



## Effect of pre-treatment conditions and freeze-drying temperature on the process kinetics and physicochemical properties of pepper

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### ABSTRACT

This study shows the effects of blanching, citric acid addition, and drying temperature on the freeze-drying kinetics, L-ascorbic acid content, colour, and antioxidant activity of freeze-dried pepper. The process was performed at 20 °C, 40 °C, and 60 °C and with a constant pressure in a drying chamber at 63 Pa. The samples of pepper were pulped before drying. Blanching of pepper reduced the drying time to approximately 30%. The shortest drying time (about 290 min) was found for blanched pepper that was freeze-dried at 60 °C, whereas the samples of pepper freeze-dried at 20 °C and without blanching required the longest drying time (about 900 min). The kinetics of freeze-drying of pepper pulp are best described by using the Page model. The addition of citric acid increased the redness and yellowness of dried pepper, whereas an increase in drying temperature caused a decrease in the total phenolics content, antioxidant activity, and colour coordinates of all samples. The highest L-ascorbic acid content was found in unblanched pepper and when the temperature of drying did not exceed 40 °C. Water blanching pretreatment had the most negative effect on total phenolics content and antioxidant activity of dried pepper.

### 1. Introduction

The pepper (*Capsicum annum*, L.), indigenous to South and Central America, is now grown worldwide and has been incorporated into many different cuisines (Vega-Gálvez et al., 2009). The dehydrated and powdered fruits of red pepper, called paprika, are most widely used as a food colorant (Topuz, Feng, & Kushad, 2009). Moreover, this spice can modify the flavour of food, due to its characteristic taste and pungency (Martín et al., 2017). Fruits of pepper can vary tremendously in colour, shape, and size, both between and within the species. Ripe pepper fruits belonging to different varieties display a range of colours, from white to deep red (Arimboor, Natarajan, Menon, Chandrasekhar, & Moorkoth, 2015). The colour and degree of pungency are valued as major quality parameters in the paprika trade. There are many nutraceutical benefits associated with the consumption of pepper. Especially, red pepper has been recognised as an excellent source of different phytochemicals such as flavonoids, quercetin, vitamin C and capsaicinoids (Vega-Gálvez et al., 2009). Furthermore, red pepper fruits contain more than 20 different carotenoids, which are the primary source of their colours (Hallmann & Rembiałkowska, 2012). Carotenoids are lipophilic yellow-orange-red pigments found in

photosynthetic plants, algae, and microorganisms. These compounds are commercially used as food and feed additives and are also used in pharmaceutical, nutraceutical, and cosmeceutical products (Arimboor et al., 2015). Red pepper contains mainly such carotenoids as β-carotene, lutein, and capsanthin (Rodríguez-Amaya, Kimura, Godoy, & Amaya-Farfan, 2008), which are source of natural colours in plants. The antioxidant potential of carotenoids is of particular significance to human health. Data obtained from epidemiological studies and clinical trials strongly support the observation that adequate carotenoid supplementation may significantly reduce the risk of several disorders that are mediated by reactive oxygen species (Fiedor & Burda, 2014). Besides, pepper is a richer source of vitamin C, more than other vegetables and fruits that are commonly recognised as a source of this vitamin (Dürüst, Sümengen, & Dürüst, 1997).

Traditionally, paprika is obtained by sun drying red peppers (Condori, Echazu, & Saravia, 2001; Topuz et al., 2009). Magied and Ali (2017) found that the solar drying method yielded high values for colour and rehydration ratio of dried pepper than the conventional drying method. However, solar drying is a long process and takes up to 10 days (Oberoi, Ku, Kaur, & Baboo, 2005); moreover, in many countries, weather conditions are inadequate for such drying. This technique

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also increases the possibility of fungal proliferation, which creates favourable conditions for mycotoxin contaminations (Arslan & Ozcan, 2011). Thus, hot-air drying is often used for pepper dehydration with a drying temperature of 50–60 °C. This method reduces the drying time to a maximum of 20 h (Minguez-Mosquera, Jaren-Galan, & Garrido-Fernandez, 1994). Unfortunately, this dehydration method negatively affects the quality attributes of paprika such as texture, flavour, colour, and nutritional value (Topuz et al., 2009). Park and Kim (2007) compared the different methods of pepper drying; they found that freeze-drying is the most suitable dehydration method for stabilizing the antioxidant compounds and red pigment of pepper. Data obtained from several other authors have also revealed that freeze-drying is one of the best food preservation methods. Compared to others food drying techniques, the most significant benefits of freeze-drying include the following: retention of morphological, biochemical, and immunological properties, high viability, long shelf life, retention of structure, and high recovery of violates (Cieurzyńska & Lenart, 2011). Especially, freeze-drying in comparison with hot-air drying results in products with higher content of vitamins and antioxidants, better colour, crisper texture, and better rehydration capacity (Orak et al., 2012; Lee, Oh, Han, & Lim, 2012; Sogi, Siddiq, Greiby, & Dolan, 2013). Unfortunately, this method of drying is time-consuming and expensive. This process can be shortened by using different drying conditions and adequate methods of raw material pretreatment (Rudy et al., 2015). Moreover, the different methods of food pretreatments before freeze-drying have a definite effect on the quality attribute of the final products (Cieurzyńska, Lenart, & Gręda, 2014; Dehghannya, Gorbani, & Ghanbarzadeh, 2017; Garcia-Noguera, Oliveira, Weller, Rodrigues, & Fernandes, 2014; Prosapio & Norton, 2017). To the best of our knowledge, these aspects have not been studied for freeze-dried pepper. Thus, the aim of this study was to determine the effect of pepper pretreatments and freeze-drying temperature on the process kinetics and physicochemical properties of dried pepper.

## 2. Materials and methods

### 2.1. Materials

Mature pods of red pepper (*Capsicum annuum*, L., cv. Kaskada) were grown and harvested in September 2017 in Lublin, Poland. The pods were stored at 4 °C before processing for a maximum period of six days. The samples of pepper were selected visually based on colour and freshness, and with no sign of mechanical damage. Next the pods were washed using tap water, cut into 1.0-cm thick slabs lengthwise, the inside seeds were removed, and the water content was determined (AOAC, 1990).

### 2.2. Pepper pretreatments

The samples of pepper were prepared for freeze-drying as follows:

- pepper was blanched in hot water (WB) (90 °C) for 1 min using a thermostated water bath (Horyzont UL-1, Poland),
- samples of pepper were placed in the microwave oven (MB)

(Daewoo KOR-GL05, China) for 1.5 min at a power of 650 W (Castro et al., 2008; Dorantes-Alvarez, Jaramillo-Flores, González, Martínez, & Parada, 2011).

After blanching, all samples were cooled at room temperature and the surface water was removed using absorbent paper. Blanched and control samples of pepper were dried after blending into a homogenous mass (30 s) using a knife blender (Braun, Model, 2001; Germany). Subsequently, citric acid (CA) (250 mg/100 fresh pepper) was to the pepper purée. In addition, a control sample (pepper purée with no acid added) was freeze dried as well.

### 2.3. Freeze-drying method

Samples of raw peppers (a single layer of about a 5 mm layer of pepper purée) were placed on a stainless steel plated with a diameter of 21 cm and frozen at –25 °C for 48 h using a freezer (Liebherr GTL-4905, Germany). The same sample was freeze dried each time using an ALPHA 1–4 laboratory freeze dryer according to the method described by Dziki et al. (2018). The process of drying was performed at 20 °C, 40 °C, and 60 °C and with a constant pressure of 63 Pa in the drying chamber until the sample reached an equilibrium moisture of about 70 g H<sub>2</sub>O/kg fresh weight (FW). After freeze-drying, the material was stored in a dark place and in tightly closed polyethylene bags at a temperature of 20–22 °C.

### 2.4. Characteristics of drying curves

Based on the measurements of mass loss taken over the course of the experiment, drying curves were obtained as functions of water ratio (MR) versus time, using the following equation (Motevali, Younji, Chayjan, Aghilinategh, & Banakar, 2013):

$$MR = \frac{u}{u_0}, \quad (1)$$

where  $u$  is the water content during drying [kg H<sub>2</sub>O/kg dry matter (DM)] and  $u_0$  is the initial water content [kg H<sub>2</sub>O/kg DM]. For modelling, the freeze-drying equations in Table 1 were tested to select the best model for describing the drying curve of peppers.

### 2.5. Sample preparation

The individual dried pepper samples were ground using the laboratory knife mill (Grindomix GM 200, Retsch, Dusseldorf, Germany). The powdered samples of paprika (particles < 0.2 mm) were then subjected to other tests.

### 2.6. Colour evaluation

The colour of dried pepper was measured using a CR-400 Chromameter (Minolta). The analyses of the colour values were performed three times with each dried pepper sample. Three parameters, L\* (lightness), a\* (redness), and b\* (yellowness), were used to study the colour changes. From the data obtained, colour intensity ( $\Delta E$ ) was

**Table 1**  
Equations applied to drying curves.

Model number	Model name	Model equation	References
1	Henderson and Pabis	$MR = a \cdot \exp(-k \cdot \tau)$	Henderson and Pabis (1961)
2	Logarithmic	$MR = a \cdot \exp(-k \cdot \tau) + b$	Sarimeseli (2011)
3	Newton	$MR = \exp(-k \cdot \tau)$	El-Beltagy, Gamea, and Amer Essa (2007)
4	Page	$MR = \exp(-k \cdot \tau^n)$	Diamante and Munro (1993)
5	Two term	$MR = a \cdot \exp(-k_1 \cdot \tau) + b \cdot \exp(-k_2 \cdot \tau)$	Arslan and Ozcan (2008)
6	Wang and Singh	$MR = 1 + a \cdot \tau + b \cdot \tau^2$	Wang and Singh (1978)

$k$ ,  $k_i$  – drying coefficients [ $\text{min}^{-1}$ ];  $a$ ,  $b$  – coefficients of the equations;  $n$  – exponent;  $\tau$  – time [min].

calculated (Jokić et al., 2009).

### 2.7. L-ascorbic acid determination

The content of L-ascorbic acid in the fresh and dried pepper was determined using Ultra-performance Liquid Chromatography (UPLC) (Spínola, Mendes, Câmara & Castilho, 2012). To prepare the extract for analysis, 0.5 g of fresh and pulped pepper or dried pepper was added to 25 mL of extraction solution (3% MPA – 8% acetic acid – 1 mM EDTA), vortexed in the darkness, and then centrifuged for 10 min at a velocity of 6000 rpm. The supernatants were filtered through 0.22- $\mu$ m PTFE filters (Milipore, USA). An Acquity UPLC system (Waters Corporation, USA) with a Waters Acquity UPLC photodiode array (PDA) detection system and Empower™ software (Waters Corporation, USA) were used to record detection signal and process the peak areas. The L-ascorbic acid in samples was determined by comparison with the retention time of standard and match with the UV absorption spectrum according to the procedure described by Nowacka et al. (2018).

### 2.8. Determination of total phenolics and antioxidant activity

To prepare the extract for analysis, 0.5 g samples of fresh or dried pepper (0.5 g) were extracted for 30 min with 10 mL of 80% ethanol (v/v). The extracts were separated via decantation and the residues were extracted again with 10 mL of 50% ethanol (v/v). Extracts were combined and stored in darkness at  $-20^{\circ}\text{C}$ .

The total phenolics content (TPC) was estimated according to the Folin–Ciocalteu method (Singleton & Rossi, 1965). The amount of total phenolics was expressed as gallic acid equivalents (GAE).

Antiradical activity (AA) was determined using an improved ABTS decolorization assay (Re et al., 1999). Chelating power (CHEL) was studied using the method of Guo, Lee, Chiang, Lin, and Chang (2001). Reducing power (RED) was determined using the method described by Oyaizu (1986). All activities were evaluated in triplicate and expressed as  $\text{EC}_{50}$  – extract concentration provided 50% of activity based on the dose-dependent mode of action.  $\text{EC}_{50}$  value (mg DM/mL) is the effective concentration at which the absorbance was 0.5 for reducing power and was obtained through interpolation from linear regression analysis.

### 2.9. Statistical analysis

Data represent the mean and standard deviation from three independent dryings. Measurement scores were subjected to an analysis of variance (ANOVA). When significant differences in ANOVA were detected, the means were compared using Tukey's test. Statistical analysis was performed at a significance level of  $\alpha = 0.05$  using Statistica 13.0 by Statsoft. A regression analysis was also performed. The coefficient of determination  $R^2$ , root mean square error (RMSE), and the reduced  $\chi^2$  values were calculated (Akpınar, Midilli, & Bicer, 2003).

## 3. Results and discussion

### 3.1. Drying kinetics

The drying curves of pepper are seen in Fig. 1. The initial moisture content of the paprika was 914 g  $\text{H}_2\text{O}$ /kg fresh weight. The addition of CA had no significant effect on the drying curves. Thus, the average values of MR for individual temperatures were obtained. The results showed that both WB and MB reduced the drying time. The shortest drying time (about 290 min) was found for pepper that was blanched using MB and WB and freeze-dried at  $60^{\circ}\text{C}$ , whereas the pepper without pretreatments and dried at same temperature required about 80 min longer drying time. The samples of pepper freeze-dried at  $20^{\circ}\text{C}$  and without blanching required the longest drying time (about 900 min). In this instance blanching reduced drying time by about 250 min. Wang et al. (2017a) showed that blanching of red pepper before air drying

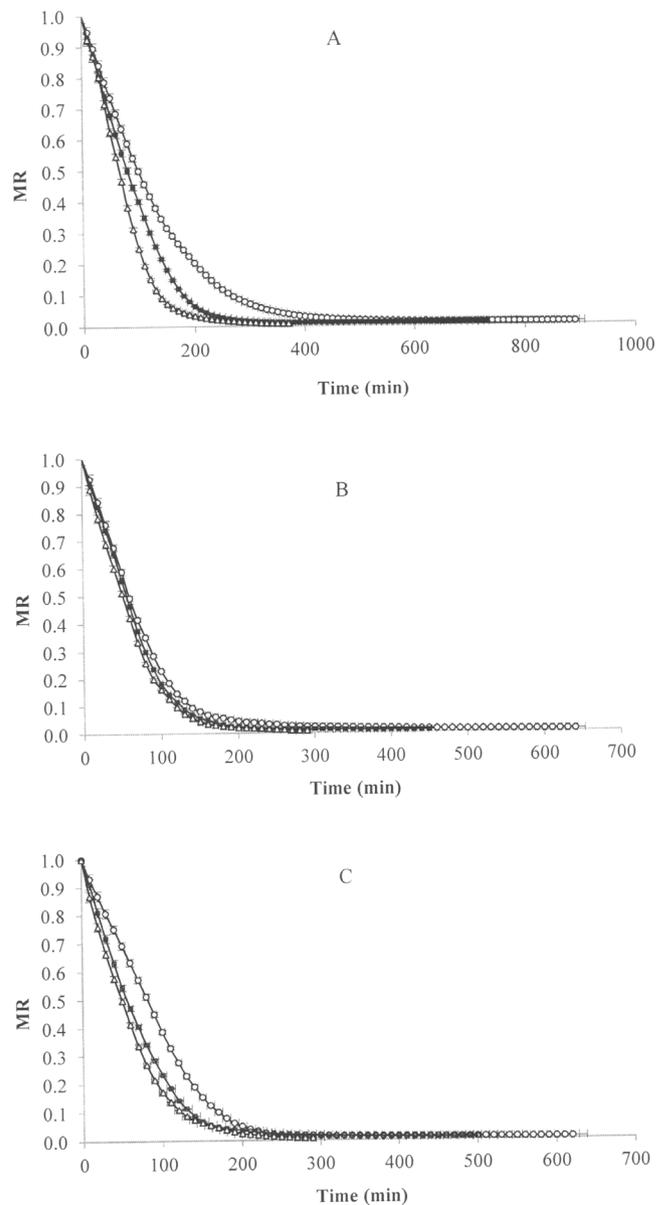


Fig. 1. Drying curves of freeze-dried pepper: A - with and without citric acid, B - microwave blanching, C - water blanching; MR - water ratio, ○–  $20^{\circ}\text{C}$ , ■–  $40^{\circ}\text{C}$ , △–  $60^{\circ}\text{C}$ .

significantly reduced the drying time. Further, an increase of freeze-drying temperature from  $20^{\circ}\text{C}$  to  $60^{\circ}\text{C}$  decreased the drying time about two-fold. This tendency was found both for control and blanched samples. Our previous study also showed similar relation between drying time of cranberries and temperature of heating plates during lyophilization (Rudy et al., 2015). The results of regression analyses for the models that were used to describe the freeze drying kinetics of the control and blanched peppers are presented in Tables 2 and 3. For almost each of the models, a good fit for the experimental data was observed. Only in the case of the model used by Wang and Singh, where the temperature of the heating plates was  $20^{\circ}\text{C}$  and  $40^{\circ}\text{C}$ , this fit was noticeably lower and the lowest values of  $R^2$  and the highest values of RMSE were obtained. Based on the results of regression Page model seems to be the best suited to describe the freeze-drying process of pepper. This model is often used as the best equation to represent the drying kinetics for the freeze-drying and hot air drying of fruits and vegetables (Evin, 2011; Marques & Freire, 2005).

**Table 2**  
Statistical analysis of models describing kinetics of freeze drying of pepper at 20 °C.

Model name	DT (°C)	Sample								
		FD			WB			MB		
		R <sup>2</sup>	RMSE	χ <sup>2</sup>	R <sup>2</sup>	RMSE	χ <sup>2</sup>	R <sup>2</sup>	RMSE	χ <sup>2</sup>
Henderson and Pabis	20	0.99618	0.00222	5.07 10 <sup>-6</sup>	0.98127	0.01057	0.00011	0.98902	0.00503	2.61 10 <sup>-5</sup>
	40	0.98687	0.00966	9.83 10 <sup>-5</sup>	0.99199	0.00369	1.42 10 <sup>-5</sup>	0.98625	0.00662	4.57 10 <sup>-5</sup>
	60	0.97933	0.01140	0.00013	0.99392	0.00267	7.66 10 <sup>-6</sup>	0.98824	0.00549	3.23 10 <sup>-5</sup>
Logarithmic	20	0.99618	0.00222	5.11 10 <sup>-6</sup>	0.98206	0.01013	0.00011	0.98930	0.00490	2.52 10 <sup>-5</sup>
	40	0.98652	0.006985	5.08 10 <sup>-5</sup>	0.99201	0.00369	1.44 10 <sup>-5</sup>	0.98652	0.00649	4.50 10 <sup>-5</sup>
Newton	60	0.98186	0.01001	0.00011	0.99517	0.00212	5.01 10 <sup>-6</sup>	0.99028	0.00453	2.28 10 <sup>-5</sup>
	20	0.99324	0.003938	1.56 10 <sup>-5</sup>	0.97375	0.01483	0.00022	0.98375	0.00745	5.63 10 <sup>-5</sup>
	40	0.98101	0.01027	0.00010	0.98846	0.00532	2.89 10 <sup>-5</sup>	0.98000	0.00963	9.48 10 <sup>-5</sup>
Page	60	0.96898	0.01711	0.00030	0.99209	0.00348	1.25 10 <sup>-5</sup>	0.98381	0.00755	5.9 10 <sup>-5</sup>
	20	0.99919	0.000468	2.24 10 <sup>-7</sup>	0.99731	0.00152	2.38 10 <sup>-6</sup>	0.99622	0.00173	3.09 10 <sup>-6</sup>
	40	0.99796	0.00149	1.24 10 <sup>-6</sup>	0.99793	0.00095	9.48 10 <sup>-7</sup>	0.99818	0.00087	7.97 10 <sup>-7</sup>
Two-factor	60	0.99828	0.00094	9.48 10 <sup>-7</sup>	0.99862	0.00061	3.92 10 <sup>-7</sup>	0.99836	0.00076	6.26 10 <sup>-7</sup>
	20	0.99618	0.00223	5.19 10 <sup>-6</sup>	0.98128	0.01058	0.00012	0.98902	0.00503	2.69 10 <sup>-5</sup>
	40	0.98687	0.00711	5.33 10 <sup>-5</sup>	0.99327	0.00310	1.04 10 <sup>-5</sup>	0.98893	0.00532	3.11 10 <sup>-5</sup>
Wang and Singh	60	0.97933	0.01140	0.00014	0.99392	0.00267	8.25 10 <sup>-6</sup>	0.98824	0.00549	3.47 10 <sup>-5</sup>
	20	0.81165	0.10986	0.01234	0.85045	0.08447	0.00737	0.62518	0.17177	0.03044
	40	0.76205	0.12876	0.01704	0.79490	0.09462	0.00932	0.83881	0.07759	0.00629
	60	0.96037	0.02186	0.00051	0.96019	0.01751	0.00033	0.96622	0.01576	0.00026

DT – drying temperature, FD – freeze drying, WB - water blanching, MB – microwave blanching.

**Table 3**  
Coefficient values in the Page model describing the freeze drying of pepper.

DT (°C)	Sample	Parameter	
		k	n
20	FD	0.0030	1.185
		0.0018	1.370
		0.0013	1.510
40	WB	0.0012	1.461
		0.0044	1.261
		0.0072	1.186
60	MB	0.0030	1.335
		0.0026	1.399
		0.0043	1.310

DT – drying temperature, FD – freeze drying, WB - water blanching, MB – microwave blanching.

**Table 4**  
Effect of freeze drying temperature and pre-treatment on colour parameters of pepper.

Sample	DT (°C)	DC			
		L*	a*	b*	ΔE
FP	–	33.1 ± 1.52 <sup>a</sup>	28.1 ± 0.73 <sup>c</sup>	19.7 ± 0.23 <sup>i</sup>	–
FD	20	39.2 ± 0.26 <sup>def</sup>	34.8 ± 0.43 <sup>f</sup>	27.0 ± 0.35 <sup>ac</sup>	11.3 ± 0.63 <sup>de</sup>
	40	41.1 ± 0.58 <sup>b</sup>	37.2 ± 0.22 <sup>g</sup>	28.8 ± 0.45 <sup>bd</sup>	14.8 ± 0.56 <sup>f</sup>
	60	35.5 ± 0.72 <sup>b</sup>	27.6 ± 0.36 <sup>c</sup>	23.8 ± 0.49 <sup>g</sup>	4.9 ± 0.33 <sup>a</sup>
WB	20	40.6 ± 0.38 <sup>gh</sup>	30.9 ± 0.39 <sup>d</sup>	28.6 ± 0.45 <sup>b</sup>	11.8 ± 0.36 <sup>e</sup>
	40	43.0 ± 0.44 <sup>i</sup>	35.6 ± 0.35 <sup>a</sup>	32.6 ± 0.47 <sup>f</sup>	17.7 ± 0.58 <sup>h</sup>
	60	38.9 ± 0.47 <sup>cde</sup>	25.9 ± 0.60 <sup>b</sup>	27.4 ± 0.40 <sup>a</sup>	10.0 ± 0.27 <sup>c</sup>
MB	20	39.8 ± 0.31 <sup>efg</sup>	32.4 ± 0.49 <sup>e</sup>	27.9 ± 0.38 <sup>ab</sup>	11.2 ± 0.43 <sup>d</sup>
	40	42.4 ± 0.67 <sup>i</sup>	36.1 ± 0.34 <sup>a</sup>	29.7 ± 0.53 <sup>de</sup>	15.5 ± 0.47 <sup>fg</sup>
	60	38.1 ± 0.50 <sup>c</sup>	26.2 ± 0.32 <sup>b</sup>	25.1 ± 0.43 <sup>h</sup>	7.8 ± 0.31 <sup>b</sup>
CA	20	40.1 ± 0.59 <sup>gh</sup>	39.3 ± 0.25 <sup>h</sup>	30.3 ± 0.56 <sup>e</sup>	16.5 ± 0.58 <sup>gh</sup>
	40	43.2 ± 0.45 <sup>i</sup>	42.8 ± 0.23 <sup>i</sup>	32.2 ± 0.23 <sup>f</sup>	21.4 ± 0.71 <sup>i</sup>
	60	38.6 ± 0.49 <sup>cd</sup>	36.1 ± 0.28 <sup>a</sup>	26.4 ± 0.50 <sup>c</sup>	11.4 ± 0.42 <sup>de</sup>

\*The values are expressed as mean ± SD (n = 3); DT – drying temperature, FP – FP – fresh pepper, DC – dimension of colour, FD – freeze dried (without pretreatment), WB – water blanching, MB – microwave blanching, CA – with citric acid addition, The values designated by the different small letters in the columns of the table are significantly different (α = 0.05).

**Table 5**  
Ascorbic acid content, total phenolics content and antioxidant activity of dried pepper.

Sample	DT (°C)	AC (mg/g DM)	TPC (mg GAE/g DM)	RED (EC <sub>50</sub> , mg DM/ml)	AA (EC <sub>50</sub> , mg DM/ml)	CHEL (EC <sub>50</sub> , mg DM/ml)
FP	–	18.62 ± 0.23 <sup>b</sup>	14.3 ± 0.27 <sup>f</sup>	5.4 ± 0.11 <sup>a</sup>	19.3 ± 0.22 <sup>a</sup>	18.7 ± 0.18 <sup>a</sup>
FD	20	17.1 ± 0.65 <sup>d</sup>	12.6 ± 0.32 <sup>e</sup>	6.1 ± 0.18 <sup>b</sup>	21.7 ± 0.47 <sup>c</sup>	20.0 ± 0.22 <sup>b</sup>
	40	16.6 ± 0.54 <sup>df</sup>	12.5 ± 0.41 <sup>e</sup>	6.0 ± 0.21 <sup>b</sup>	22.3 ± 0.39 <sup>c</sup>	22.6 ± 0.34 <sup>c</sup>
	60	13.9 ± 0.44 <sup>abc</sup>	11.8 ± 0.28 <sup>d</sup>	6.7 ± 0.23 <sup>c</sup>	26.1 ± 0.52 <sup>ef</sup>	25.1 ± 0.43 <sup>c</sup>
WB	20	13.9 ± 0.45 <sup>abc</sup>	10.8 ± 0.26 <sup>ab</sup>	7.1 ± 0.16 <sup>d</sup>	26.9 ± 0.61 <sup>f</sup>	26.6 ± 0.21 <sup>g</sup>
	40	13.5 ± 0.43 <sup>ab</sup>	10.6 ± 0.32 <sup>ab</sup>	7.3 ± 0.23 <sup>d</sup>	27.2 ± 0.76 <sup>f</sup>	26.2 ± 0.27 <sup>g</sup>
	60	9.7 ± 0.34 <sup>g</sup>	10.2 ± 0.36 <sup>ab</sup>	7.5 ± 0.26 <sup>d</sup>	29.9 ± 0.94 <sup>g</sup>	28.4 ± 0.29 <sup>h</sup>
MBs	20	15.4 ± 0.46 <sup>ef</sup>	10.9 ± 0.21 <sup>bc</sup>	6.2 ± 0.13 <sup>b</sup>	24.2 ± 0.61 <sup>d</sup>	24.2 ± 0.26 <sup>e</sup>
	40	15.0 ± 0.47 <sup>ce</sup>	11.3 ± 0.29 <sup>c</sup>	6.4 ± 0.19 <sup>b</sup>	25.2 ± 0.63 <sup>de</sup>	24.7 ± 0.34 <sup>ef</sup>
	60	12.4 ± 0.46 <sup>a</sup>	10.5 ± 0.27 <sup>b</sup>	6.8 ± 0.15 <sup>c</sup>	27.4 ± 1.12 <sup>f</sup>	25.4 ± 0.42 <sup>f</sup>
CA	20	17.4 ± 0.56 <sup>d</sup>	12.1 ± 0.35 <sup>c</sup>	6.2 ± 0.08 <sup>a</sup>	20.6 ± 0.69 <sup>b</sup>	23.3 ± 0.38 <sup>d</sup>
	40	17.1 ± 0.55 <sup>d</sup>	12.5 ± 0.23 <sup>c</sup>	6.4 ± 0.20 <sup>ab</sup>	22.7 ± 0.73 <sup>c</sup>	23.5 ± 0.44 <sup>d</sup>
	60	14.7 ± 0.63 <sup>bce</sup>	11.6 ± 0.43 <sup>d</sup>	6.9 ± 0.21 <sup>c</sup>	25.1 ± 0.58 <sup>d</sup>	22.7 ± 0.36 <sup>c</sup>

\*The values are expressed as mean ± SD (n = 3); DT – drying temperature, FP – fresh pepper, FD – freeze dried (without pretreatment), WB – water blanching, MB – microwave blanching, CA – with citric acid addition, AC – L-ascorbic acid content, TPC – total phenolics content, RED – reducing power, ABTS – antiradical activity, CHEL – chelating power. The values designated by the different small letters in the columns of the table are significantly different ( $\alpha = 0.05$ ).

paprika colour quality. Moreover, higher temperature of drying can also increase the rate of carotenoids degradation (Vega-Gálvez, Lemus-Mondaca, Bilbao-Sáinz, Fito, & Andrés, 2008). Dried pepper with the addition of CA was more red and was characterized by the highest values of colour intensity in comparison to dried material without acid addition. Moreover, blanching caused a slight decrease in the redness and increase in the yellowness of pepper, whereas the method of blanching had little or no effect on colour coordinates. Wang et al. (2017b) found similar results after blanching pepper before air drying.

### 3.3. L-ascorbic acid changes

The L-ascorbic acid content (AC) of fresh pepper was 18.6 mg/g DM. L-ascorbic acid can be easily degraded, depending on many factors such as temperature, pH, light, and presence of enzymes and/or oxygen. Thus, many studies on food processes take vitamin C as a quality indicator of the foods (Santos & Silva, 2008). The variations in AC content during pepper drying are shown in Table 5. Freeze-drying the control samples and the samples of pepper with CA caused the lowest decrease in AC when compared to peppers before drying. The maximum AC was obtained after drying the peppers at 20 °C and 40 °C. The increase in the drying temperature from 20 °C to 40 °C had no significant effect on the AC. With an increase in drying temperature up to 60 °C, a decrease in AC was observed (from 14% for samples with CA to 18% for WB pepper). The lowest values of AC were obtained for samples blanched before freeze-drying. However, for the WB samples, the highest decrease in LA was observed. Another study confirmed that blanching the pepper significantly decreased the AC (Castro et al., 2008). A study by Wang et al. (2017a) showed that WB pepper in comparison with MB was characterized by the lowest retention of L-ascorbic acid. We also found positive correlation between the redness of the dried pepper and AC ( $r = 0.820$ ,  $p = 0.001$ ).

### 3.4. Total phenolics content and antioxidant activity

The phenolics content (TPC) of fresh pepper was 14.27 mg GAE/g DM. The variation in the TPC content during pepper drying at different temperatures is shown in Table 5. The freeze-drying caused a decrease in TPC. The maximum TPC was obtained during the drying of untreated pepper at 20 °C and 40 °C, and for samples with CA addition (average 12.4 mg GAE/g DM). With an increase in drying temperature from 40 °C to 60 °C, a decrease in TPC was observed. The lowest values of TPC was obtained for blanched peppers (from 10.2 to 11.3 mg GAE/g DM). The

method of blanching had little effect on TPC. Slightly higher values of TPC were obtained for MB of pepper than for WB fruits. However, the significant differences were obtained only when the temperature of FD was 40 °C. The antioxidant activities (RED, AA, CHEL) of fresh pepper expressed as EC<sub>50</sub> were 5.4, 19.3 and 18.7 mg DM/mL, respectively. The freeze drying caused a decrease in RED, AA, and CHEL. The highest EC<sub>50</sub> values (the lowest antioxidant activity) were obtained for WB and dried pepper (average 7.4, 28.0 and 27.1 mg DM/mL, for RED, AA and HEL, respectively), whereas the lowest EC<sub>50</sub> were found for dried pepper without pretreatments and with the addition of CA (6.4, 23.1 and 22.9 mg DM/mL, for RED, AA and HEL, respectively) (Table 5). In comparison to WB samples, the MB samples of dried pepper were characterized by higher antioxidant activity. Wang et al. (2017a) showed that MB can enhance the antioxidant activity of pepper, whereas WB had an adverse effect because of leaching of hydrophilic antioxidants into the hot water. An increase in drying temperature from 20 °C to 40 °C had no significant effect on antioxidant activity. However, when the drying temperature was 60 °C, a slight but significant decrease in antioxidant activity was observed. Many authors found that the processing temperature significantly influenced the antioxidant capacity of pepper. Rufián-Henares, Guerra-Hernández, and García-Villanova (2013) found that a higher temperature of dehydration exerts a strong effect on the degradation of antioxidant compounds. Zhou et al. (2016) showed that during hot air drying, the total phenolics content and antioxidant activity in red pepper decreased proportionate to increases in the drying temperature.

## 4. Conclusions

Blanching of pepper before freeze-drying significantly reduced the drying time (to approximately 30% of the original), whereas the addition of CA had no significant effect on drying kinetics. An increase in freeze-drying temperature from 20 °C to 60 °C approximately halved the drying time and produced paprika with a lower lightness, redness, and yellowness. The highest values of redness and yellowness of dried product were obtained for pepper with CA addition. Freeze-drying caused a decrease both in TPC and antioxidant activity. The highest decrease was observed for blanched pepper. Taking into account the drying temperature and the methods of pretreatments, the highest quality dried product (redder and with the highest TPC, AA, and L-ascorbic acid content) was obtained when CA was added to the pepper before freeze drying and when the temperature of the process was 40 °C.

## References

- Akpınar, E., Midilli, A., & Bicer, Y. (2003). Single layer drying behaviour of potato slices in a convective cyclone dryer and mathematical modeling. *Energy Conversion and Management*, 44, 1689–1705.
- AOAC International (1990). Method 934.06 moisture in dried fruits. *Official methods of the association of analytical chemists* (15th ed.). Virginia, USA: AOAC.
- Arimboor, R., Natarajan, R. B., Menon, K. R., Chandrasekhar, L. P., & Moorkoth, V. (2015). Red pepper (*Capsicum annuum*) carotenoids as a source of natural food colors: Analysis and stability — a review. *Journal of Food Science & Technology*, 52, 1258–1271.
- Arslan, D., & Ozcan, M. M. (2008). Evaluation of drying methods with respect to drying kinetics, mineral content and colour characteristics of rosemary leaves. *Energy Conversion and Management*, 49, 1258–1264.
- Arslan, D., & Ozcan, M. M. (2011). Dehydration of red bell-pepper (*Capsicum annuum* L.): Change in drying behavior, colour and antioxidant content. *Food and Bioprocess Processing*, 89, 504–513.
- Castro, S. M., Saraiva, J. A., Lopes-da-Silva, J. A., Delgado, I., Van Loey, A., Smout, C., et al. (2008). Effect of thermal blanching and of high pressure treatments on sweet green and red bell pepper fruits (*Capsicum annuum* L.). *Food Chemistry*, 107, 1436–1449.
- Ciurzyńska, A., & Lenart, A. (2011). Freeze-drying - application in food processing and biotechnology - a review. *Polish Journal of Food and Nutrition Sciences*, 61(3), 165–171.
- Ciurzyńska, A., Lenart, A., & Gręda, K. J. (2014). Effect of pre-treatment conditions on content and activity of water and colour of freeze-dried pumpkin. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 59, 1075–1081.
- Condori, M., Echazu, R., & Saravia, L. (2001). Solar drying of sweet pepper and garlic using the tunnel greenhouse drier. *Renewable Energy*, 22, 447–460.
- Dehghannya, J., Gorbani, R., & Ghanbarzadeh, B. (2017). Influence of combined pre-treatments on color parameters during convective drying of Mirabelle plum (*Prunus domestica* subsp. *syriaca*). *Heat and Mass Transfer*, 53(7), 2425–2433.
- Diamante, L. M., & Munro, P. A. (1993). Mathematical modelling of the thin layer solar drying of sweet potato slices. *Solar Energy*, 51, 271–276.
- Dorantes-Alvarez, L., Jaramillo-Flores, E., González, K., Martínez, R., & Parada, L. (2011). *Procedia Food Science*, 1, 178–183.
- Dürüst, N., Sümengen, D., & Dürüst, Y. (1997). Ascorbic acid and element contents of foods of Trabzon (Turkey). *Journal of Agricultural and Food Chemistry*, 45, 2085–2087.
- Dziki, D., Polak, R., Rudy, S., Krzykowski, A., Gawlik-Dziki, U., Różyło, R., et al. (2018). Simulation of the process kinetics and analysis of physicochemical properties in the freeze drying of kale. *International Agrophysics*, 32, 49–56.
- El-Beltagy, A., Gamea, G. R., & Amer Essa, A. H. (2007). Solar drying characteristics of strawberry. *Journal of Food Engineering*, 78, 456–464.
- Evin, D. (2011). Microwave drying and moisture diffusivity of white mulberry: Experimental and mathematical modeling. *Journal of Mechanical Science and Technology*, 25, 2711–2718.
- Fiedor, J., & Burda, K. (2014). Potential role of carotenoids as antioxidants in human health and disease. *Nutrients*, 6, 466–488.
- García-Noguera, J., Oliveira, F. I. P., Weller, C. L., Rodrigues, S., & Fernandes, F. A. N. (2014). Effect of ultrasonic and osmotic dehydration pre-treatments on the colour of freeze dried strawberries. *Journal of Food Science & Technology*, 51, 2222–2227 (the different method of).
- Gomez, R., Pardo, J. E., Navarro, F., & Varon, R. (1998). Colour differences in paprika pepper varieties (*Capsicum annuum* L.) cultivated in a greenhouse and in the open air. *Journal of the Science of Food and Agriculture*, 77, 268–272.
- Guo, J. T., Lee, H. L., Chiang, S. H., Lin, H. I., & Chang, C. Y. (2001). Antioxidant properties of the extracts from different parts of broccoli in Taiwan. *Journal of Food and Drug Analysis*, 9, 96–101.
- Hallmann, E., & Rembiałkowska, E. (2012). Characterisation of antioxidant compounds in sweet bell pepper (*Capsicum annuum* L.) under organic and conventional growing systems. *Journal of the Science of Food and Agriculture*, 92, 2409–2415.
- Henderson, S. M., & Pabis, S. (1961). Grain drying theory. II. Temperature effects on drying coefficients. *Journal of Agricultural Engineering Research*, 6, 169–174.
- Jokić, S., Velić, D., Bilić, M., Lukinac, J., Planić, M., & Bucić-Kojić, A. (2009). Influence of process parameters and pre-treatments on quality and drying kinetics of apple sample. *Czech Journal of Food Sciences*, 27, 88–94.
- Lee, C.-., Oh, H.-., Han, S.-., & Lim, S.-. (2012). Effects of hot air and freeze drying methods on physicochemical properties of citrus 'hallabong' powders. *Food Science and Biotechnology*, 21, 1633–1639.
- Magied, M. M. A., & Ali, M. M. (2017). Effect of drying method on physical properties and bioactive compounds of red chili pepper "*Capsicum annuum* L. *Current Nutrition & Food Science*, 13, 43–49.
- Marques, L. G., & Freire, J. T. (2005). Analysis of freeze-drying of tropical fruits. *Drying Technology*, 23, 2169–2184.
- Martín, A., Hernández, A., Aranda, E., Casquete, R., Velázquez, R., Bartolomé, T., et al. (2017). Impact of volatile composition on the sensorial attributes of dried paprikas. *Food Research International*, 100, 691–697.
- Minguez-Mosquera, M. I., Jaren-Galan, M., & Garrido-Fernandez, J. (1994). Competition between the processes of biosynthesis and degradation of carotenoids during the drying of peppers. *Journal of Agricultural and Food Chemistry*, 42, 645–648.
- Motevali, A., Younji, S., Chayjan, R. A., Aghilinategh, N., & Banakar, A. (2013). Drying kinetics of dill leaves in a convective dryer. *International Agrophysics*, 27, 39–47.
- Nowacka, M., Fijałkowska, A., Dadan, M., Rybak, K., Wiktor, A., & Witrowa-Rajchert, D. (2018). Effect of ultrasound treatment during osmotic dehydration on bioactive compounds of cranberries. *Ultrasonics*, 83, 18–25.
- Oberoi, H. S., Ku, M. A., Kaur, J., & Baboo, B. (2005). Quality of red chilli variety as affected by different drying methods. *Journal of Food Science and Technology-mysore*, 42, 384–387.
- Orak, H. H., Aktas, T., Yagar, H., Isbilir, S. S., Ekinci, N., & Sahin, F. H. (2012). Effects of hot air and freeze drying methods on antioxidant activity, colour and some nutritional characteristics of strawberry tree (*arbutus unedo* L) fruit. *Food Science and Technology International*, 18, 391–402.
- Oyaizu, M. (1986). Studies on products of browning reaction - antioxidative activities of products of browning reaction prepared from glucosamine. *The Japanese Journal of Nutrition and Dietetics*, 44, 307–315.
- Park, J. H., & Kim, C. S. (2007). The stability of colour and antioxidant compounds in paprika (*Capsicum annuum* L.) powder during the drying and storing process. *Food Science and Biotechnology*, 16, 187–192.
- Prosapio, V., & Norton, I. (2017). Influence of osmotic dehydration pre-treatment on oven drying and freeze drying performance. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 80, 401–408.
- Re, R., Pellegrini, N., Proteggente, A., Pannala, A., Yang, M., & Rice-Evans, C. (1999). Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radical Biology and Medicine*, 6, 1231–1237.
- Rodríguez-Amaya, D. B., Kimura, M., Godoy, H., & Amaya-Farfan, J. (2008). Updated Brazilian database on food carotenoids: Factors affecting carotenoid. *Journal of Food Composition and Analysis*, 21, 445–463.
- Rudy, S., Dziki, D., Krzykowski, A., Gawlik-Dziki, U., Polak, R., Różyło, R., et al. (2015). Influence of pre-treatments and freeze-drying temperature on the process kinetics and selected physico-chemical properties of cranberries (*Vaccinium macrocarpon* Ait.). *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 63, 497–503.
- Rufián-Henares, J.Á., Guerra-Hernández, E., & García-Villanova, B. (2013). Effect of red sweet pepper dehydration conditions on maillard reaction, ascorbic acid and antioxidant activity. *Journal of Food Engineering*, 118, 150–156.
- Santos, P. H. S., & Silva, M. A. (2008). Retention of vitamin C in drying processes of fruits and vegetables - a review. *Drying Technology*, 26, 1421–1437.
- Sarimeseli, A. (2011). Microwave drying characteristics of coriander (*Coriandrum sativum* L.) leaves. *Energy Conversion and Management*, 52, 1449–1453.
- Singleton, V. L., & Rossi, J. A. (1965). Colorimetry of total phenolics with phosphomolybdic-phosphotungstic acid reagents. *American Journal of Enology and Viticulture*, 16, 144–158.
- Sogi, D. A., Siddiqi, M., Greiby, I., & Dolan, K. D. (2013). Total phenolics, antioxidant activity, and functional properties of 'Tommy Atkins' mango peel and kernel as affected by drying methods. *Food Chemistry*, 141, 2649–2655.
- Spínola, V., Mendes, B., Câmara, J. S., & Castilho, P. C. (2012). An improved and fast UHPLC-PDA methodology for determination of L-ascorbic and dehydroascorbic acids in fruits and vegetables. evaluation of degradation rate during storage. *Analytical and Bioanalytical Chemistry*, 403(4), 1049–1058.
- Topuz, A., Feng, H., & Kushad, M. (2009). The effect of drying method and storage on color characteristics of paprika. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 42, 1667–1673.
- Vega-Gálvez, A., Di Scala, K., Rodríguez, K., Lemus-Mondaca, R., Miranda, M., López, J., et al. (2009). Effect of air-drying temperature on physico-chemical properties, antioxidant capacity, colour and total phenolic content of red pepper (*Capsicum annuum*, L. var. *Hungarian*). *Food Chemistry*, 117, 647–653.
- Vega-Gálvez, A., Lemus-Mondaca, R., Bilbao-Sáinz, C., Fito, P., & Andrés, A. (2008). Effect of air drying temperature on the quality of rehydrated dried red bell pepper (var. lamuyo). *Journal of Food Engineering*, 85(1), 42–50.
- Wang, J., Fang, X.-M., Mujumdar, A. S., Qian, J.-Y., Zhang, Q., Yang, X.-H., et al. (2017b). Effect of high-humidity hot air impingement blanching (HHAIB) on drying and quality of red pepper (*Capsicum annuum* L.). *Food Chemistry*, 220, 145–152.
- Wang, C. Y., & Singh, R. P. (1978). Use of variable equilibrium moisture content in modeling rice drying. *Transactions of the ASAE*, 11, 668–672.
- Wang, J., Yang, X., Mujumdar, A. S., Wang, D., Zhao, J., Fang, X., et al. (2017a). Effects of various blanching methods on weight loss, enzymes inactivation, phytochemical contents, antioxidant capacity, ultrastructure and drying kinetics of red bell pepper (*Capsicum annuum* L.). *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 77, 337–347.
- Zhou, L., Cao, Z., Bi, J., Yi, J., Chen, Q., Wu, X., et al. (2016). Degradation kinetics of total phenolic compounds, capsaicinoids and antioxidant activity in red pepper during hot air and infrared drying process. *International Journal of Food Science and Technology*, 51, 842–853.