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Thermal Degradation and Thermodynamic Properties of Ascorbic Acid, Total Phenolic Content and Antioxidant Activity of Convective Dried Kiwi Fruits

Fadime Begüm Tepe[1,a,*]

[1]*Department of Food Technology, Şebinkarahisar Vocational School of Technical Science, Giresun University, 28400 Giresun, Türkiye*
[*]*Corresponding author*

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ABSTRACT

In the current study, thermal degradation and thermodynamic properties of ascorbic acid, total phenolic content, and antioxidant activity of convective dried kiwi fruits were investigated. To determine kinetic model describing thermal degradation of these parameters, zero order, first order, fractional conversion and Weibull model were used. Weibull model gave the best fitting to thermal degradation of these parameters. Moreover, the rate constant of the thermal degradation reaction increased with the increment in drying temperatures, meaning that these degradation reactions were temperature dependent. On the other hand, total phenolic content had the lowest activation energy and ΔH value indicating the lowest thermal stability in comparison to others. The degradation reactions endothermically occurred during drying process. Additionally, it was seen that the reason for the higher degradation rate at higher temperatures as the absolute values of ΔS increased. It is important to evaluate thermal degradation of bioactive compounds for the design of the thermal processes. This study will be beneficial for the convective drying process design due to these results.

[a] *begumotag@gmail.com* *https://orcid.org/0000-0003-4989-5354*

Introduction

Thermal processing of the foods has been commonly applied method to prevent or reduce diseases resulted from foods by pathogens and food spoilages by enzymes and microorganisms (Ling et al., 2015). In general, when the thermal process is mentioned, the heating to temperature is considered between 50-150 °C depending on the pH of the food and desired shelf-life (Patras et al., 2010). To provide the food safety and shelf-life requirements, the thermal processes may be applied long time at high temperatures that cause more heat exposure to foods. As a result of this exposure, critical chemical, and physical alterations, reducing food quality, can occur in the foods. Consequently, loss of food quality during thermal treatment becomes the significant issue in terms of consumer acceptance (Ling et al., 2015).

One of the most used thermal treatments in food industry is convective drying whose mechanism is removing water from the foods enabling lower water activity, to prevent growth of microorganisms, to provide longer shelf-life of the foods and to block chemical reactions (Tepe and Kadakal, 2022; Sun et al., 2021; Tunckal and Doymaz, 2020). In addition to this, Reduction in weight and volume occur, and thus obtaining lower packing, transportation, and storage costs (Tepe and Tepe, 2020;). However, convective drying brings some drawbacks like loss of product quality at high temperatures (Huang et al., 2020; Kumar et al., 2020). Especially in fruits and vegetables, ascorbic acid has been considered as the main quality indicator due to thermo-sensitivity to determine loss of quality. In general, well retained ascorbic acid at the of the process indicates lower loss of other nutrients (Wang et al., 2019; Tepe et al., 2022). Ascorbic acid is strong antioxidant that can prevent some diseases such as cancer, cardiovascular diseases, and scurvy Munyaka et al., 2010; Valente et al., 2011; Kurowaza et al., 2014). Degradation of ascorbic acid can vary upon the many factors such as temperature, oxygen, pH, light, metallic catalyzers, and presence of enzymes (Kurowaza et al., 2014). Two pathways; aerobic and anaerobic have been reported by Peleg et al. (2018). The aerobic pathway is

related to presence of oxygen. On the contrary, the anaerobic pathway is independent oxygen and is mainly associated with temperature (Athmaselvi et al. 2016). Moreover, phenolic compounds as the bioactive molecules in plant-based foods is the other group considerably affected by convective drying (Ouyang et al., 2021). Alean et al. (2016) noted higher drying temperature higher phenolic compounds loss. The degradation of phenolic compounds is associated with the binding to other chemical compounds or chemically structural changes during drying process (Mendez-Lagunas et al., 2017). On the other hand, ascorbic acid and phenolic compounds are the important antioxidants that are naturally found in fruits and vegetables (Sevindik et al., 2017; Tepe et al., 2022). Preservation ability from oxidative stress of the antioxidants result from the free radical scavenging ability, free radical formation suppressing, reducing hydrogen peroxide, and quenching superoxide and singlet oxygen (Akgül et al., 2022; Mohammed et al., 2022; Tepe et al., 2022). As an expectation, degradation of these antioxidative compounds generally increases during drying and thus, occurring decrement in the antioxidant activity (Mendez-Lagunas et al., 2017).

Understanding of the thermal properties of the foods and quantitative change of quality parameters is capable of proper design of the thermal process such as drying (Ling et al., 2015). In this context, kinetic models are one of the most potential methods for description of the quality parameters change, occurring in foods. These models can be non-linear or linear forms of the rate law equations (Dhakal et al., 2018). In this study, degradation kinetics and thermodynamic properties of the ascorbic acid (ASA), total phenolic content (TPC) and antioxidant activity (AA) of the kiwi fruits, considered as rich fruit in terms of ASA, TPC and AA, during convective drying. Thus, the results of the study can shed the light for design the convective drying process in the future.

Materials and Methods

Sample Preparation and Drying Procedure

Kiwi fruits, used in the current study, were obtained from a local market in Denizli, Turkey. Following washing to remove non-food materials, kiwi fruits were sliced 5±0.5 mm thickness. 50 g sample kiwi fruit slices were put on the drying tray and placed in the drying oven (Yücebas¸ Makine Ltd. Inc., Izmir, Turkey). Drying procedure was 60°C, 70°C and 80°C at 2 m s^{-1} air velocity and 20% relative humidity. Drying process was completed when the final moisture content of samples reached to 7% (WB).

Ascorbic Acid Extraction

ASA extraction was carried out according to the method suggested by Tepe et al. (2022). After homogenization of 5 g sample with distilled water (1:9, w: v) via a laboratory-type blender, homogenized samples were centrifugated at 4500 rpm for 10 min (Nüve NF800R). Following the centrifugation, the supernatants were collected and filtered via microfilter with a 0.45 µm pore size. The filtrated samples were injected into the HPLC device. The ASA extraction was performed in duplicate.

Chromatographic Conditions of Ascorbic Acid

The HPLC (SHIMADZU) device consisted of a column oven (SHIMADZU CTO-20A), a pump (SHIMADZU LC-20AD), a degasser (SHIMADZU DGU-20A3) and a photo diode array (PDA) detector (SPD-M20A). The column and mobile phase for elution was Coregel 87H3 (7.8x300 mm) and 0.01 N H_2SO_4 which is HPLC purity, respectively. The mobile phase was isocratic with a 1 mL min^{-1} flow rate. The injection volume was 20 µL. For detection, detector wavelength was set at 254 nm.

Total Phenolic Content and Antioxidant Activity Analysis

Method recommended by Tepe et al. (2022) was used to methanolic extraction of TPC and AA. Analyzes of TPC and AC were performed with methanolic extraction suggested by Tepe et al. (2022). 5 g sample of kiwifruit samples were weighted and mixed with 45 mL methanol solution consisted of 90% methanol and 10% distilled water. After homogenization, the homogenized samples were centrifuged at 4500 rpm for 10 min. Then, the supernatants were taken and filtrated using a filter paper.

TPC analysis were performed according to the colorimetric method suggested by Singleton and Rossi (1965). After the mixing of 300 µL of methanolic extract and 1500 µL of Folin-Ciocalteu solution (%10 v: v), the mixture was kept in a dark place for 3 min. 1200 µL of aqueous Na_2CO_3 was added into the mixture after 3 min. Following the incubating of the mixture in a dark place for 2 h, the absorbance of the samples was measured at 760 nm wavelength through a spectrophotometer (T80, PG Ins. UK.). TPC analysis was performed duplicate.

A slightly modified version of the method suggested by Thaipong et al. (2006) was used for the AA analysis. 2850 µL of DPPH solution, whose absorbance was 1.1 at 515 nm, and 150 µL methanolic extract were mixed and incubated in a dark place at ambient temperature for 60 min. After the incubation, the absorbance of the samples was measured at 515 nm. Each sample was analyzed in duplicate.

Kinetic Parameters

The degradation kinetics of ASA, TPC and AA during drying process have been shown to follow the zero order (Eq. 1), first order (Eq. 2), fractional conversion (Eq. 3) or Weibull (Eq. 4) models (Yang et al., 2018) as given below. The most suitable kinetic model was selected with the lowest RMSE and the highest R^2 values.

$$C_t = C_0 - kt \quad (1)$$

$$C_t = C_0 \exp(-kt) \quad (2)$$

$$\frac{C_t - C_f}{C_0 - C_f} = \exp(-kt) \quad (3)$$

$$\frac{C_t}{C_0} = \exp\left[-\left(\frac{t}{\alpha}\right)^{\beta}\right] \quad (4)$$

C_0: Initial value of the compound,
C_t: Corresponding value at time t,
C_f: the amount of the final dried sample,
k: rate constant (h^{-1}),

t: time (h),
α: Weibull scale parameter (h)
β: shape parameter (dimensionless)

Activation energy of the degradation reaction of the compounds was calculated according to the Arrhenius equation given below (Yang et al., 2018).

$$k=k_0 \exp\left(-\frac{E_a}{RT}\right) \quad (5)$$

k_0: frequency factor (min^{-1})
k: rate constant (min^{-1})
R: universal gas constant (8.314 x 10^{-3} kJ mol^{-1} K^{-1} or 1.987 x 10^{-3} kcal mol^{-1} K^{-1})
T: absolute temperature (K)
Ea: activation energy (kJ mol^{-1} or kcal mol^{-1})

Quotient indicator (Q_{10}) expresses temperature-dependence of reaction rate and calculated with the equation 5 (Tepe and Ekinci, 2021):

$$Q_{10} = \left(\frac{k_2}{k_1}\right)^{(10/T_2-T_1)} \quad (5)$$

Thermodynamic Properties

The following equations (Eq. 6, Eq. 7, Eq. 8 and Eq. 9) were suggested for thermodynamic properties (n=1) by Kechinski et al. (2010):

$$\vartheta=\left(\frac{k_B.T}{h}\right) \quad (6)$$

$$k=\vartheta.\exp\left(\frac{-\Delta G}{R.T}\right) \quad (7)$$

$$\Delta H=E_a-n.R.T \quad (8)$$

$$\Delta G = \Delta H - T.\Delta S \quad (9)$$

k_B: Boltzmann's constant (1.381 x 10^{-23} J K^{-1})
h: Plank's constant (6.626 x1 0^{-34} J s^{-1} K^{-1})
ΔG: Gibb's free energy (kcal mol^{-1} or kJ mol^{-1})
ΔH: Enthalpy change (kcal mol^{-1} or kJ mol^{-1})
ΔS: Entropy change (cal mol^{-1} or J mol^{-1})

Statistical Analysis

Non-linear curve fitting toolbox with the trust-region algorithm in MATLAB software (R2015a, version 8.5) was used for the determination of statistical parameters and curve fitting. The most appropriate kinetic model was selected according to the higher values of R^2 and lower values of $\chi 2$ and RMSE values (Tepe and Ekinci, 2021).

Results and Discussion

Thermal Degradation of Ascorbic Acid

For the degradation kinetics of the food components, zero-order and first-order models have been mostly used (Heldman, 2013). However, more than these models must be investigated for the selectin of the most suitable model. The kinetic modelling of the degradation of ASA was given in Table 1. As seen from the Table 1, to determine the most proper model describing thermal degradation of ASA, statistical parameters of zero-order, first-order, fractional conversion, and Weibull kinetic models were compared. The most suitable kinetic model was found as Weibull model according to the RMSE and R^2 values for the thermal degradation of ASA. Degradation curves of the ASA were presented in Figure 1. Likewise, Akar and Barutçu Mazı (2019) noted that the Weibull model gave the best fitting for the degradation of ASA in kiwi fruits dried by different methods when compared to zero-order and first-order models. Mrad et al. (2012) also reported that the Weibull model was the best model for the describing of degradation of ASA in pears. Dağhan et al. (2018) and Niu et al. (2021) noted similar findings in Urfa pepper and winter jujube during drying, respectively. As seen from the Table 1, rate constant of the Weibull model increased with the increment of drying temperature, meaning that, degradation rate of ASA increased at higher drying temperatures, as expected due to thermo-sensitivity of ASA. Consequently, degradation of ASA is temperature dependent. Similarly, Orikasa et al. (2014) and Akar and Barutçu Mazı (2019) notified higher degradation rate of ASA at higher drying temperatures. On the other hand, Table 4 presented activation energy, Q_{10} values and thermodynamic properties of ASA, TPC, and AA. Reaction's temperature sensitivity could be reflected by activation energy. Higher activation energy indicates higher sensitivity to temperature changes. At the same time, higher activation energy reflects higher stability to thermal degradation (Bell, 2020). Activation energy of ASA was calculated as 31.96 kJ mol^{-1}. Orikasa et al. (2008) reported that the activation energy of ASA in convective dried kiwi fruits at 40-50-60-70°C was 38.6 kJ mol^{-1}. Mrad et al. (2012) also noted that the activation energy of ASA was 15.7 kJ mol^{-1} in pears. The activation energy of ASA in whole jujube fruits was stated as 80.87 kJ mol^{-1} by Tepe and Ekinci (2021). The results of the study were slightly lower than reported by Orikasa (2008), highly lower than reported Tepe and Ekinci (2021) and higher than reported Mrad et al. (2012). In different fruits and drying conditions, as expected different results were reported. It means that thermal degradation of ASA depends on food matrix, drying conditions, form of the foods (whole or sliced). Q_{10} value indicates the effect of temperature increment by 10°C on the rate of reaction. This value is mostly used as an indicator temperature's sensitivity of the reaction. Higher Q_{10} values denote greater temperature sensitivity (Bell, 2020). As understood from the Table 4, the sensitivity of the ASA thermal degradation reaction was highly affected by 10°C increasing in drying temperature. The reaction rate similarly increased from 60 to 70°C and from 70 to 80°C.

Thermal Degradation of Total Phenolic Content

The kinetic modelling of the TPC thermal degradation was given in Table 2 and degradation curves at different temperatures were shown in Figure 2. In comparison to other kinetic models, thermal degradation of TPC were suitably fitted to Weibull model with the lowest RMSE and the highest R^2 values. On the contrary, Akdaş and Başlar (2015) and Sarpong et al. (2018) noted that the thermal degradation of TPC in mandarin and banana during drying was fitted to first-order model, respectively.

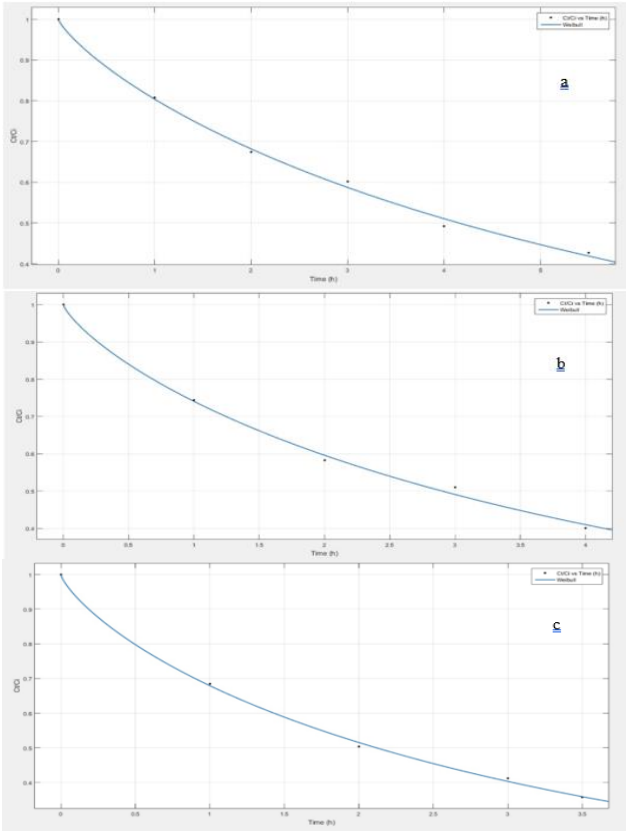

Figure 1. Kinetic modelling (Weibull Model) of ascorbic acid in convective dried kiwi fruits (a: 60°C, b: 70°C, c: 80°C)

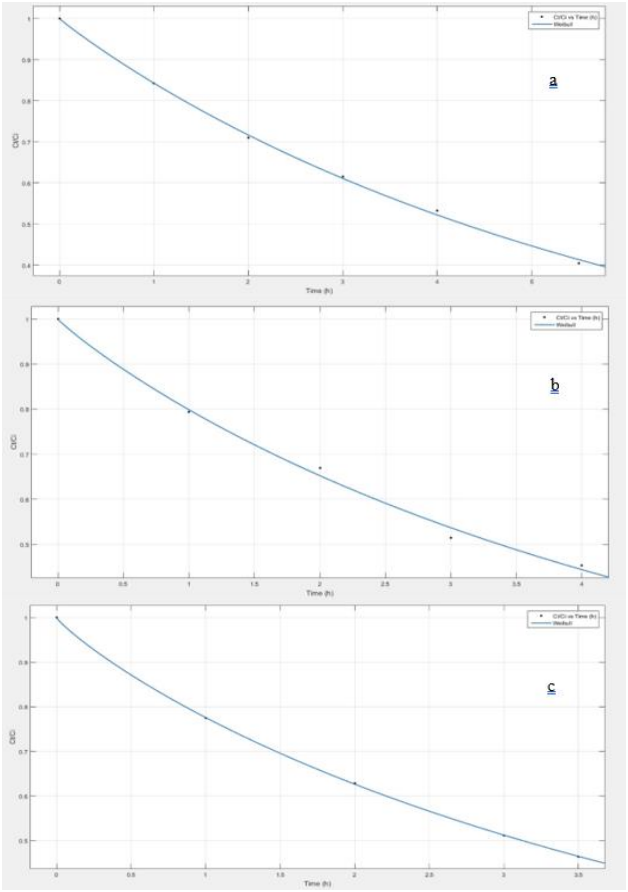

Figure 2. Kinetic modelling (Weibull Model) of total phenolic content in convective dried kiwi fruits (a: 60°C, b: 70°C, c: 80°C)

Mrad et al. (2012) stated pseudo first-order model as the most appropriate model for the degradation of TPC in pears during drying. These differences could be explained with the author preference to kinetic model, food matrix and drying conditions. As seen from the Table 2, degradation rate constant of TPC raised as the temperature increased. Akdaş and Başlar (2015), Sarpong et al. (2018) and Mrad et al. (2012) reported similar findings in different foods. Additionally, activation energy of the degradation of TPC was found as 13.65 kJ mol^{-1}. Sarpong et al. (2018) notified that the activation energy of TPC degradation was 14.29 kJ mol^{-1}. This result was in good agreement with the results of this study. Activation energy of TPC thermal degradation in Granny Smith, Starking Delicious, and Golden Delicious apple varieties during drying at 65, 70, and 75°C were reported to be found as 32.48, 27.52 and 29.84 kJ mol^{-1}, respectively (Ertekin Filiz and Seydim, 2018). Akdaş and Başlar (2015) reported the activation energy as 55.037 kJ mol^{-1} for oven-dried mandarin at 55, 65, and 75°C. The result of current study was lower than those reported by Ertekin Filiz and Seydim (2018) and Akdaş and Başlar (2015). In addition to activation energy, Q_{10} values of the TPC degradation was found as 1.25 for from 60°C to 70°C and 1.06 for from 70°C to 80°C. In the light of these values, TPC degradation was more affected by 10°C temperature increment from 60 to 70°C than 70°C to 80°C.

Thermal Degradation of Antioxidant Activity

Kinetic modelling of AA thermal degradation was given in Table 3 and degradation curves were presented in Figure 3. Thermal degradation of AA followed to Weibull model in comparison to statistical parameters of the other kinetic models used in the current study. Thermal degradation kinetics of AA in other dried fruits and vegetables were fitted to first-order model by authors (Başlar et al., 2014; Oancea et al., 2017; Sarpong et al., 2018; Jha and Sit, 2020; Tepe and Ekinci, 2021). With the results of the current study, it was clear that Weibull model is more effective model to describe the degradation of AA. As seen from the Table 3, the degradation rate constant of AA increased with the increment of drying temperature. It was obvious that AA degradation was temperature dependent. This result showed good agreement with those reports by Oancea et al. (2017), Sarpong et al. (2018), Jha and Sit (2020), and Tepe and Ekinci (2021). On the other hand, activation energy of the AA degradation was calculated as 15.5 kJ mol^{-1}. Sarpong et al. (2018) reported the activation energy of AA degradation in dried banana fruits varied in the range of 14.06-16.36 kJ mol^{-1}. Jha and Sit (2020) stated that the activation energy of AA degradation in dried *Terminalia chebula* Retz. fruits were calculated as 13.83 kJ mol^{-1}. Tepe and Ekinci (2021) noted the activation energy of AA degradation in whole dried jujube fruits as 44.68 kJ mol^{-1}. The result of current study was similar to reports by Sarpong et al. (2018) and Jha and Sit (2020). Moreover, Q10 values of the AA degradation were calculated as 1.07 and 1.17 from 60°C to 70°C and 70°C to 80°C, respectively. Temperature sensitivity of AA thermal degradation was found as similar.

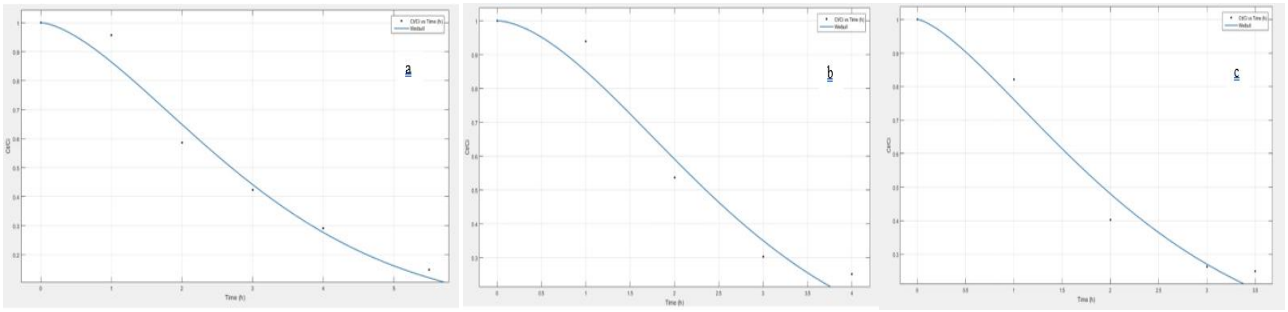

Figure 3. Kinetic modelling (Weibull Model) of antioxidant activity in convective dried kiwi fruits (a: 60°C, b: 70°C, c: 80°C)

Table 1. Statistical parameters of kinetic models and rate constants of ascorbic acid thermal degradation

Kinetic Models	Temperature (°C)	k (mg 100g^{-1} h^{-1})	k (h^{-1})	α	1/α (h^{-1})	β	RMSE	R^2
Zero order	60	47.22					26.13	0.9428
	70	66.26					29.73	0.9430
	80	81.32					35.00	0.9365
First Order	60		0.1641				12.69	0.9865
	70		0.2325				15.13	0.9852
	80		0.3062				15.60	0.9874
Fractional Conversion	60		0.4502				0.05133	0.9806
	70		0.6097				0.04722	0.9853
	80		0.7597				0.04192	0.9893
Weibull	60			6.529	0.1532	0.8110	0.01313	0.9969
	70			4.627	0.2161	0.7853	0.01426	0.9972
	80			3.394	0.2946	0.7777	0.00899	0.9991

Table 2. Statistical parameters of kinetic models and rate constants of total phenolic content thermal degradation

Kinetic Models	Temperature (°C)	k (mg 100g^{-1} h^{-1})	k (h^{-1})	α	1/α (h^{-1})	β	RMSE	R^2
Zero order	60	195.4					63.39	0.9796
	70	253.5					79.77	0.9711
	80	276.6					73.02	0.9751
First Order	60		0.1615				16.48	0.9986
	70		0.2055				35.72	0.9942
	80		0.2219				25.49	0.9970
Fractional Conversion	60		0.3746				0.06455	0.9681
	70		0.5686				0.07776	0.9627
	80		0.6614				0.06662	0.9730
Weibull	60			6.254	0.159898	0.9635	0.008027	0.9989
	70			5.005	0.19980	0.9272	0.017260	0.9954
	80			4.736	0.21110	0.8818	0.001801	0.9999

Table 3. Statistical parameters of kinetic models and rate constants of antioxidant activity thermal degradation

Kinetic Models	Temperature (°C)	k (mg 100g^{-1} h^{-1})	k (h^{-1})	α	1/α (h^{-1})	β	RMSE	R^2
Zero order	60	0.3220					0.1743	0.9452
	70	0.4056					0.2013	0.9312
	80	0.4433					0.1749	0.9461
First Order	60		0.3042				0.1800	0.9412
	70		0.3424				0.2294	0.9106
	80		0.4120				0.1683	0.9501
Fractional Conversion	60		0.3567				0.1332	0.8950
	70		0.4899				0.1868	0.8399
	80		0.6517				0.1486	0.8943
Weibull	60			3.412	0.2931	1.570	0.05884	0.9774
	70			2.918	0.3427	1.704	0.07778	0.9628
	80			2.484	0.4026	1.428	0.06465	0.9733

Table 4. Activation energy, Q_{10} values and thermodynamic properties of ascorbic acid, total phenolic content, and antioxidant activity thermal degradation

Compound	Temperature (°C)	Ea (kJ mol^{-1})	Q_{10}(60-70°C)	Q_{10}(70-80°C)	ΔG (kJ mol^{-1})	ΔH (kJ mol^{-1})	ΔS (J mol^{-1})
Ascorbic acid	60	31.96	1.41	1.36	87.06	29.19	-173.77
	70				88.77	29.11	-173.95
	80				90.54	29.03	-174.25
TPC	60	13.65	1.25	1.06	86.94	10.88	-228.40
	70				89.00	10.80	-227.99
	80				91.51	10.76	-228.89
Antioxidant Activity	60	15.5	1.07	1.17	85.26	12.73	-217.8
	70				87.46	12.65	-218.11
	80				89.62	12.57	-218.29

Thermodynamic Properties of Ascorbic Acid, Total Phenolic Content and Antioxidant Activity

The enthalpy change (ΔH), Gibb's free energy (ΔG) and the entropy change (ΔS) of ASA, TPC and AA were given in Table 4. ΔH defined as the energy difference between the reactant and the activated complex during the drying process (Sarpong et al., 2018). ΔG is the fundamental criterion for spontaneity of chemical reaction (Mercali et al., 2015). The disorder change of molecules in a system is represented with ΔS (Sarpong et al., 2018). Besides, positive sign of ΔH represent endothermic reaction, which indicates reaction requiring energy (Kechinski et al. 2010; Al-Zubaidy and Khalil 2007). ΔH values of the degradation of ASA, TPC and AA were determined as from 29.03 to 29.19, 10.76 to 10.88 and 12.57 to 12.73 kJ mol^{-1}, respectively. TPC degradation had the lowest ΔH values, which indicates highly degradative nature in comparison to ASA and AA. Temperature variation had little effect on ΔH due to the small amount of the ideal gas constant (R). ΔH values of ASA, TPC and AA was found as positive, meaning that degradation reactions of these compounds endothermically occurred. ΔG values of ASA, TPC and AA thermal degradation were calculated as from 87.06 to 90.54, 86.94 to 91.51 and 85.26 to 89.62 kJ mol^{-1}, respectively: an indicative of non-spontaneous reactions. It was clear that total energy in the system at the approach of the reagents increased could be explained with the closeness of ΔG values.

It is the evidence for higher degradation rate of ASA, TPC and AA. The negative value of ΔS indicates lower structural freedom than the reactant of transition state. In addition to this, the absolute value of ΔS, creating the difference in thermodynamic equilibrium from the system at the beginning of the process, increased due to high temperatures. It is the evidance for higher degradation rate of ASA, TPC and AA.

Conclusions

In the current study, thermal degradation kinetics and thermodynamic properties of ASA, TPC and AA of kiwi fruits dried at different temperatures. The results of the study were summarized below.

- Thermal degradation of ASA, TPC and AA were well described by Weibull model.
- The reaction rate of ASA, TPC and AA increased with the increment of drying temperature, meaning that the degradation of these compounds was temperature dependent.
- TPC had the lowest thermal stability, whereas the degradation of ASA was more stable than TPC and AA. However, ASA degradation had the highest sensitivity to temperature changes.
- The lowest ΔH value was obtained from the TPC degradation, indicating the highest sensitivity to thermal degradation. Besides, the degradation of these compounds was found as endothermic reaction.
- The absolute value of ΔS increased as the temperature increased. It was the explanation the high degradation rates at higher temperatures.

In conclusion, to understand thermal degradation kinetics and thermodynamic properties of these compounds, proper designation of the drying process could be facilitated. The results of the current study may be beneficial for convective drying process of kiwi fruits.

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