated salts NaCl, KCl, and BaCl₂ showed adsorption phenomena which indicated the addition of a particular water substance in the sample to the equilibrium limit. The EMC under these conditions is 0.29-0.60 kg H₂O/kg butterfly-pea solid (solid) and 0.29-0.64 kg H₂O/kg butterfly-pea solid (powder).

The results of the experimental EMC measurement with various a_w levels were then modeled using the GAB and Peleg Models. These two models have been the best sorption models on whole dried butterfly pea [7] with a running of 30-50°C. The advantage of the GAB model is that constant M₀ can describe the critical limit of the monolayer moisture content. The results of this experimental fitting show that the water content of the monolayer in the region of a_w 0.075-0.126 kg H₂O/kg butterfly-pea solid (whole) and a_w 0.076-0.136 kg H₂O/kg butterfly-pea solid (powder). The constants and fitness values for the sorption model can be seen in Table 1. The best model to describe the sorption behavior of butterfly-pea is the Peleg Model. The Peleg Model provides model accuracy up to R²>0.9900. Based on the BET classification, it was shown that whole dried and powder-dried butterfly pea had a type III curve characteristic.



Figure 1 Butterfly-pea sorption modeling on Peleg

3.2. Net isosteric heatHeading

The net isosteric heat behavior during sorption on butterfly pea (whole and powder) can be seen in Figure 2. The behavior of whole and powder-dried butterfly pea showed the same pattern. For each increase in EMC, there is a decrease in the net isosteric heat. The maximum energy required for sorption at EMC conditions of 0.05 kg H_2O/kg butterfly-pea solid is 0.132 kJ/mol (whole) and 0.167 kJ/mol (powder). The comparison of values on butterfly pea showed that the net isosteric heat of the powder was greater than that of the whole dry. It is associated with the condition of the hygroscopic level of the sample (seen in Table 2). The high hygroscopic level indicates that the butterfly pea powdered has greater desorption and adsorption power proportional to the energy used. However, this is uncommon for whole dried butterfly pea, which has a lower hygroscopicity than powder.



Figure 2 Net isosteric on butterfly-pea during sorption

3.3. Differential entropy heat

The differential entropy heat behavior during sorption on butterfly pea can be seen in Figure 3. The behavior of whole dried butterfly pea and powder showed the same pattern. With each increase in EMC, there is a decrease in differential entropy heat. The maximum entropy energy at EMC conditions of 0.05 kg H₂O/kg butterfly-pea solid is 0.009 kJ/mol.K (whole) and 0.008 kJ/mol.K (powder). The difference in the number of entropy in the butterfly-pea sample is associated with the level of hygroscopicity. At a high level of hygroscopicity, there is more fluid mobility of water vapor.

For this reason, dried butterfly-pea powder has a lower level of impairing the mobility of water vapor. This case is also associated with the integral entropy heat behavior, which indicates the mobility capacity of water molecules [12]. The increase in entropy causes disturbances in the mobility of water molecules, especially at the monolayer water content. Barati et al. [1] informed that the differential entropy behavior is still related to a strong dependence on the moisture content, especially under low states.



Figure 3 The differential entropy on butterfly-pea during sorption

3.4. Spreading pressure

Figure 4 shows the spreading pressure behavior on butterfly pea (whole and powder) during sorption. The determination of spreading pressure showed an increase with increasing a_w. In addition, the lower the incubation temperature, the lower the spreading pressure. At a_w 0.05 the minimum values are 0.335-0.379 kJ/mol.K (whole) and 0.406-0.411 kJ/mol.K (powder). The a_w condition is 0.90, the maximum value are 0.496-0.559 kJ/mol.K (whole) and 0.566-0.593 kJ/mol.K (powder).



Figure 4 Spreading pressure on butterfly-pea during sorption

The spreading pressure is the free energy on the substance's surface and indicates increased tension during sorption [11]. From the data presented in this study, spreading pressure is associated with the degree of hygroscopicity and impaired mobility by the heat of entropy. From a hygroscopic point of view, butterfly pea powder has a higher hygroscopic level than a whole butterfly pea. Therefore the sorption process is better. In addition, the condition of the powder also indicates less impaired entropy mobility. In this condition, informing the fluid mobility of water molecules directions increases the spreading pressure.

3.5. Hygroscopicity

The appearance of the powder and whole butterfly-pea condition during storage did not transform extensively (Figure 5). Qualifies low-temperature storage conditions and 75% RH to prevent undesirable discoloration. Based on statistical analysis, the difference in sample form did not significantly (p>0.05) affect changes in hygroscopicity. Table 2 shows the hygroscopicity data of whole dried butterfly pea and powder. The hygroscopicity value of butterfly pea was 29.29±0.91% (powder) and 28.87±0.42% (whole). The increase in the percent hygroscopic than whole dried butterfly pea. This condition was correlated with lower whole dry butterfly-pea EMC in this study. The hygroscopicity data in this study were classified as extremely hygroscopic. The results are more hygroscopic than the encapsulated level of blackberry extract, which was 14.49-19.42% [26]; paprika by 6.86-15.33% [32]; grugru palm powder by 5.17-6.39% [20]. According to Schuck et al. [30], at 75%, RH has a value of more than 25%, indicating that the food product is extremely hygroscopic.



Figure 5 Butterfly-pea during storage at hygroscopicity condition (RH 75%)

3.6. Degree of caking

Table 2 shows the degree of caking on powdered dried butterfly pea. In this study, the value of the degree of caking was carried out on the butterfly-pea powder product. The degree of cacking butterfly-pea dry powder is 10.20±1.59% and is often found in some foodstuffs, i.e., grugru palm powder [20]. Experimental data showed that 10.20% of the total mass of butterfly-pea powder experienced a decrease in product quality. The degree of caking is a reaction condition that is not required. This case is related to the agglomerated and sticky powder matesrial resulting in a functional decline [20]. In addition, Oliveira et al. [20] revealed that the hygroscopic treatment variation was also determined by powder granulation. This case indicates that the insignifican reduction has a large surface area with various active sites.

Table 2 Degree	of caking and	Hygroscopicity	v of butterfly-pea	(RH 75%)
				(====

Samples Hygroscopicity (%)		Degree of Cacing (%)	
Whole	28.87±0.42ª	ND	
Powder	29.29±0.91ª	10.20±1.59	

ND: not detected. Columns with lowercase denoted not significant (p>0.05) differences between samples form. Each value represents the mean±SD (n=3).

3.7. Drying and storage recommendations

Recommendations for drying and storing butterfly-pea products can be used in the sorption behavior approach. Storage recommendations in this study are at a temperature of 27° C and a_{w} 0.60 (60% RH). Based on Hawa et al. [9] provide storage recommendations at these temperatures.

From the sorption curve at 27°C (Figure 1), it was found that the drying recommendation was 21.57% kg H_2O/kg butterfly-pea solid (whole); 22.45% kg H_2O/kg butterfly-pea solid (powder). This case is related to the EMC condition of the material in butterfly pea, which is stored at that temperature to reach equilibrium. This approach study minimizes changes in the quality characteristics of butterfly pea due to sorption. In addition, drying under EMC conditions results in the use of more economical drying energy.

3.8. Color changes

The color change is significant in creating consumer acceptance of butterfly-pea products. Table 3 informs that dried whole and powdered butterfly pea experienced an increase in the attributes of L* (brightness) and ΔE (total color difference). A statistical analysis of all samples indicates that the temperature difference did not significantly (p>0.05) affect L* and ΔE changes. The increase experienced in this type of butterfly-pea product was due to storage time and increased treatment temperature. The increase in L* indicated that the whole butterfly-pea product and powder underwent color fading before turning dark. An increase in L* and ΔE attributes has been reported by Rabeler and Feyissa [25] on chicken breast meat with thermal treatment. During storage, the color change is due to temperature and

oxygen in the product incubation chamber [23]. The increase in the L* attribute is often associated with a non-enzymatic browning reaction at an increase in treatment temperature [29]. In a study conducted by Buve et al. [2], changes in ΔE were associated with changes in anthocyanin content.

	Forms	T (ºC)	L*		ΔΕ	
			0 day	10 days	0 day	10 days
	Whole	13	80.75±1.04 ^A	81.82±0.78 ^{aA}	1.941±1.03 ^A	3.00±0.80 ^{aA}
		18		84.14±0.73 ^{aA}		3.00±0.80 ^{aA}
		27		85.89±1.21 ^{aA}		5.32±0.73 ^{aA}
	Powder	13	32.45±0.04 ^B	33.12±1.13 ^{aB}	0.423±0.05 ^B	1.71 ± 0.64 ^{aB}
		18		33.70±0.43 ^{aB}		2.01±0.29 ^{aB}
		27		34.17±0.03 ^{aB}		2.30±0.06 ^{aB}

Table 3 Color measurement during storage at hygroscopicity condition (RH 75%)

Columns with lowercase denoted not significant (p>0.05) differences between temperatures. Columns with uppercase denoted significant (p<0.05) differences samples form. Each value represents the mean±SD (n=3).

3.9. DPPH radical scavenging activity

Storage at 75% RH at each incubation temperature level caused a decrease in the percentage of inhibition over a period. This case can be seen in Table 4, where the percentage of inhibition decreased during ten days of storage. Statistical analysis of all samples indicated that the temperature difference did not significantly (p>0.05) affect the change in %inhibition, both the temperature difference and the sample form. At the beginning of storage of dried whole and powdered butterfly pea was 73.43±1.17%. The identification of DPPH radical scavenging activity revealed that storage at 13°C decreased slowly and at 27°C decreased faster. It is was concluded that the incubation temperature changed the percentage of inhibition in dried whole and powder butterfly pea. Decrease in antioxidants during storage has been reported by Zheng and Lu [34] in pineapple juice products and Oms-oliu et al. [21] on cut watermelon. Storage at low temperatures of 6-23°C tends not to experience significant changes in antioxidant activity [10].

Table 4 DPPH radical scavenging activity at hygroscopicity condition (RH 75%)

Forme	Т (°С)	DPPH (%inhibition)			
FORMS		0 day	5 days	10 days	
	13		66.50±1.34 ^{aA}	65.65±0.59 ^{aA}	
Whole	18	73.43±1.17 ^A	66.33±1.17 ^{aA}	64.64±0.29 ^{aA}	
	27		63.28±1.06 ^{aA}	62.77±0.78 ^{aA}	
	13		65.14±0.59 ^{aA}	64.97±0.51 ^{aA}	
Powder	18	73.43±1.17 ^A	63.45±1.02 ^{aA}	62.44±1.76 ^{aA}	
	27		59.73±1.06 ^{aA}	57.36±0.78 aA	

Columns with lowercase denoted not significant (p>0.05) differences between temperatures. Columns with uppercase denoted not significant (p>0.05) differences samples form. Each value represents the mean±SD (n=3).

4. Conclusion

This study has exposed moisture sorption isotherm, hygroscopicity, degree of caking, color measurement, and DPPH radical scavenging activity. The Peleg Model best describes the desorption-adsorption behavior of dried whole and powdered butterfly pea. The net isosteric behavior and differential entropy decrease in energy with increasing EMC. The increase in aw affects the increase in the spreading pressure of the butterfly pea. Storage recommendations are carried out at a temperature of 27° C at 60% RH, considering that this temperature is near the average room temperature in Indonesia. The hygroscopicity of whole dried butterfly pea and powder is categorized as extremely hygroscopic. The L* and Δ E attribute analysis results showed an increase with increasing time and temperature. In addition, the

antioxidant activity of dried whole and powdered butterfly pea decreased with increasing time and temperature. Antioxidant activity and color did not significantly affect temperature.

Compliance with ethical standards

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Disclosure of Conflict of interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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