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Mathematical Modeling to Describe Drying Behavior of Kyoho (*Vitis labruscana*) Skin Waste: Drying Kinetics and Quality Attributes

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Abstract: Grape skin (Kyoho: *Vitis labruscana*), a by-product of processed grapes, was experimentally investigated for its drying behavior at different drying temperatures with five thin layer drying models. Moreover, we determined the effect of drying temperature on the bioactive capacity of Kyoho skin. The experimental moisture ratio decreased with increasing drying temperature. The drying process was predicted by mathematical models, such as Page (303.15 K: R^2 = 0.9815, 333.15 K: R^2 = 0.9685) and two-term (313.15 K: R^2 = 0.9639, 323.15 K: R^2 = 0.9737) models. Moisture diffusivity (D_{eff}) ranged from 2.87 × 10^{-8} to 9.82 × 10^{-8} m^2/s, with an activation energy (E_a) of 33.78 ± 1.06 kJ/mol. Total phenolic compounds (0.37 ± 0.04 to 0.23 ± 0.03 mg GAE/g) and antioxidant activities (DPPH• activity of 93.06 to 73.31%) of Kyoho skin were significantly affected by drying temperature. Thus, this study concluded that the drying process decreased the bioactive potential of grape skin; therefore, we recommend that the food processing industry needs to consider drying variables during the processing of grape skin-based value-added products for improved food production.

Keywords: grape skin waste; agricultural management; oven drying; thin layer mathematical models; drying kinetics; grape skin waste recycling; food productivity; antioxidant potential

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1. Introduction

Grapes are one of the most culturally and economically important horticultural crops in the world. A survey conducted by archaeologists proposed that cultivation of domesticated grapes (*Vitis vinifera* subsp) began as early as 6000 to 8000 years ago from the wild ancestor of grape (*Vitis vinifera* subsp. *Sylvestris*) in the Near East [1]. Kyoho (*Vitis labruscana*), a new commercial grape variety cultivated in Taiwan [2], has received significant consumer attention due to consumer appeal and market availability and low price (compared to imported grape cultivars) in Taiwan. In light of recent grape production statistics in Taiwan, Kyoho has contributed significantly to the growth of the national economy [2]. The cultivation of Kyoho in Taiwan resulted in aggregate production of 100,000 t per year [2], in which >30% was discarded as skin waste. However, visual observation of Taiwan's grape-consuming market has indicated a major challenge in the utilization of grape residues (i.e., skin and seed). For instance, Taiwanese consumers prefer to eat the flesh parts of grapes and discard the skins and seeds, which creates an agricultural waste that has become a challenge to the environment and the food industry.

The transformation of agricultural waste into value-added foods has the potential to develop new and energy-efficient processing technologies, as well as to feed the projected increasing human population. In fact, consumers' interest in waste utilization has risen (with a focus on value addition) due to popular television shows, international food shows (e.g., the Taipei International Food Show, Taiwan: https://www.foodtaipei.com.tw, accessed on 10 July 2019), and food science education, but the industry has failed to

implement the utilization and/or value addition of these residues, due to a lack of skills regarding the processing methods in developing and/or under-developed countries.

Early scientific advances in the field of food processing have led to the development of grape utilization methods [3] for functional food applications. Drying is one of the processing methods used to maintain safety and extend the shelf-life of produce [4]. Several studies reported that the drying methods effected several changes in quality aspects and bioactive content of the product [5], which demonstrated that drying temperature and time could affect the final quality of the product. For example, Sridhar and Charles [6] studied the influence of drying temperature on physico-chemical properties of Kyoho seeds and concluded the significant changes in physico-chemical properties of grape seeds are affected by drying temperature. Several studies reported an increase/decrease in bioactive potential of different fruit crops, which remains a challenge for many food engineers by playing a proactive role in optimizing drying temperatures and time to preserve the quality of the product.

Mathematical modeling is a relatively suitable and sustainable approach for describing the mechanism of the drying process [4]. Recent theoretical advances in mathematical modeling of drying have revealed that the drying process (mathematical fitting) of agricultural products requires specific statistical methods for the accurate explanation of drying kinetics [7]. Therefore, in general, thin layer models are widely used models to explain the water loss phenomenon and heat penetration mechanism during the hot air oven drying process [8]. Among the thin layer models, semi-empirical models, such as Lewis, Page, Henderson–Pabis, logarithmic, and two-term models, are used to describe the moisture transfer from agricultural materials [9]. Some research has been conducted on the processing of grape by-products, such as seeds [10] and pomace [11]; however, there have been no controlled studies that explored the drying mechanism of Kyoho skin. Recently, approaches have been developed over the last two years for the effective utilization of grape skin using air-borne ultrasonic application [12]. Hence, working experience with the Kyoho cultivar and the local grape consumer market scenario prompted us to explore the possible ways of processing grape by-products, particularly Kyoho skin, for food quality consideration.

Therefore, the study investigated the effect of drying temperatures (303.15, 313.15, 323.15, and 333.15 K) on the drying characteristics of Kyoho skin using five thin layer drying models (i.e., Lewis, Page, Henderson–Pabis, logarithmic, and two-term). In addition, the food quality of the dried Kyoho skin was evaluated based on the influence of drying conditions on the bioavailability of bioactive compounds.

2. Materials and Methods

2.1. Chemicals and Instruments

Sodium hydroxide, sodium carbonate, sodium nitrite, ABTS (2,2′-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid), potassium persulfate, and aluminum chloride were obtained from Showa Chemical Industry Co., Ltd. (Tokyo, Japan). Folin–Ciocalteu's phenol reagent (2 N; purity $\geq$ 98%), 2,2 diphenyl-1-picrylhydrazyl (DPPH), gallic acid, and ethanol were purchased from Sigma-Aldrich GmbH (Steinheim, Germany). Ascorbic acid, methanol, and quercetin were obtained from Fisher Scientific (Taipei, Taiwan). All other chemical reagents used in this study were of analytical grade. Spectrophotometric analysis was carried out using a DU® 730 UV/Vis Spectrophotometer (Beckman-Coulter, Pasadena, CA, USA), whereas color analysis was performed by a colorimeter (ZE 2000, Nippon Denshoku Industries Co., Ltd., Tokyo, Japan). Moisture content was determined using a moisture analyzer (MX-50, A&D Company Ltd., Tokyo, Japan).

2.2. Sample Preparation

Fresh Kyoho (*Vitis labruscana*) grape samples were purchased from the local commercial market in the town of Neipu (22°36′53.1252″ N and 120° 34′14.862″ E), Pingtung, Taiwan. Fresh grape samples, with uniform size and color without surface injuries, were transferred into low-density polyethylene (LDPE) sampling bags (280 × 400 mm), trans-

ported to the laboratory within 10 min, and then stored at 4 ± 1 °C, until the drying experiments were conducted.

Grape samples were washed with tap water to remove foreign impurities, and blotting paper was used to remove surface water from grape samples. Lengthwise cuts were performed manually to separate the slip-skin (skin is relatively loose and peels easily) and washed with running water at 25 °C for 30 s to remove adhering juice [2]. The initial moisture content of Kyoho skin was determined in triplicate by the moisture analyzer ±0.20%. Kyoho skin samples were packed in sampling bags and stored at 4 ± 1 °C until drying experiments (usually within 5 days).

2.3. Drying Process and Experimental Drying Kinetics

Drying experiments of Kyoho skin were conducted in a conventional oven (DOS-45, Deng Yng, Taipei, Taiwan) at four constant drying temperatures (303.15, 313.15, 323.15, and 333.15 K at a constant relative humidity of 70 ± 2%; sample load density of 0.67 ± 0.24 kg/m^2) with an air velocity of 1.0 m/s, respectively. Samples dried at room temperature, 25 °C (at a constant relative humidity of 70 ± 2%; sample load density of 0.67 ± 0.24 kg/m^2), with an air velocity of 1.0 m/s were used as the control for color experiments. Kyoho skin samples were uniformly placed in rectangular stainless steel drying trays (23 cm in height × 33 cm in width) and dried in a hot air oven at the drying temperatures. Extreme care was taken to weigh the sample during drying and weight measurements were quickly performed between the experiments to avoid any interference. The initial and final weights of Kyoho skin samples were determined using an electronic balance (TB- 214, Denver Instrument, Taipei, Taiwan) at every 0.50 h interval, until the weight reached a constant weight. The final moisture content of Kyoho skin samples was estimated (±0.20%) using a moisture analyzer. For quality analysis, dried Kyoho skin samples were ground to a fine powder using a laboratory grinder (Yu Chi Machinery Co., Ltd., Taichung, Taiwan) and sieved by certified standard sieves (US Standard Sieve Series, ASTME No. 20 and Tyler Standard Sieve Series—20 Mesh). The dried Kyoho skin samples (Figure 1) were stored in airtight, plastic zip-lock re-closable packing bags (120 × 170 mm) and placed in a humidity controller (RH—50%) at optimum temperature until further analysis, for no longer than 3 days. Bioactive compounds from Kyoho skin were extracted with the acetone: water (4:1 v/v) solvent extraction method according to a previous study [2]. The extracts were stored in air-tight disposable scintillation bottles at 4 ± 1 °C for bioactive compound analysis.

2.4. Modeling of Experimental Drying Kinetics

Experimental drying data were reported in terms of moisture ratio (MR) using Equation (1), according to the previous study conducted on different drying materials [4,13].

$$\text{Moisture ratio (MR)} = \left[\frac{M_t - M_e}{M_o - M_e}\right] \quad (1)$$

However, as stated in the literature, we assumed that the value of equilibrium moisture content was relatively smaller (usually less than M_o) and was considered as zero. Therefore, Equation (1) was modified to Equation (2).

$$\text{Moisture ratio (MR)} = \left[\frac{M_t}{M_o}\right] \quad (2)$$

In Equations (1) and (2), MR is the moisture ratio (dimensionless), M_t is the moisture content at drying time t (h), M_o is the initial moisture content, and M_e is the equilibrium moisture content.

Kyoho skin drying data were fitted to the popularly used five thin layer models, including Lewis, Page, Henderson–Pabis [10], logarithmic [4,14], and two-term empirical model [5], as shown in Table 1. The kinetic parameters of the models for each drying

temperature were calculated by non-linear regression techniques, using the Curve Fitting Toolbox™—MATLAB R2018b (MathWorks®).

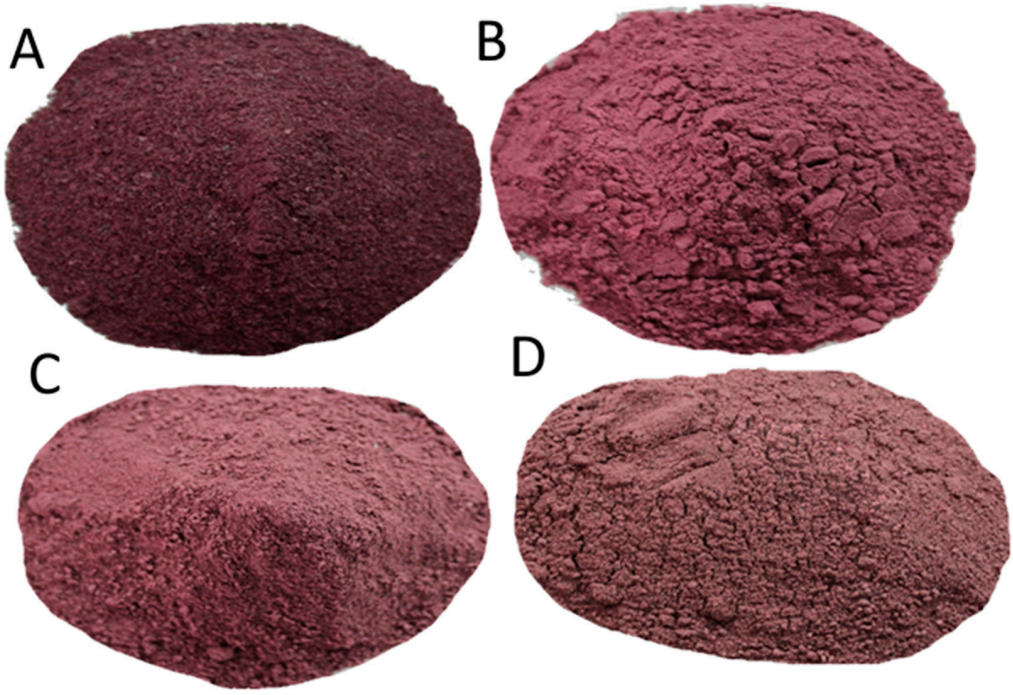

Figure 1. Visual appearance of Kyoho skin powder affected by drying temperature. (**A**) 303.15 K, (**B**) 313.15 K, (**C**) 323.15 K, and (**D**) 333.15 K.

Table 1. Empirical models used to describe the drying kinetics of Kyoho skin [1].

Model	Equation
Lewis	$MR = \frac{M_t - M_e}{M_o - M_e} = (\exp - kt)$
Page	$MR = \frac{M_t - M_e}{M_o - M_e} = \exp(-kt^N)$
Henderson–Pabis	$MR = \frac{M_t - M_e}{M_o - M_e} = a \exp(-kt)$
Logarithmic	$MR = \frac{M_t - M_e}{M_o - M_e} = a \exp(-kt) + c$
Two-term	$MR = \frac{M_t - M_e}{M_o - M_e} = a_1 \exp(-k_1 t) + a_2 \exp(-k_2 t)$

[1] MR represents the moisture ratio, M_t is the moisture at any time t during drying (% dry basis), M_o is the initial moisture content (% dry basis), M_e is the equilibrium moisture content, k (k_1 and k_2) is the drying rate constant, and N, c and a (a_1 and a_2) are parameters in thin layer models.

To determine the most suitable drying model, goodness of fit for each model was performed based on the following statistical parameters: coefficient of determination (R^2), chi-square (χ^2), root-mean-square error (RMSE), according to the study by Younis et al. [4], and sum squared errors (SSE), according to a study undertaken by Vega-Gálvez et al. [15]. Higher R^2 values and lower χ^2, RMSE, and SSE values provided the better fit of the model. Statistical parameters were calculated as shown in the following Equations (3)–(6):

$$\text{Coefficient of determination } \left(R^2\right) = \left[\frac{\sum\left(MR_{prd} - \sum MR_{exp}\right)^2}{\sum\left(\overline{MR}_{prd} - \sum MR_{exp}\right)^2}\right] \quad (3)$$

$$\because \overline{MR}_{prd} = \text{the average predicted moisture ratio}$$

$$\text{Chi} - \text{square}\left(\chi^2\right) = \left[\frac{\sum\left(\text{MR}_{\text{exp}} - \text{MR}_{\text{prd}}\right)^2}{\text{N} - \text{n}}\right] \quad (4)$$

$$\text{Root} - \text{mean} - \text{square error (RMSE)} = \sqrt{\left[\frac{\sum\left(\text{MR}_{\text{prd}} - \text{MR}_{\text{exp}}\right)^2}{\text{N}}\right]} \quad (5)$$

$$\text{Sum squared errors (SSE)} = \left[\frac{1}{\text{N}}\sum\left(\text{MR}_{\text{exp}} - \text{MR}_{\text{prd}}\right)^2\right] \quad (6)$$

where MR_{exp} and MR_{prd} are the experimental and predicted dimensionless moisture ratios, respectively, N is the number of experimental points, and n is the number of constant values of the models.

2.5. Effective Moisture Diffusivity (D_{eff}) and Activation Energy (E_a)

Drying experiments of Kyoho skin were conducted in a conventional oven at four constant drying temperatures. Kyoho skin samples were placed uniformly in rectangular stainless-steel trays and dried in the hot air oven at the drying temperatures. The effective moisture diffusivity was used to describe the drying characteristics of Kyoho skin by assuming the slab geometry of samples according to Fick's law of diffusion [16], as shown in Equation (7).

$$\text{Moisture ratio (MR)} = \left[\frac{\text{M}_\text{t} - \text{M}_\text{e}}{\text{M}_\text{o} - \text{M}_\text{e}}\right] = \left[\frac{8}{\pi^2}\sum\nolimits_{\text{n}-1}^{\infty}\frac{1}{(2\text{n}+1)^2}\exp\left(\frac{-(2\text{n}+1)\pi^2\text{D}_{\text{eff}}\text{t}}{4\text{H}^2}\right)\right] \quad (7)$$

However, Equation (7) was simplified for long drying periods and the values of M_e are relatively small. Therefore, the modified form of Equation (7) is presented as Equation (8), as follows:

$$\ln\text{Moisture ration (MR)} = \left[\frac{\text{M}_\text{t}}{\text{M}_\text{o}}\right] = \ln\left[\frac{8}{\pi^2} - \left(\frac{\pi^2\text{D}_{\text{eff}}\text{t}}{4\text{H}^2}\right)\right] \quad (8)$$

In Equations (7) and (8), M_t is the moisture content (% dry basis) at a specific time interval, M_o is the initial moisture content (% dry basis), M_e is the equilibrium moisture content, H is the spatial dimension of the Kyoho skin (m), n is a positive integer, D_{eff} is the effective moisture diffusivity (m^2/s), and t is the drying time (h).

A straight line with a slope was constructed from the data of the experimental moisture ratio (ln MR) and drying time (h) according to the study by Aregbesola et al. [16], as shown in Equation (9).

$$\text{Slope} = \ln\left[\frac{\pi^2\text{D}_{\text{eff}}}{4\text{H}^2}\right] \quad (9)$$

Temperature reliance of the effective moisture diffusivity was described by an Arrhenius-type equation according to Roberts et al. [10] and Aregbesola et al. [16], as shown in Equation (10).

$$\text{D}_{\text{eff}} = \text{D}_\text{o}\exp\left[\frac{-\text{E}_\text{a}}{\text{RT}}\right] \quad (10)$$

In Equation (10), D_o is the Arrhenius factor (m^2/s), E_a is the activation energy (kJ/mol), R is the universal gas constant (kJ/mol K), and T is the drying temperature (K).

In order to obtain values of activation energy (E_a), plots between ln (D_{eff}) vs. the reciprocal of the drying temperature (1/T, K) were constructed.

2.6. Color Characterization

The color for all dried Kyoho samples (powder form) was obtained using a colorimeter, calibrated by a standardized white tile [17]. Measurements were made using the D65

illuminant and 2° standard observer. The total color difference (ΔE) was calculated and compared with the Kyoho skin powder that was dried at room temperature (reference), as shown in the following Equation (11).

$$\Delta E = \left[\Delta L^2 + \Delta a^2 + \Delta b^2\right]^{\frac{1}{2}} \quad (11)$$

Here, ΔL = the difference in lightness, Δa = the difference in intensity of the red color, and Δb = the difference in intensity of the yellow color.

2.7. Rehydration

Rehydration of Kyoho skin powder was determined by the method described by Adiletta et al. [5]. Briefly, Kyoho skin powder samples (1 g) were rehydrated in distilled water at 25 °C for 5 h. The ratio of Kyoho skin powder and water volume was maintained as 1:30 [5]. The rehydration of Kyoho skin powder was calculated as g/g of the sample from the water observed by the sample before and after rehydration, according to the following Equation (12).

$$\text{Weight gain } (g/g) = \left[\frac{\text{Weight of rehydrated sample } (g) - \text{Dry weight of sample } (g)}{\text{Dry weight of sample } (g)}\right] \quad (12)$$

2.8. Total Phenolic and Flavonoid Contents

The total phenolic content (TPC) in the Kyoho skin affected by drying temperature was determined by the Folin–Ciocalteu colorimetric method according to a study by Cruz et al. [12]. The experiment was calibrated using gallic acid as the standard and the results were expressed as mg of gallic acid equivalents (mg GAE/g of sample) per g of sample (dry weight). The amount of total flavonoid content (TFC) in the Kyoho skin affected by drying temperature was estimated using the method of Sridhar and Charles [18]. Total flavonoid content was calculated using quercetin as the standard and expressed as mg of quercetin equivalent (mg QE/g of sample) per g of sample (dry weight).

2.9. Antioxidant Activity

For DPPH, the free radical scavenging activity of Kyoho skin at different drying temperatures was estimated based on the study conducted by Katalinić et al. [19]. A DPPH$^{\bullet}$ working solution was prepared by dissolving DPPH$^{\bullet}$ stock solution (4 mg DPPH in 100 mL of ethanol) to obtain the initial absorbance of 1.07 ± 0.01 at 517 nm. Briefly, 100 µL of the Kyoho skin samples was added to 3 mL of DPPH$^{\bullet}$ ethanolic solution. The mixtures were vigorously shaken and left to stand for 60 min in the dark at room temperature. The changes in absorbance were measured at 517 nm against a blank of ethanol using a DU® 730 UV/Vis spectrophotometer. A decrease in absorbance of the reaction mixture indicates a higher inhibition percentage of free radicals. The ability of Kyoho skin samples to inhibit DPPH$^{\bullet}$ was calculated using the following Equation (13).

$$\%\ \text{Inhibition} = \left[\left(\frac{A_{control} - A_{sample}}{A_{control}}\right) \times 100\right] \quad (13)$$

where $A_{control}$ is the absorbance of the control without the test sample, and $A_{extract}$ is the absorbance of the Kyoho skin extracts at the different drying temperatures.

ABTS radical scavenging activity was estimated using the method described by Doshi et al. [20], with some modifications. ABTS$^{\bullet+}$ (ABTS radical cation) was prepared by mixing ABTS aqueous solution (7 mM) and potassium persulfate (2.45 mM) in distilled water and incubated for 12–14 h in the dark at room temperature. The ABTS solution was diluted (28 times) with methanol to obtain the absorbance of 0.72 ± 0.05 at 734 nm. Then, 1950 µL of ABTS$^{\bullet+}$ solution was added to 50 µL of the standard (ascorbic acid) and Kyoho skin samples dried at different temperatures. The mixtures were then vortexed

and absorbance was recorded at 734 nm after 10 min. The radical scavenging activity was calculated as% inhibition by Kyoho skin samples using Equation (13).

2.10. Correlation Analysis

Pearson's correlation is the widely employed method to determine the linear association between any two variables using Pearson's model (two-tailed test of significance). Pearson's correlation coefficient (r) between phenolics and antioxidant activities affected by drying temperature were calculated according to IBM® SPSS® Statistics version 22.0 (2013), as shown in the following Equation (14):

$$r_{xy} = \left[\frac{C_{xy}}{\sqrt{C_{xx} C_{yy}}} \right] \quad (14)$$

where C_{xy} = the covariance of x and y, C_{xx} = the variance of x, and C_{yy} = the variance of y.

2.11. Statistical Analysis

The data were expressed as the mean ± standard deviation (SD) of three experimental measurements. Multivariate analysis of variance (MANOVA) was used at the $p < 0.05$ level of significance using Duncan's multiple range test for comparison of mean differences affected by drying temperature. All statistical and correlation analyses were performed using IBM® SPSS® Statistics version 22.0 (Armonk, NY, USA). Model fitting was performed in order to fit experimental data using the Curve Fitting Toolbox™—MATLAB R2018b (MathWorks®, The MathWorks, Inc., Natick, MA, USA).

3. Results and Discussion

3.1. General

The initial moisture content of Kyoho skin samples was observed as 87.51 ± 0.05% (wet basis). The overall mean weight of Kyoho skin was recorded as 1.50 ± 0.43 g per fruit.

3.2. Drying Behavior of Kyoho Skin

The changes in moisture ratio with drying time for different drying temperatures (303.15–333.15 K) are illustrated in Figure 2A. The experimental results clearly demonstrated that the moisture ratio exponentially decreased with an increase in drying temperature (303.15–333.15 K). The results revealed that the hot air oven drying method considerably decreased drying time as a function of drying temperature. For example, the total drying time to reach the constant weight (equilibrium moisture content of 6.50 ± 2.77%) for the Kyoho skin samples ranged from 3 (333.15 K) to 8.50 h (303.15 K). This observation further confirmed that the Kyoho skin samples reached a constant weight in less drying time (3 h) at a higher temperature, whereas the same sample reported a higher drying time (8.50 h) to reach a constant weight at the temperature of 303.15 K. Therefore, the observed differences in drying times might be due to the drying temperature that affected the drying mechanism of Kyoho skin. These results are similar to a study that reported faster dehydration rates with higher temperatures [10,21]. Generally, the drying process of any material is directly linked to the characteristics of the drying material. We used Kyoho skin, which contains an epidermis and six to ten superimposed layers of small thick-walled cells [22,23]. The amorphous layer (wax), present in the outer skin epidermis, was an effective factor in the drying process of Kyoho skin, which might have affected the drying rate. The changing trends in the drying rates with moisture content accelerated the falling rate period without a constant rate period at all investigated temperatures. Thus, the complete drying mechanism of Kyoho skin occurred in the falling rate period, which led to the assumption that the diffusion mechanism was the main dominant parameter that controlled moisture transfer in Kyoho skin samples, as observed for many agricultural products [10,24]. Overall, these results corroborated previous findings on the drying mechanism of many agricultural products [4,25]. However, only a few studies have focused on the drying mechanism of

red grape skin and have documented the absence of the constant rate period [12]. These results concluded that drying temperature had an effect on the drying rate, which could be considered for the development of Kyoho skin value-added products.

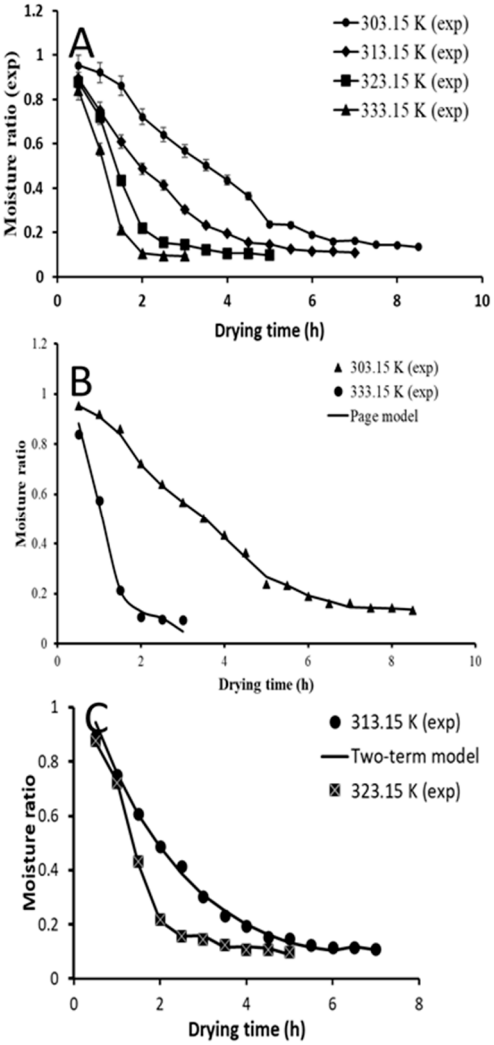

Figure 2. Relationship between moisture ratio (experimental) vs. drying time of Kyoho skin (**A**), Page model at 303.15 K and 333.15 K (**B**), and (**C**) two-term model at 313.15 K and 323.15 K. Error bar represents the standard deviation of the mean.

3.3. Mathematical Modeling and Goodness of Fit

Drying kinetics of Kyoho skin samples were evaluated based on the experimental moisture ratio at different drying temperatures and fitted with drying time using five drying models (Table 2). As expected, the values of the drying constant (k) increased with increases in drying temperature, except for the two-term model, which led to the assumption of the existence of a relationship between k values and drying temperature. Moreover, k values might be related to the diffusion coefficient, which represented the relationship between k and drying temperatures and was the basis for predicted changes in k values as a function of temperature. These results were in agreement with a study that reported decreased and/or increased drying constants as a function of drying temperature [26,27]. On the other hand, other model parameters (N, a, and c) fluctuated among the investigated temperatures. Thus, we failed to determine any definite trend of the parameters in our study, but further observations proposed that these parameters might be useful as model coefficients to minimize statistical parameters (exclusively χ^2) in a mathematical model. The lack of a definite trend of parameters (N, a, and c) in our study was in agreement with the study

conducted by Aregbesola et al. [16]. More recently, computational modeling has emerged as a novel modeling technique, which can be used to understand the mathematical model coefficients [28].

The coefficient of determination (Table 2) and the goodness of fit statistical results (Table 3) were used to determine the most suitable model that could predict the drying behavior of Kyoho skins. The best suitable models were selected based on the highest coefficient of determination (R^2) and the lowest goodness of fit statistical parameters (χ^2, RMSE, and SSE), as stated in the literature available for modeling studies on agricultural products [4,10,15]. The five models were interpreted based on the R^2 values that fit the experimental data. Overall, higher R^2 values (closer to 1) were considered to evaluate the prediction of the models. A study by Doymaz [29] reported that the R^2 values > 0.90 are an acceptable level to explain the prediction phenomenon of the model. The goodness of fit statistical analysis data are presented in Table 3. The values of χ^2, RMSE, and SSE were observed in the range of 0.0220–4.3924, 0.0504–0.1793, and 0.0154–0.1096, respectively, at all investigated temperatures. For all examined models, the Page model reported the lowest goodness of fit at the temperatures of 303.15 K (R^2 = 0.9815, χ^2 = 0.0239, RMSE = 0.0504, and SSE = 0.0381) and 333.15 K (R^2 = 0.9685, χ^2 = 0.0220, RMSE = 0.0622, and SSE = 0.0154), whereas the two-term model presented a better fit based on the experimental moisture ratio at the temperatures of 313.15 K (R^2 = 0.9639, χ^2 = 0.0543, RMSE = 0.0600, and SSE = 0.0360) and 323.15 K (R^2 = 0.9737, χ^2 = 0.0383, RMSE = 0.0564, and SSE = 0.0191). These higher values of R^2 and lower values of the goodness of fit of selected models might be ascribed to the small proportion of the variance in the experimental moisture ratio that was predicted from the drying time. Based on the statistical analysis of our study, Page and two-term models were the best models for the prediction of the drying mechanism using Kyoho skin at the investigated temperatures. We further presented the graphical correlation between the experimental and the predicted moisture ratio for the selected models (Figure 2B,C). These findings agreed with previous works on drying models of grapes [30,31], grape seeds [10], grape pomace [32], and other agriculture products, such as onion slices [33], cassava chips [34], and banana peels [35]. Therefore, Page and two-term models were found to be the most effective at predicting the drying process of Kyoho skins and the most useful for the measurement of parameters in designing specialized drying (grape skin) equipment.

3.4. Effective Moisture Diffusivity (D_{eff}) and Activation Energy (E_a)

The effective moisture diffusivity for each drying temperature is summarized in Figure 3. The mean values of D_{eff} were computed based on the constructed slope and the results of the calculated D_{eff} for drying temperatures are highlighted in Table 4. The D_{eff} values were observed as positive values in the range of $2.87 \pm 1.37 \times 10^{-8}$ to $9.82 \pm 1.74 \times 10^{-8}$ m^2/s. The calculated D_{eff} data showed that the D_{eff} increased with a gradual increase in drying temperature; for instance, at 303.15 K, the D_{eff} value was observed as $2.87 \pm 1.37 \times 10^{-8}$ m^2/s, and the gradual increase in temperature (approximately 1.0329 times) presented 1.28-times higher D_{eff} than the initial D_{eff} values. These results confirmed the association between drying temperature and D_{eff}. The increased trend of D_{eff} values could be attributed to the drying kinetics and transfer of moisture during the drying process. These results are consistent with the drying study of grape by-products that explained the proportional relationship between D_{eff} and temperature [36]. The D_{eff} values observed in our study were slightly higher than those for grape by-products reported by Celma et al. [36]. However, these discrepancies might be related to initial moisture content, changes in sample temperature, and negligible shrinkage of the sample [4]. There are, however, other possible explanations, including drying conditions that highlighted the differences in D_{eff} values, where sun drying (open) reported higher D_{eff} than shade drying [37] and thickness of the skin might be influenced in the drying process.

Table 2. Kinetic parameters and coefficient of determination (R^2) of the drying models for Kyoho skin [1].

Temperature (K)	Lewis		Page			Henderson–Pabis			Logarithmic				Two-Term				
	k	R^2	k	N	R^2	k	a	R^2	k	a	c	R^2	k_1	k_2	a_1	a_2	R^2
303.15	0.2311 ± 0.01	0.9236	0.1171 ± 0.05	1.4720 ± 0.17	0.9815	0.2827 ± 0.10	1.2060 ± 0.71	0.9640	0.2441 ± 0.08	1.2470 ± 1.05	−0.0715 ± 0.03	0.9659	−2.2010 ± 0.71	0.2896 ± 0.05	2.414 ± 0.21 × 10^{-10}	1.2310 ± 0.31	0.9643
313.15	0.3685 ± 0.10	0.9395	0.2815 ± 0.08	1.2520 ± 0.13	0.9577	0.4270 ± 0.19	1.1560 ± 0.22	0.9582	0.4686 ± 0.16	1.1520 ± 0.08	0.0320 ± 0.004	0.9595	−1.0570 ± 0.04	1.1820 ± 0.54	4.1050 ± 1.48 × 10^{-5}	1.1820 ± 0.076	0.9639
323.15	0.5615 ± 0.18	0.9158	0.4438 ± 0.10	1.3800 ± 0.51	0.9479	0.7283 ± 0.31	1.306 ± 1.05	0.9606	0.8887 ± 0.28	1.3500 ± 0.34	0.0601 ± 0.04	0.9692	0.7949 ± 0.33	−0.8514 ± 0.08	1.372 ± 0.22	0.0011 ± 0.001	0.9737
333.15	0.7772 ± 0.05	0.8741	0.6230 ± 0.20	1.7810 ± 0.66	0.9685	1.1430 ± 0.90	1.5290 ± 0.07	0.9667	−0.0014 ± 0.01	−293.50 ± 147.08	294.30 ± 125.70	0.8040	0.3829 ± 0.17	0.4025 ± 0.16	−19.28 ± 2.57	20.45 ± 4.08	0.9378

[1] Data are expressed as average ± standard deviation of three replicates (n = 3). T is temperature in Kelvin, k (k_1 and k_2) is drying rate constant, N, a (a_1 and a_2), and c are the parameters of the drying models. R^2 is determined by the average values (n = 3) of kinetic parameters.

Table 3. Average values of goodness of fit criteria for drying models for Kyoho skin [1].

Temperature (K)	Lewis			Page			Henderson–Pabis			Logarithmic			Two-Term		
	χ^2	RMSE	SSE	χ^2	RMSE	SSE	χ^2	RMSE	SSE	χ^2	RMSE	SSE	χ^2	RMSE	SSE
303.15	0.0827	0.0827	0.1096	0.0239	0.0504	0.0381	0.0586	0.0586	0.0519	0.0590	0.0591	0.0488	0.0627	0.0627	0.0512
313.15	0.0614	0.0681	0.0664	0.1009	0.0603	0.0422	0.0545	0.0607	0.0417	4.3924	0.0606	0.0403	0.0543	0.0600	0.0360
323.15	0.0618	0.0824	0.0611	0.0808	0.0688	0.0378	0.0436	0.0598	0.0286	0.0573	0.0565	0.0223	0.0383	0.0564	0.0191
333.15	0.0622	0.1113	0.0619	0.0220	0.0622	0.0154	0.0330	0.0640	0.0164	0.1889	0.1793	0.0964	0.0485	0.1236	0.0305

[1] Chi-square (χ^2), root-mean-square errors (RMSE), and sum of square errors (SSE). The goodness of fit for each model was calculated based on the average values (n = 3) of predicted and experimental data.

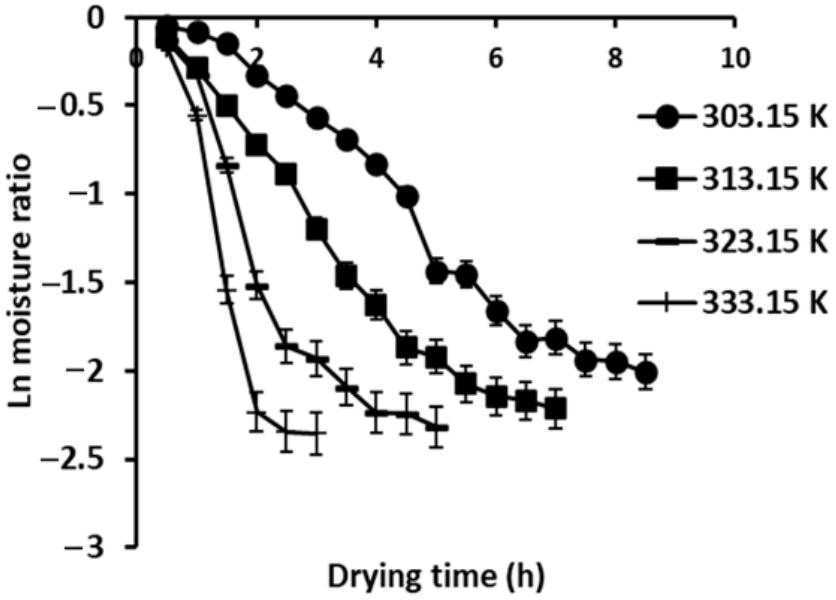

Figure 3. Plot of the ln moisture ratio (ln MR) *versus* drying time (h) of Kyoho skin. Error bar represents the standard deviation of the mean.

Table 4. Effective moisture diffusivity and activation energy for Kyoho skin at different drying temperatures [1].

Temperature (K)	D_{eff} (m^2/s) × 10^{-8}	E_a(kJ/mol)	R^2
303.15	2.87 ± 1.37 [a]	33.78 ± 1.06	0.9355
313.15	3.60 ± 1.08 [b]		0.9852
323.15	5.15 ± 1.00 [c]		0.9733
333.15	9.82 ± 1.74 [d]		0.9804

[1] Data are expressed as average ± standard deviation of three replicates. The values not bearing common superscripts ([a–d]) within the same column differ significantly ($p < 0.001$). D_{eff} is effective moisture diffusivity, E_a is activation energy and R^2 is coefficient of determination calculated by the average values ($n = 3$).

The natural logarithmic form of D_{eff} as a function of the reciprocal temperature for each drying temperature was plotted to construct a straight line ((R^2 = 0.9355) and is illustrated in Figure 4. The constructed straight line presented a negative slope, which indicated a decrease in D_{eff} of the sample with an increase in the reciprocal of absolute temperature during the drying process. The value of activation energy (E_a) was calculated based on the constructed slope and the values are summarized in Table 4. The E_a was observed as 33.78 ± 1.06 kJ/mol in the temperature range of 303.15–333.15 K. The results indicated that 33.78 ± 1.06 kJ/mol of energy must be provided to Kyoho skin samples during the drying process in a hot air oven to effect moisture transfer. These values agreed with the studies proposed earlier for different agriculture products, including crushed Hass avocado seeds [25] and pistachio [16]. Moreover, similar results were also recorded in a previous study on hull-less seed pumpkin using a different drying methods [38]. The results of our study concluded that the internal mass transfer in Kyoho skins, exclusively governed by the diffusion mechanism and the linear relationship of D_{eff}, could be described by an Arrhenius equation.

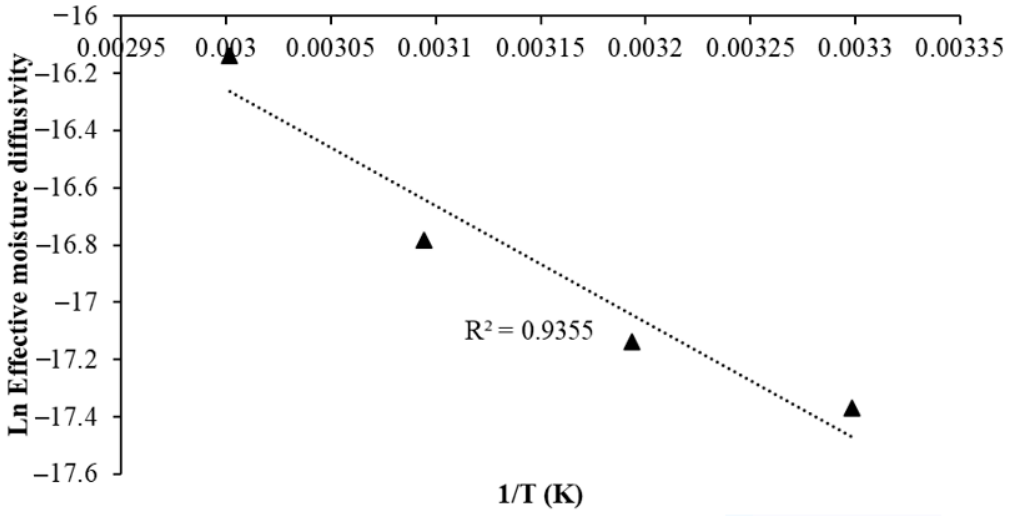

Figure 4. Logarithmic of effective moisture diffusivity versus absolute temperature (1/T, K) of Kyoho skin.

3.5. Color

The effects of drying temperature on the surface color parameters of Kyoho skin powder are shown in Table 5. Statistical analysis between the surface color parameters and drying temperature of Kyoho skin powder indicated significant difference ($F_{(12, 13.52)}$ = 174.03, ($p < 0.001$, Wilk's $\Lambda = 0.02 \times 10^{-4}$)). Values of the lightness (L) and green/red colors (a) were significantly increased between 24.67 ± 0.38 to 30.41 ± 0.15 and 18.28 ± 0.11 to 21.53 ± 0.17, respectively, at all investigated temperatures, whereas blue/yellow (b) significantly fluctuated between 2.71 ± 0.01 and 2.06 ± 0.02. The fluctuations in b values were attributed to alterations in non-enzymatic reactions [39]. Generally, the initial purple-black color of Kyoho grape skins significantly changed to a whitish-purple color with increases in drying temperature (Figure 1). Redness significantly increased, except at 313.15 K, which was reported as 16.62 ± 0.05. The effect of drying temperature on the total color difference (ΔE) of Kyoho skin powder is presented in Table 5. Application of statistical analysis revealed that drying temperature had a significant effect on the ΔE of Kyoho skin powder, which further supported the visual appearance of Kyoho skin powder at different temperatures (Figure 1). The lowest (1.45 ± 0.23) and highest (5.66 ± 0.06) values of ΔE were observed at 303.15 K and 333.15 K, respectively. Color changes in Kyoho powder might be attributed to the effect of drying temperature on heat sensitive compounds, the destruction of color pigments, or chemically reactive compounds (sugars and amino acids) in samples [39,40]. Similar color change observations were recorded in fruits as a function of drying temperature [41–43]. The findings from this study suggested the importance of drying temperature in preserving the color attributes of the Kyoho skin for the evaluation of consumer acceptance.

Table 5. Effect of drying temperature on color parameters of Kyoho skin powder [1].

Temperature (K)	L	a	b	ΔE
303.15	24.67 ± 0.38 [a]	18.28 ± 0.11 [b]	2.71 ± 0.01 [b]	1.45 ±0.23 [a]
313.15	26.28 ± 0.27 [b]	16.62 ± 0.05 [a]	3.49 ± 0.05 [d]	3.22 ± 0.04 [b]
323.15	28.74 ± 0.78 [c]	20.20 ± 0.47 [c]	3.05 ± 0.03 [c]	3.83 ± 0.67 [b]
333.15	30.41 ± 0.15 [d]	21.53 ± 0.17 [d]	2.06 ± 0.02 [a]	5.66 ± 0.06 [c]

[1] The results are given as the mean and standard deviation of three independent determinations (n = 3). The values not bearing common superscripts ([a–d]) within the same column differ significantly ($F_{(12, 13.52)}$ = 174.03, ($p < 0.001$, Wilk's $\Lambda = 0.02 \times 10^{-4}$)). L = black to white (0 to 100), a = green (−a) to red (+a), b = blue (−b) to yellow (+b), and ΔE = the total color difference.

3.6. Rehydration

The average values of rehydration, as affected by drying temperature, are presented in Figure 5. The results demonstrated that the levels of rehydration of Kyoho skin powder, as affected by drying temperature, differed significantly ($p < 0.05$). The mean values of rehydration ranged from 2.92 to 1.57 g/g for all investigated temperatures. The highest mean value of rehydration (2.92 g/g) was observed at a lower drying temperature of 303.15 K, whereas the lowest rehydration (1.57 g/g) was recorded at a higher drying temperature of 333.15 K. Rehydration decreased proportionally with an increase in drying temperatures from 303.15 to 333.15 K, which was attributed to the longer drying period at the initial temperature (303.15 K) and shorter drying period at a higher temperature (333.15 K). The rehydration mechanism in Kyoho skin powder was mostly affected by drying temperature. However, there are other factors, including drying method, pretreatments, physical structure, and chemical composition [5]. The behavior of water absorption of Kyoho skin powder was observed as almost linear, with $R^2 = 0.9607$ (Figure 5). Several studies suggested that longer drying times may collapse and/or modify the structural integrity [5,44] of the cells, allowing water to enter the cell. These results confirmed that the effects of drying temperature on rehydration were useful to understand the water absorption mechanism of powders in the formulation of food products and emulsions.

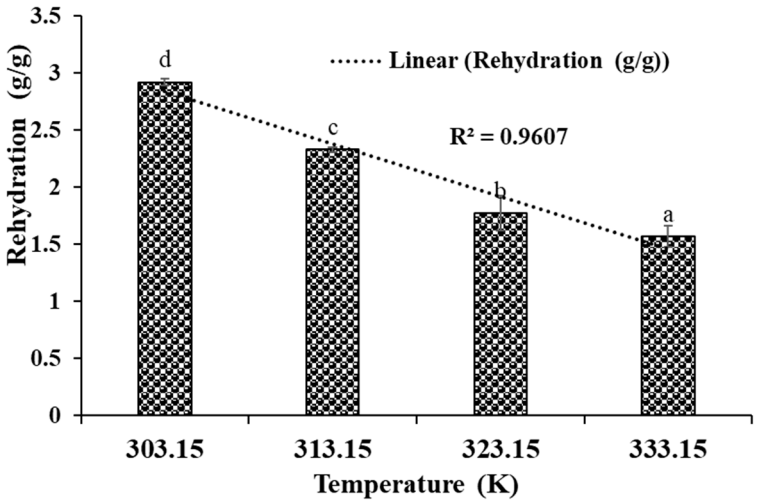

Figure 5. Effect of drying temperature on rehydration of Kyoho skin powder. Error bar represents the standard deviation of the mean and different letters (a to d) indicate significant difference ($p < 0.05$).

3.7. Total Phenolic and Flavonoid Contents

The results demonstrated that the levels of total phenolic and flavonoid compounds in Kyoho skin differed significantly ($p < 0.01$) as a function of drying temperature (Table 6). The phenolic and flavonoid contents ranged from 0.37 ± 0.04–0.23 ± 0.03 mg GAE/g to 2.36 ± 0.78–1.05 ± 0.33 mg QE/g at all investigated temperatures. The highest total phenolic content (TPC) was found to be 0.37 ± 0.04 mg GAE/g at 303.15 K, whereas the lowest content (0.23 ± 0.03 mg GAE/g) was observed at 333.15 K. TPC of the Kyoho skin decreased in the following order: 0.37 ± 0.04 mg GAE/g (303.15 K), 0.33 ± 0.03 mg GAE/g (313.15 K), 0.30 ± 0.04 mg GAE/g (323.15 K), and 0.23 ± 0.03 mg GAE/g (333.15 K). This trend in Kyoho skin demonstrated a decrease in TPC of the sample with an increase in drying temperature. Similar conclusions were reported in a study by Alara et al. [37], in which drying temperature affected the TPC of plants. In addition, total flavonoid content (TFC) followed a similar trend as TPC, as affected by drying temperature (Table 6). It was interesting to note that the TPC was less degraded (1.608-stimes) than TFC (2.240-times) at 303.15 to 333.15 K. These results were likely to be related to the lower sensitivity of TFC compared to TPC present in Kyoho skin. Generally, phenolic compounds and pigments (i.e., carotenoids) are heat-sensitive and may undergo loss in functional properties during

heat treatment or drying [45]. The decrease in TPC during drying was likely to be related to the binding of polyphenols with proteins and/or structural changes in polyphenols, which prevented or reduced their isolation during the extraction process [39]. These results agreed with a report on the effects of drying temperature on TPC of grape pomace peel [46], which concluded that drying of grape peel at 100 °C and 140 °C significantly decreases phenolic content. Similarly, Kumar, et al. [47] and Lim and Eom [43] reported the variations in total phenolic and flavonoid contents of dropped *Citrus sinensis* L. Osbeck fruits and thermally dried flesh and peel of astringent persimmon fruit, respectively. A key study by Çoklar and Akbulut [48] stated that the degradation of phenolic compounds was explained by the thermal degradation and/or oxidation of phenolic compounds. Another study by Pedroza et al. [49] demonstrated that the cell structure and/or composition could protect phenolic compounds. The damage to cell structure and/or composition during drying might be impacted by the degradation of phenolics in Kyoho skin.

Table 6. Total phenolic and flavonoid contents of Kyoho skin as affected by drying temperature [1].

Temperature (K)	TPC (mg GAE/g)	TFC (mg QE/g)
303.15	0.37 ± 0.04^{b}	2.36 ± 0.78^{b}
313.15	0.33 ± 0.03^{b}	2.19 ± 0.74^{ab}
323.15	0.30 ± 0.04^{ab}	1.35 ± 0.43^{ab}
333.15	0.23 ± 0.03^{a}	1.05 ± 0.33^{a}

[1] The results are given as the mean and standard deviation of three independent determinations (n = 3). The values not bearing common superscripts ($^{a–b}$) within the same column differ significantly ($F_{(6, 14)} = 5.10$, ($p < 0.01$, Wilk's $\Lambda = 0.09$)). TPC = total phenolic content, TFC = total flavonoid content, GAE = gallic acid equivalent, and QE = quercetin equivalent.

3.8. Antioxidant Activity (DPPH• and ABTS• Scavenging)

Antioxidant activity of Kyoho skin was significantly affected ($F_{(6, 14)} = 380.26$, ($p < 0.001$, Wilk's $\Lambda = 0.37 \times 10^{-4}$)) as a function of drying temperature (Table 7). For instance, DPPH• scavenging activity of Kyoho skin ranged from 93.06 ± 0.48 to 73.31 ± 1.13% as a function of drying temperature. Initially, at 303.15 K, Kyoho skin showed a higher inhibition efficacy, which was found to be 93.06 ± 0.48%. However, the% inhibition capacity of DPPH• decreased proportionally with an increase in drying temperature. In addition, the effects of drying temperature on DPPH• scavenging activity were similar between TPC and TFC, which confirmed the dependence of antioxidant activity on TPC and TFC. Similarly, ABTS• scavenging activity varied in our study and ranged from 96.69 ± 1.17 to 11.87 ± 1.89% in the range of 303.15 to 333.15 K (Table 7), which further confirmed the effect of drying temperature on Kyoho skin. The results indicated that ABTS• scavenging activity followed the same trend as DPPH• scavenging activity, TPC, and TFC. Therefore, evaluation of antioxidant activity using both DPPH• and ABTS• methods reported a decreased trend as drying temperature increased, which demonstrated the significant effect of drying temperature on Kyoho skin. Our findings broadly supported the work of other related studies on antioxidant activities as affected by drying temperature [5,43,50]. This was an important finding in understanding the effect of drying temperature on the antioxidant activity of Kyoho skin. Therefore, we recommended using low drying temperatures to preserve the antioxidant potential of the Kyoho skin for industrial food applications.

Table 7. Antioxidant activities of Kyoho skin extracts as affected by drying temperature [1].

Temperature (K)	Antioxidant Activity	
	DPPH• Method	**ABTS• Method**
303.15	93.06 ± 0.48 [c]	96.69 ± 1.17 [d]
313.15	84.57 ± 0.19 [b]	82.24 ± 0.94 [c]
323.15	85.38 ± 0.40 [b]	43.14 ± 2.02 [b]
333.15	73.31 ± 1.13 [a]	11.87 ± 1.89 [a]

[1] The results are given as the mean and standard deviation of three independent determinations ($n = 3$). The values not bearing common superscripts ([a–d]) within the same column differ significantly ($F_{(6, 14)} = 380.26$, ($p < 0.001$, Wilk's $\Lambda = 0.37 \times 10^{-4}$)). DPPH• = 2,2 diphenyl-1-picrylhydrazyl radical activity and ABTS• = 2,2′-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid).

3.9. Correlation Analysis

In order to determine the association between phenolics and antioxidant activities, we employed Pearson's linear correlation between the antioxidant capacities and the phenolic substances (TPC and TFC), as affected by drying temperature (Table 8). The drying temperatures of 303.15 K and 313.15 K followed similar correlation tendencies, but correlation values differed between the phenolics and antioxidant activities of Kyoho skin. For example, higher linear correlations between TPC and TFC ($r \geq 0.95$) were observed at 303.15 K and 313.15 K, which were statistically insignificant at the 0.05 level. Antioxidant activity measured by the ABTS• method negatively correlated with TPC, TFC, and DPPH• at 303.15 K and 313.15 K. On the other hand, a significant correlation was reported between TPC and TFC at 323.15 K and 333.15 K, which was statistically significant at the 0.01 level (two-tailed). Interestingly, antioxidant activity measured by the ABTS• method positively correlated with TPC and TFC (r = 0.50) at 323.15 K, whereas the same method reported a negative correlation with TPC and TFC (−0.82) at 333.15 K. These differences in correlations were attributed to the drying temperature that disrupted the association between the phenolic and antioxidant activities of Kyoho skin [6]. There are, however, other possible explanations, including the nature of antioxidants and oxygen, and other processing factors [39] that might have contributed to these correlations. The findings reported here demonstrated that the quantitative estimations of polyphenols and antioxidant capacities were affected by drying temperature, which could be considered when drying the Kyoho skin for phenolic extraction and for other functional food formulations.

Table 8. Correlation analysis between phenolics, flavonoids, and antioxidant activities of Kyoho skin as affected by drying temperature [1].

Parameter	303.15 K				313.15 K			
	TPC	**TFC**	**DPPH•**	**ABTS•**	**TPC**	**TFC**	**DPPH•**	**ABTS•**
TPC	1	0.95	0.94	−0.33	1	0.99	0.53	−1 *
TFC		1	0.79	−0.61		1	0.42	−0.99
DPPH•			1	−0.01			1	−0.51
ABTS•				1				1
Parameter	**323.15 K**				**333.15 K**			
	TPC	**TFC**	**DPPH•**	**ABTS•**	**TPC**	**TFC**	**DPPH•**	**ABTS•**
TPC	1	1 **	0.50	0.07	1	1 **	-0.82	−0.93
TFC		1	0.50	0.08		1	-0.82	−0.93
DPPH•			1	−0.81			1	0.97
ABTS•				1				1

[1] TPC = total phenolic content, TFC = total flavonoid content, DPPH• = DPPH• scavenging activity, and ABTS• = ABTS• scavenging activity. ** Correlation is significant at the 0.01 level (2-tailed) and * correlation is significant at the 0.05 level (2-tailed).

4. Conclusions

The drying process of Kyoho skin occurred in the falling rate period, which meant that the water removal process from Kyoho skins was governed by the diffusion mechanism. Page and two-term models were the most suitable models for the prediction of Kyoho skin drying behavior at 303.15 K and 333.15 K and 313.15 K and 323.15 K, respectively. The E_a of the samples was 33.78 ± 1.06 kJ/mol, with varied color change that was affected by the drying temperature. Rehydration decreased with an increase in drying temperature. TPC, TFC, and their antioxidant activities reported decreasing trends as the drying temperature increased, which demonstrated the effect of drying temperature on the loss of bioactive potential. Moreover, variations in the correlation analysis were described as a function of drying temperature. Thus, we concluded that drying temperature should be considered in order to preserve the bioactive potential of Kyoho skin when developing Kyoho skin-based functional foods.

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