

Acta Sci. Pol. Technol. Aliment. 23(4) 2024, 491–502

ORIGINAL PAPER

<http://doi.org/10.17306/J.AFS.001212>

Received: 30.04.2024 Accepted: 2.09.2024

MODELING THE DEGRADATION OF BIOACTIVE COMPOUNDS DURING AIR CONVECTION DRYING OF ASPARAGUS ROOTS (*ASPARAGUS OFFICINALIS* **L.)**

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ABSTRACT

Background. Asparagus root (*Asparagus officinalis* L.) is one of the herbaceous by-products with potential for making added-valued food products, due to its excellent nutritional properties and flavor/fragrance. In order to maintain its quality, several drying methods were investigated to select an advanced one that could be widely applied in the processing of dried vegetable products.

Materials and methods. A thin layer at 40–90°C using a thin-layer drying oven was investigated to study the thermal degradation kinetic of bioactive compounds, including vitamin C, phenolic, flavonoid, saponin and antioxidant capacity of dried asparagus roots. Linear and non-linear regression analysis (zero-, first and second-order) was employed to assess the parameters, which helped select the optimal model that accurately characterizes an efficient drying process for asparagus roots.

Results. The first-order model was described for vitamin C degradation and matched the experimental data well. The decomposition of phenolic, flavonoid, saponin and antioxidant capacity followed the second-order model with the highest coefficient of determination value ($R^2 > 0.99$) and the lowest root mean squared error (RMSE) value. Enhanced levels of bioactive compounds and antioxidant capacity notably slowed down the degradation of anthocyanins at a temperature of 70℃.

Conclusion. This study reflected the thermal degradation kinetic for the drying process. The results could provide useful information for the optimizing the drying of asparagus roots with the aim being to obtain a product with high nutritive quality and bioactivity.

Keywords: antioxidant capacity, bioactive compounds, degradation, kinetic models, temperature

INTRODUCTION

Asparagus (*Asparagus officinalis* L.) is considered one of the world's top 20 most valuable vegetable crops due to its exceptional nutritional attributes. Its unique flavor and fragrance stem from a variety of volatile components, including pyrazines and compounds containing sulfur (Pegiou et al., 2019). With

a history of cultivation spanning over two millennia across the globe, asparagus has firmly established itself as a cultivated plant. Asparagus is a perennial herb that typically takes 7–8 years to mature for commercial use. During harvest, the roots and rhizomes are often left in the fields as by-products (Viera-Alcaide

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et al., 2021). There are two main types of asparagus: green and white, both with high annual consumption. The global asparagus market is expected to grow at a consistent rate of 3% annually. Asparagus contains various phytochemicals, including saponins, flavonoids, vitamins, along with other polysaccharides and dietary fiber (Fuentes-Alventosa et al., 2013; Zhang et al., 2019). These compositions exhibit a spectrum of effects such as anti-cancer, antitumor, antioxidant, immunomodulatory, hypoglycemic, anti-hypertensive, and anti-epileptic actions, thereby boosting the crop's value (Pegiou et al., 2019; Guo et al., 2020). Additionally, bioactive compound concentrations such as polysaccharides, specifically fructans are primarily stored in the roots, with a significant accumulation in the lower sections of the spears, surpassing other segments in content (Suzuki et al., 2011; Guo et al., 2020; Witzel and Matros, 2020). Despite these attributes, there are still few studies focusing on the use of this by-product. Consequently, the asparagus root remains a latent source material for the creation of value-added products, contributing to the amplification of the crop's value and the judicious utilization of this by-product.

Various technical methodologies are employed for the post-harvest of crop products. Drying is one such method, offering the dual benefits of cost reduction in handling and distribution, along with an extension of the shelf life for moisture-rich raw materials (Chen and Mujumdar, 2009). This drying process operates based on the principles of concurrent heat and mass transfer. Heat infiltrates the food item, inducing the movement of moisture from within to the surface, subsequently evaporating into the air stream as vapor. The oven-drying technique, especially when utilizing a thin layer, is a widely adopted approach, which is due to its economic viability and suitability for plantbased materials (Chen and Mujumdar, 2009).

Effective control over operational parameters during the process and the anticipation of drying efficacy via mathematical models were critical for ensuring the quality of the end product. Using mathematical kinetic models including zero-, first-, and secondorder models to explain the degradation of bioactive compounds offers a more comprehensive understanding of the degradation kinetics. Linear and non-linear regression models allow for a more flexible approach to modeling degradation kinetics, taking into account factors beyond just temperature, such as time and initial compound concentration. This enables a more accurate characterization of the degradation process and provides insights into the underlying mechanisms driving the degradation of bioactive compounds (Ramachandran et al., 2018). This paper revolved around assessing the influence of different temperatures (ranging from 40°C to 90°C) within the thin-layer drying, in comparison with samples dried using a solar system and sun exposure. The objective was to ascertain the impact of these methods on the physicochemical attributes of dried asparagus roots, which shows potential as a raw material for creating value-added products.

MATERIAL AND METHODS

Materials and equipment

High-quality *Asparagus officinalis* L. roots, which were fresh without physical damage and non-infestation, were acquired from My Thoi Ward in Long Xuyen city, An Giang province, Vietnam.

The drying process was carried out employing various drying techniques, using a Forced Convection Drying Oven (ESCO, OFA-110-8, Indonesia).

Experimental design

The raw asparagus roots, having undergone sorting and washing, received a preliminary treatment of blanching (immersion in hot water at 85°C for 2 minutes) prior to the drying process. A completely randomized experiment was set up with three replications, and each sample consisted of 2 kg of green asparagus roots, which were cut into pieces measuring approximately 1 cm. Following this blanching, the samples were spread evenly onto stainless steel trays (measuring 40×60 cm²) and subjected to drying within a Forced Convection Oven (ESCO, OFA-110-8, Indonesia). The oven operated with an airflow velocity of 1m/s, while the temperature ranged from 40°C to 90°C.

To determine the initial moisture content before initiating the drying procedure, a slightly modified approach based on Nielsen's method (2010) was employed. The drying process continued until the material's moisture content dropped below 0.1 kg H_2O/kg dry matter, equivalent to a moisture content of 10%.

Analysis methods Determination of vitamin C

Acid ascorbic was detected based on the 2,4-dinitrophenyl hydrazine colorimetric method described by Sharaa and Mussa (2019) with some modifications. Approximately 1 g of the sample with 5 mL of the solution containing 3% meta-phosphoric acid (w/v) and 8% glacial acetic acid (v/v) were taken in a 15 mL centrifuge tube. The centrifuge tube was placed on the reciprocating shaker (Stuart, UK) for 1 hour. 1 mL of supernatant after centrifugation was mixed with 0.5 mL of 3% bromine, 0.25 mL of 10% thiourea and 0.25 mL of 2,4-dinitrophenyl hydrazine. The mixture was incubated for 3 hours at 37°C. After that, 10 mL of 85% H_2SO_4 was added to the tube to form a red complex. The solution was cooled to room temperature and analyzed with an absorption at 521 nm. The concentration of vitamin C was calculated with a standard ascorbic acid graph, $y = 0.2253x + 0.0024$ $(R² = 0.9999)$, where *y* is the absorbance and *x* is the concentration of solution in the tube.

Determination of total phenolic

Phenolic content (mg TAE/kg of dry matter) was indicated based on the reaction with Folin-Ciocalteu (Sumaiyah et al., 2015). In brief, 0.15 mL of the sample was mixed with 1.2 mL of distilled water and 0.45 mL of 5% (w/v) Na_2CO_3 in a test tube. The mixture was added to 0.1 mL of Folin-Ciocalteu reagent and left at room temperature for 90 mins for the reaction to take place. Phenolic in the extract reacts with Folin-Ciocalteu to form a phosphomolybdenum complex with blue color in the alkaline medium. The concentration of total phenolics was calculated according to the standard tannic acid graph (TAE), $y = 0.0021x + 0.0064$ (R² = 0.9999), where *y* is the absorbance and *x* is the concentration of the solution in the tube.

Determination of total flavonoid

Flavonoid content was observed through the colorimetric reaction using the aluminum chloride, with some modifications from the method described by Sumaiyah et al. (2015). The reaction creates a stable acid complex by $AICI_3$ with the C-4 keto groups and the hydroxyl C-3 or C-5 group of the flavones and flavonol. In brief, 0.1 mL of the sample was mixed with 1.2 mL of distilled water and 30 μ L of 5% (w/v) NaNO₂. After 5 mins, the mixture was supplemented with 10% (w/v) AlCl₃.H₂0 (60 µL), 0.2 mL of 1 M NaOH and 0.11 mL of water. The solution was then measured at 510 nm. The total flavonoid concentration was calculated based on the standard quercetin graph (QE), $y = 8.2634x +$ 0.0182 ($\mathbb{R}^2 = 0.9999$), where *y* is the absorbance, and *x* is the concentration of the solution in the tube.

Determination of saponin

Saponin content was carried out using the vanillinsulfuric acid method (Le et al., 2018). A red-violet reaction is from the oxidation of triterpene saponins by sulfuric acid and vanillin. Approximately 0.25 mL of the sample was supplemented with 0.25 mL of 8% (w/v) vanillin in ethanol 96% and 2.5 mL of 72% $H₂SO₄$ acid. The mixture was incubated for 30 minutes at 60°C and then cooled at room temperature. The solution was measured at 560 nm. The concentration of saponin was calculated by a standard saponin graph (SE), $y = 0.1348x + 0.0075$ (R² = 0.9999), where *y* is the absorbance and x is the concentration of solution in the tube.

Determination of DPPH (2,2-diphenyl-1-picrylhydrazyl) scavenging activity

The antioxidant activity of the sample was determined by the free radical scavenging capacity of 2,2-diphenyl-1-picrylhydrazyl (DPPH). In accordance with Molyneux (2004), a DPPH assay was carried out with slight modifications, a nd t he s ample (1.5 m L) was mixed with DPPH solution in the ratio of 1:1 (v/v). This assay is based on the electron transfer that produces a purple solution in ethanol and was analyzed at 517 nm. Inhibition of DPPH free radicals was calculated using equation 1:

Inhibition of DPPH radical (%) = $100 \times (A_c - A_s)/A_c$ (1)

where:

 A_c is the absorbance of the control

 A_s is the absorbance of the sample.

Determination of ferric reducing antioxidant power (FRAP)

A FRAP assay (mM of FeSO_4/g dry matter) was measured using the method described by Sudha et al. (2012), with some modifications. This method is based on the reduction of tripyridyltriazine complex Fe $(TPTZ)^{3+}$ to blue colored Fe $(TPTZ)^{2+}$ by antioxidants in an acidic medium. The FRAP reagent contained 100 mL of 200 mM acetate buffer (pH 3.6), 10 mL of 20 mM $FeCl₃$.6H₂O and 10 mL of a 10 mM TPTZ in 40 mM HCl. 0.05 mL of the sample was supplemented with 1.5 mL of the FRAP reagent and 0.15 mL of distilled water. The mixture was incubated at 37°C for 8 mins. It was later analyzed at an absorption of 593 nm.

Computation the kinetics of phytochemical compositions degradations

The zero-, first-, and second-order kinetic models were computed using equations 2, 3, and 4, respectively. The most suitable model for each function was determined by selecting the one with the highest the coefficient of determination (R^2) value and the lowest root mean squared error (RMSE) value, following the methodology outlined by Charurungsipong et al. (2020). Statistical analysis of the drying kinetics was conducted using Statgraphics Centurion XVI (U.S.A.) software. Linear and non-linear regressions analysis was employed to assess the parameters, aiding the selection of the optimal model that accurately characterizes the efficient drying process of asparagus roots.

Zero-order:
$$
C = -kt + C_o
$$
 (2)

First-order:
$$
ln(C/C_0) = -kt
$$
 (3)

Second-order:
$$
(1/C) - (1/C_0) = kt
$$
 (4)

where:

 C_{o} is an initial target function (vitamin C, phenolic, flavonoid, saponin) content

- C is a target function content after time (hours) of drying
- the constant k is a parameter representing the rate at which a substance degrades or decays over time (hours $^{-1}$).

RESULTS AND DISCUSSION

Changes in bioactive compositions of asparagus roots at different drying conditions

The drying process hinges significantly on temperature, and numerous studies have delved into the correlation between vitamin C retention, temperature, and degradation kinetics. This degradation could arise from both vitamin C oxidation and thermal breakdown (Wang et al., 2019). As the drying temperature increased from 40°C to 90°C, the vitamin C content exhibited a notable and rapid decline (Fig. 1a). The most significant reduction, amounting to 67.77%, occurred when asparagus was dried at the highest temperature of 90 \degree C (1.29 g/100 g of dry matter), which was approximately 1.5 times greater than the loss observed in samples dried at 70°C for the same duration of 6 hours (2.08 g/100 g of dry matter). Conversely, samples dried at lower temperatures (40°C, 50°C and 60°C) retained a higher proportion of vitamin C, with losses approximately totaling 58% (2.76, 2.22 and 2.20 g/100 g of dry matter, respectively). This phenomenon could be attributed to the extended drying times associated with temperatures of 40°C, 50°C and 60°C, which reached to 16, 10, and 9 hours, respectively.

Fig. 1. Change in vitamin C and phenolic contents during drying process at different drying temperatures

Prolonged exposure to elevated temperatures during the drying process could induce irreversible oxidation processes, leading to a reduction in vitamin C retention, as vitamin C is particularly sensitive to heat and susceptible to degradation throughout the drying process (Di Scala et al., 2013; Afolabi et al., 2015). This was illustrated by the degradation rate (k), which depends on the investigating factors (drying temperature and time). A significant rise in the degradation rate (k) occurred with an increase in temperature (Table 1),

which was explained based on the rate of a reaction that typically increases exponentially with temperature (Ramachandran et al., 2018).

Many researchers have found that when it came to the degradation of ascorbic acid in various materials due to heat or storage, the first-order kinetic model provided an effective and accurate fit (Gamboa-Santos et al., 2014; Wang et al., 2019). In the study by Gamboa-Santos et al. (2014) on the strawberry-drying process at 40–70°C, it was found that the first-order

 Y – target functions; x – drying time (hours).

reaction model provided a good fit with the experimental figures (\mathbb{R}^2 in the range of 0.97–0.99). As with previous studies, the vitamin C decomposition of asparagus roots dried at 40–90°C was described with the experimental outcomes using the first-order model with the R^2 valued up to 0.994 (Table 1).

The decomposition of phenolic compound primarily is caused by oxidation, the breaking of covalent bonds, or the intensification of oxidation reactions due to oxidative enzyme (polyphenol oxidase and peroxidase) and thermal treatment (Schweiggert et al., 2007; Zoric et al., 2014; Lien and Toan, 2017). The retention of phenolic compounds declined with the rise in temperature and drying time (Figure 1b). In particular, the phenolic content remaining in the asparagus roots dried at 80°C and 90°C stood at 0.30 and 0.23 g TAE/100 g of dry matter (about 66% and 52%, respectively). It should be noted that the substantial preservation of phenolic compounds registered levels approaching over 70% when the drying conducted at 50°C, 60°C and 70°C, whereas at the lowest drying temperature (40°C), the phenolic retention stood at 61% (0.27 g TAE/100 g of dry matter) with regard to the longest processing duration of up to 16 hours. A decrease in total phenolic compounds as well as other bioactive compounds was observed clearly through the degradation rate (k), which rose along with the higher drying temperatures (Table 2). High temperatures provide more thermal energy, which facilitates the breaking of chemical bonds and the degradation or decay processes. As a result, molecules may degrade more rapidly at elevated temperatures (Lien and Toan, 2017; Ramachandran et al., 2018).

These findings can be rationalized by the fact that the enzyme polyphenol oxidase becomes inactive when exposed to temperatures of 80°C for 10 minutes, and is completely deactivated within the range of 90°C to 100°C (Miranda et al., 2010; ElGamal et al., 2023). However, during the drying process at 40° C, 50°C, and 60°C, the retention of bioactive compounds was possible but at lower levels than those dried at 70°C. This decline was attributed to the extended drying duration at lower temperatures (ranging from 40° C to 60° C), during which these compounds were susceptible to decomposition when exposed to atmospheric oxygen. For the samples dried at a high temperature $(>80^{\circ}C)$, the oxidative potential of phenolic compounds was significantly enhanced due to the increase of reaction rates, which consequently resulted in reduced phenolic content (Vega-Gálvez et al., 2009; Abhay et al., 2016). However, the degradation of phenolic compounds during the drying process followed the second-order kinetics, which was described with the experimental data and correlation coefficients $(R²)$ were up to 0.995 (Table 1).

The effects of temperature on the degradation of flavonoid and saponin contents were similar to the changes of vitamin C and phenolic compounds during the drying process (Fig. 2). The higher the temperature, the greater the decomposition, which supports the data of the degradation rate constant *k* shown in Table 2. The flavonoid and saponin retentions value

Fig. 2. Change in flavonoid and saponin contents during the drying process at different drying temperatures

 Y – target functions; x – drying time (hours).

remained lower than 67% and 55%, respectively. It is notable that the substantial preservation of flavonoid and saponin compounds with the highest levels (approximately 0.37 g QE/100 g of dry matter and 2.98 g SE/100 g of dry matter, respectively) appeared when the drying temperature was higher than 80°C, whereas the lowest retention of flavonoid and saponin was at 21% (0.12 g QE/100 g of dry matter) and 24% (1.32 g SE/100 g of dry matter), respectively, with the longest drying duration, up to 16 hours at 40°C.

Flavonoid and saponin exhibit similar thermal degradation patterns to vitamin C (Kavita et al., 2006). Conversely, flavonoid has reductive properties and is susceptible to oxidation during the drying process, with higher drying temperatures accelerating their reaction rate (ElGamal et al., 2023). As the group of flavonoid and saponin shares a common molecular feature, it is characterized by the presence of a benzene ring and varying numbers of hydroxyl groups (-OH) known for their strong reducibility. Their degradation mechanisms are described when they are exposed to heat,

and these hydroxyl groups are often transformed into aldehyde groups, ketone groups, or carboxyl groups through oxidation reactions. Consequently, the dehydrogenation of the hydroxyl group (-OH) in flavonoid and saponin leads to the formation of aldehyde groups (-CHO), which are subsequently oxidized into carboxyl groups (ElGamal et al., 2023). As is the case with phenolic, the decomposition of flavonoid and saponin compounds was both well demonstrated by the second-order kinetics, with the most reliability of experimental data ($\mathbb{R}^2 > 0.98$). Consequently, lower drying temperatures were favored in order to maximize bioactive compounds retention during the whole process.

Changes in antioxidant activity of asparagus roots in different drying conditions

The degradation of antioxidant compounds such as phenolics, vitamin C, flavonoid, etc. leads to decreased antioxidant activity. The changes in the antioxidant capacity (DPPH and FRAP) at different temperatures during the drying process are shown in Figure 3. Research on antioxidant activity has consistently demonstrated a positive association with phenolic compounds and their derivatives. Chemical transformations affecting the levels of phenolic and flavonoid compounds in dried products can take place during the drying process at temperatures within the range from 40°C to 80°C (Vidinamo et al., 2022). Excessively elevated temperatures are found to release a greater amount of bioactive compounds (Maisuthisakul et al., 2007; Saha and Tamrakar, 2011). It is worth noting that excessively high temperatures can adversely impact bioactive compounds by causing structural degradation in certain phenolic compounds (Pashazadeh et al., 2020; Zannou et al., 2021). Nonetheless, asparagus roots dried at 70°C gave the highest phenolic and flavonoid content, thus DPPH and FRAP also presented a high percentage at this temperature, at 43.18% and 44.62 mM $FeSO_4/100$ g of dry matter, respectively. This result is similar to the study carried out with dates dried at an optimum temperature at 70°C (Deng et al., 2018; Hassan et al., 2022). The increased antioxidant potential observed in dried fruits and vegetables might be attributed to the enhanced antioxidant activity of partially oxidized polyphenols compared to their nonoxidized counterparts (Dalla Nora et al., 2014). Furthermore, the presence of Maillard reaction products, which can form during heat treatment and often possess robust antioxidant properties, can contribute to the heightened antioxidant capacity observed after the drying process (Kamiloglu et al., 2016).

The data of degradation rate constant *k* in Table 3 shows that the drying temperature had an effect on the antioxidant capacity (DPPH and FRAP) of the product. The second-order kinetic model was considered as describing the degradation of the antioxidant capacity well, which was seen through both indicators' (DPPH and FRAP) inhibitory percentage of dried asparagus roots with the highest reliability and correlation coefficients $(R^2 > 0.99)$.

Fig. 3. Change in DPPH (%) and FRAP contents during drying process at different drying temperatures

DPPH $(\%)$				FRAP (mM Fe ²⁺ /g dry matter)		
Drying modes	Equation	\mathbb{R}^2	RMSE	Equation	\mathbb{R}^2	RMSE
Zero-order Model: $C = -kt + C$						
40° C	$y = -0.841x + 51.674$	0.828	46.236	$y = -1.767x + 65.952$	0.918	4.106
50° C	$y = -1.354x + 52.666$	0.881	41.187	$y = -2.528x + 70.269$	0.968	2.261
60° C	$y = -1.742x + 54.436$	0.896	2.318	$y = -2.616x + 69.063$	0.960	2.036
70° C	$y = -2.359x + 56.380$	0.965	1.051	$y = -4.610x + 69.490$	0.951	2.442
80° C	$y = -2.402x + 57.253$	0.983	0.728	$y = -4.497x + 69.802$	0.952	2.347
90° C	$y = -2.425x + 56.259$	0.918	1.706	$y = -4.538x + 69.796$	0.942	2.638
First-order Model: $ln(C/Co) = -kt$						
40° C	$y = -0.018x$	0.851	0.059	$y = -0.033x$	0.947	0.060
50° C	$y = -0.029x$	0.905	0.049	$y = -0.044x$	0.979	0.033
60° C	$y = -0.036x$	0.910	0.051	$y = -0.045x$	0.978	0.030
70° C	$y = -0.048x$	0.972	0.023	$y = -0.081x$	0.968	0.044
80° C	$y = -0.048x$	0.988	0.015	$y = -0.079x$	0.967	0.042
90° C	$y = -0.048x$	0.929	0.040	$y = -0.079x$	0.960	0.047
Second-order Model: $(1/C) - (1/C0) = kt$						
40° C	$y = 0.0003x$	0.871	0.000	$y = 0.001x$	0.969	0.000
50° C	$y = 0.001x$	0.925	0.004	$y = 0.001x$	0.986	0.004
60° C	$y = 0.001x$	0.923	0.001	$y = 0.001x$	0.989	0.000
70° C	$y = 0.001x$	0.992	0.000	$y = 0.001x$	0.980	0.001
80°C	$y = 0.001x$	0.979	0.001	$y = 0.001x$	0.978	0.001
90° C	$y = 0.001x$	0.939	0.001	$y = 0.001x$	0.975	0.001

Table 3. The kinetic models of DPPH and FRAP inhibitory degradation at different drying modes

 Y – target functions; x – drying time (hours).

CONCLUSION

This study reflected the thermal degradation kinetic of bioactive compounds and their antioxidant in asparagus roots during the drying process. The mathematical kinetic model of vitamin C degradation followed the first-order model, with the highest R^2 (0.99) and the lowest RMSE (0.03). Other bioactive compounds including phenolic, flavonoid, saponin, DPPH and FRAP provided a good fit with the second-order model. The \mathbb{R}^2 and RMSE of this model ranged from 0.98–0.99 and 0.02–0.84, respectively. Moreover, from the results obtained, the most sensitive temperature of the drying process was 70°C. From the experimental data and the correlations among the indicators determined, useful information can be provided in order to describe the drying process of other crop products with the aim of obtaining a product with high nutritive quality and bioactivity.

DECLARATIONS

Data statement

All data supporting this study has been included in this manuscript.

Ethical Approval

Not applicable.

Competing Interests

The authors declare that they have no conflicts of interest.

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