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THE INFLUENCE OF THE HEATING AGENT TEMPERATURE ON THE KINETICS OF THE CONVECTIVE DRYING PROCESS AND THE CONTENT OF BIOACTIVE COMPOUNDS IN APPLE POMACE

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Abstract. An environmentally ecological approach to the reuse of agro-industrial products in the food industry involves extracting biologically active substances or using them as supplementary products rich in dietary fibers. Apple pomace obtained after juice extraction presents a product rich in biologically active substances, soluble and insoluble fibers, organic acids, and minerals. Convective drying of apple pomace is an alternative method for long-term preservation by maintaining the biologically active substances. The research aimed to study the influence of temperature in the range of 60-80 °C on the drying of apple pomace to a final moisture content of 12.0±0.13% using the convective method, and the effect of temperature on the yield of bioactive compounds and antioxidant activity in Gold Delicious apple pomace. Changing the air temperature during convective drying from 60 to 80 °C reduced the drying time by 1.6 times and demonstrated a 1.4-fold increase in drying rate. The increase in the thermal agent's temperature led to an increase in the kinetic characteristic values; K_I values increased by 4%, and K_{II} values increased by 44%. Spectrophotometric analysis demonstrated that apple pomace dried at a temperature of 70 °C had the highest total content of polyphenols, tannins, and carotenoids. Thus, the convective drying method of apple pomace in the temperature range of 60-80 °C exerts a significant influence on the composition of biologically active compounds in the apple pomace composition.

Keywords: *apple pomace, convective drying, temperatures, extracts, biologically active compounds, polyphenols, caratenoids, antioxidant activity.*

Rezumat. O direcție ecologică de reutilizăre a produselor agro-industriale în industria alimentară este extragerea substanțelor biologic active sau utilizarea lor ca produse suplimentare bogate în fibre alimentare. Tescovina de mere obținută după stoarcerea sucului prezintă un produs bogat atât în substanțe biologic active cât și în fibre solubile și insolubile, acizi organici și minerale. Uscarea convectivă a tescovinei este o metodă alternativă de conservare pe termen îndelungat prin păstrarea substanțelor bilogic active.

Scopul cercetării a fost de a studia influența temperaturii în intervalul de 60-80 °C la uscarea tescovinei până la umiditatea finală 12,0±0,13% prin metoda convectivă, și influența temperaturii asupra randamentului de compuși bioactivi și activității antioxidante în tescovina de mere Gold Delicios. Modificarea temperaturii aerului pe parcursul uscării convective de la 60 la 80 °C a redus durata de uscare de 1,6 ori, și a demostrat o creştere a vitezei de uscare de 1,4 ori. Creșterea temperaturii agentului termic a condus la creșterea valorilor caracteristicilor cinetice în cazul coeficientului K_I valorile au crescut cu 4%, iar pentru K_{II} cu 44%. Analiza spectrofotometrică a demonstrat ca în tescovina de mere uscată la temperatura de 70 °C a avut cel mai ridicat conținut total de polifenoli, taninuri și caratenoizi. Astfel, metoda convectivă de uscare a tescovinei de mere în intervalul de temperaturi 60-80 °C manifestă o influență marcantă asupra compoziției compușilor biologic activi din compoziția tescovinei de mere.

Cuvinte cheie: *tescovina de mere, uscare convectivă, temperature, extracte, compuși biologic activi, polifenoli, caratenoizi, activitate antioxidantă.*

1. Introduction

One of the priority tasks for the reuse of agro-industrial products would be their utilization for extracting biologically active substances present in them or for use as supplements to improve food products and create food items with valuable nutritional properties for human health [1]. Apple pomace is obtained after squeezing the juice from fruits, which constitutes 30% of the residual mass. These agro-industrial wastes present a homogeneous mass, composed of the fruit's peel, pulp, and seed chamber, with a light brown color, a pleasant apple scent, and a slightly sour taste. Apple pomace is rich in polyphenols, carotenoids, flavonoids, fatty acids, vitamin C, and dietary fibers, which would allow its use in producing new food products in the bakery, dairy, or confectionery sectors [2]. The use of apple pomace in the diet is attributed to its therapeutic benefits on the body. The most important active substance contained in apples is pectin. Thanks to pectin, apples can combat the effects of diarrhea, gastritis, or colitis, reduce cholesterol, stabilize glucose levels, address various skin eruptions, and contribute to wound healing by restoring tissue elasticity [2,3].

For long-term use of apple pomace, it is necessary to preserve it while maintaining its biologically active substances. Drying is one of the widely used methods for preserving horticultural products [4]. Through drying, water is removed until a final concentration is reached, thus ensuring the microbial stability of the product and minimizing changes in chemical and physical composition during storage. The convective drying method presents an alternative for preserving pomace by transferring heat to the drying product through the thermal agent [5]. The hot air drying process involves both heat and mass transfer, with water diffusing out of horticultural products. The diffusion process of moisture is a three-dimensional complex of food at the surface, being a key factor in controlling the drying rate, where understanding the phenomena that occur in food, especially heat and mass transfer, is necessary [6]. This is due to the dependence between the total energy of diffusion, temperature, time, and air velocity [7]. Drying is not only a preservation method but also offers other advantages for food products undergoing this process: ease of packaging, transportation, and storage due to reduced weight, avoidance of costly refrigeration systems [8]. The degree of contraction and damage to the internal structure of plant tissues depends on the drying method applied and the parameters underlying it: temperature and air velocity.

Convective drying is a frequently used method for dehydrating food products, characterized as a method requiring high temperature and air velocity, with the finished product having low porosity, high density, pleasant appearance and color, and preservation of biologically active components [9]. Researching the drying process of pomace is necessary for understanding the heat and mass transport mechanism and is a prerequisite for the mathematical description of the entire process. Modeling the process, calculating kinetic coefficients, and constructing drying curves at various temperatures and drying rates play an important role in optimizing or controlling the industrial drying conditions of pomace [10].

The aim of the research was to analyze the drying kinetics of apple pomace depending on the thermal agent's temperature and the influence of heat treatment on the content of biologically active compounds and antioxidant activity.

2. Materials and Methods

2.1 Preparation of samples for drying

The harvest of "Golden Delicious" apples used for the research was collected in the fall of 2021 from "AgroProdusct" S.R.L, Briceni, Colicăuți commune, (48°18′36″N 27°8′54″E), Republic of Moldova, which has apple orchards covering over 200 hectares, and stored until the spring of 2022. Apple pomace for drying was obtained after squeezing the juice from Golden Delicious apples. After juice extraction, the pomace was blanched in a 0.2% (anhydrous) citric acid solution for 10 minutes to inhibit enzyme activity and oxidation processes that could alter the properties of the pomace. Subsequently, the pomace was pressed at a temperature of 25 ± 1 °C and arranged on a tray in a single layer for convective drying at different temperatures of the thermal agent.

2.2 Drying of apple pomace

The study of the drying kinetics of apple pomace as a function of the thermal agent's temperature was carried out in a convective drying installation developed within the Department of Mechanical Engineering, Technical University of Moldova (UTM), with heated air as the thermal agent [11]. The thermal agent's temperature was set at 60°C, 70°C, and 80°C, with an air velocity of 1.5±0.1 m/s.

A sample of apple pomace weighing 100 g and with a moisture content of 85.5±2% was placed in the drying chamber on a tray. The parameters of mass reduction were recorded every 5 minutes, with the ambient parameters being an air temperature of 23±1°C and relative humidity of 57±2%. The sample was dried until reaching a final moisture content of 12.0±0.05%. The equilibrium moisture content (u_{ech}) of apple pomace was calculated using Filonenko's formula [12] and amounted to 10.82%.

The dryer was connected to a computer that allowed automatic data recording. The obtained results were processed using graphical and mathematical methods, based on which drying curves and drying rates of apple pomace were constructed depending on the thermal agent's temperature [11]. Based on the obtained data, drying curves *U=f(τ)* and drying rate curves $\frac{dU}{d\tau} = f(U)$ were constructed and analyzed.

The drying coefficients for the first and second periods K_I and K_{II} were calculated according to formulas 1 and 2 [13]:

$$K_I = \frac{du / d\tau}{A \cdot (x_S - x_0)} \tag{1}$$

$$K_{II} = \frac{du / d\tau}{u'_{cr} - u_{ech}}, \quad (2)$$

where: A – the contact area of the apple core with the thermal agent, m^2;
$du/d\tau$ – drying speed in the first period, %/s;
x_0 and x_s the moisture content of fresh and saturated air, kg/kg dry air;
$u'_{cr} - u_{ech}$ - critical and equilibrium humidity, %.

The initial and final moisture content of apple pomace were experimentally determined by drying in an oven at 105.0 ± 0.1°C to a constant mass, according to ISO 1026:1982 [14].

After convective drying, the pomace was ground to a particle size of 140±10μ, sieved, packaged, and stored in the dark at room temperature. The influence of temperature on the biological value of apple pomace, especially the total content of polyphenols, tannins, carotenoids, and antioxidant activity, was evaluated.

2.3. The total content of polyphenols and tannins based on the Folin–Ciocalteu reagent

The total polyphenol content (TPC) and tannin content (TC) were determined according to the Bouyahya method with some modifications [15]. Extracts were prepared as follows: 1 g of dried apple pomace samples were weighed, dissolved in 10 mL of 60% (*v/v*) ethanol solution, sonicated in an ultrasonic bath at room temperature for 15 minutes. After sonication, the extracts were centrifuged at 5000 rpm for 10 minutes, filtered, and stored in tubes under refrigeration.

TPC was determined by adding 0.2 mL of apple pomace extract to a test tube, along with 1 mL of Folin-Ciocalteu solution and 3 mL of sodium carbonate solution (20%). The resulting mixture was allowed to react for one hour in the dark. The absorbance of the mixture was determined at 765 nm. The result was expressed in milligrams of gallic acid equivalents (mg GAE) per 100 mg of dried pomace (mg GAE/100 g DW).

The total tannin content was determined based on the method of Waterman and Mole [16] using the Folin–Ciocalteu reagent. The absorbance of the mixture was read at a wavelength of 750 nm and expressed in milligrams of tannic acid equivalents (mg TAE) per 100 g of dried pomace (mg TAE/100 g DW).

2.4. Total carotenoid content

The total carotenoid content (TCC) was determined based on the method described by Ghendov-Mosanu et al. [17]. 2 g of dried sample were dissolved in 25 mL of a solution (methanol/ ethyl acetate/petroleum ether, 1/1/1, *v/v/v*). The mixture was subjected to ultrasound-assisted extraction (amplitude - 100%; frequency - 37 kHz) for 15 minutes at room temperature. The extracts were then centrifuged at 5000 rpm for 10 minutes. TCC was performed in duplicate and stored in dark bottles in the refrigerator. The extracts were analyzed using a spectrophotometer at a wavelength of 450 nm and expressed in mg/100 g DW of apple pomace [17].

2.5. Antioxidant activity based on the DPPH• radical reagent

The method for determining the antioxidant activity (AA) in 60% (*v/v*) hydroalcoholic extracts of apple pomace was carried out based on the method described by Paulpriya et al. [18]. The results were expressed in micromoles Trolox equivalent (TE) per 100 g of dry weight

(DW) of apple pomace powder (μmol TE/100 g DW) using the calibration curve (0-500 μmol/L, R^2=0.9992) with Trolox.

2.6 Statistical data analysis

All calculations were performed using Microsoft Office Excel 2010 (Microsoft, Redmond, WA, USA). The data obtained in this study are presented as mean values ± standard error of the mean calculated from three parallel experiments. The comparison of average values was conducted using the one-way analysis of variance (ANOVA) with Tukey's test at a significance level of $p \leqslant 0.05$. This analysis was carried out using the Stat graphics program Centurion XVI 16.1.17 (Stat graphics Technologies, Inc., The Plains, VI, USA).

3. Results and Discussions

Drying represents an efficient method of preservation for fruits. Drying can maintain the quality of finished products, but the decisive factor in this process is the chosen method, the equipment, and the parameters of the thermal agent. Various studies on fruit preservation through drying report high product quality, low microbial contamination, and the preservation of functional bioactive substances. The most common drying method is convective drying [19].

Figure 1 shows the drying curves of apple pomace at temperatures of 60, 70, and 80 °C, and with a thermal agent velocity of 1.5±0.1 m/s. Analyzing the drying curves of apple pomace, a decrease in drying time is observed as the thermal agent temperature increases. At 60°C, the drying time is 230 minutes, reducing to 190 minutes at 70°C, and further to 145 minutes at 80°C. Thus, increasing the air temperature from 60 to 80°C reduced the drying time by 1.6 times. Additionally, there was a reduction in drying time of 15.38% in the first period and 45.45% in the second period, Table 1.

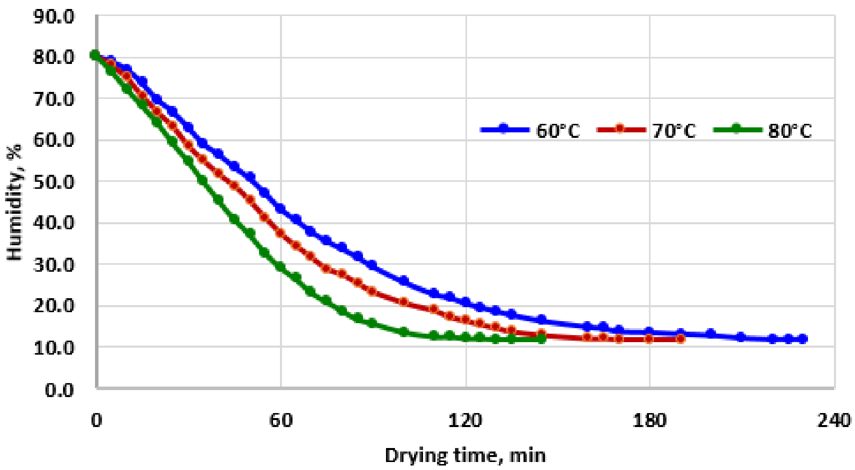

Figure 1. Drying curves of apple pomace at different thermal agent temperatures.

Some bibliographic sources mention that during the drying of plum pomace in the temperature range of 60-80°C, the drying time was reduced by 50%, which influenced the content of acids and the structure of the pomace [20]. Research on drying Baru fruits to achieve a moisture content of 0.065 ± 0.018 (db) resulted in drying times of 266.3, 166.9, 30.8, and 22.8 hours at drying temperatures of 40, 60, 80, and 100°C, respectively. Similarly, a dependence between increasing thermal agent temperature and reduced drying time was observed [21]. A similar relationship between increased thermal agent temperature and decreased drying time was also observed in the drying of grape seeds. Increasing the thermal agent temperature from 60-100°C led to a 1.4 times reduction in the drying process [22].

Convective drying studies on grape pomace demonstrated that drying the pomace at 35°C for 12 hours, 50°C for 5 hours, and 70°C for 3 hours reduced the initial moisture content of the skin from 63.58 ± 3.29% to 6.99 ± 0.83%, and of the pomace from 49.36 ± 3.45% to 8.32 ± 0.96%. In these parameters, a similar dependence between increasing thermal agent temperature and reduced drying time was observed [23]. Literary sources suggest that modern drying conservation technologies for food products reduce energy consumption by up to 80% and improve drying efficiency by up to 26.5% in terms of reduced drying time [24]. Figure 2 represents drying rate curves of apple pomace at different thermal agent temperatures.

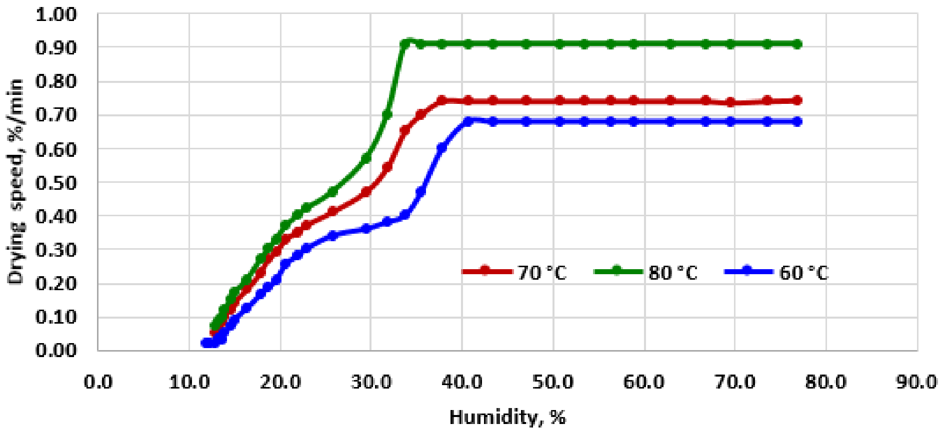

Figure 2. Curves of the drying speed of apple pomace at different temperatures of the thermal agent.

The analysis of the drying rate curves of apple pomace using convective heat input demonstrates the occurrence of mass transfer mechanism in the drying processes. Some sources also report a variation in mass transfer with temperature variation in various food products [25]. The drying rate curves showed the presence of a constant drying rate period (the first period) and a variable drying rate period (the second period).

Upon analyzing the drying rate curves, it was observed that increasing the thermal agent temperature from 60 to 80°C resulted in an increase in drying rate during the first period as follows: 0.66%/min (60°C), 0.72%/min (70°C), and 0.90%/min (80°C). Thus, the drying rate increased by 1.36 times. The change in drying temperature did not significantly influence the overall nature of the drying rate curves. Analyzing the graphs of the drying rate curves during the variable drying rate period revealed a second critical point that shifted from 34% to 26%. Based on the graphs of the drying curves and the drying rate curve, the kinetic characteristics of the drying process were calculated and are presented in Table 1.

The results from Table 1 indicate that with the increase in the drying agent temperature from 60 to 80 °C, there is an increase in the values of kinetic characteristics. In the case of the coefficient K_I, the values increased by 4%, and for K_{II} by 44%, thus demonstrating a significant influence on the variable drying rate period (the second period). It is noted that during convective drying, the removal of moisture from apple pomace is hindered due to the opposing orientation of the temperature and humidity gradient. Other sources in the literature also mention that during the drying of apricots, the constant drying rate in the first period increased depending on the increase in the drying agent temperature (60-100°C) by 1.4 times, while in the second period, it increased by 2.77 times, indicating an intensification of mass and heat exchange [26]. When drying cherries, changing the temperature of the drying agent from 60-100°C influenced the values of the drying constants according to the exponential law, demonstrating an increase in the rate constant in the first K_I period by 3.2 times, and in the second K_{II} period increased 4.7 times [13].

Table 1

Kinetic characteristics of the convective drying process of apple pomace *

Temperature of drying agent, °C	Drying rate in the first drying period, $(du/d\tau)_I$, %/min	Drying coefficient K_I, %/(m²·s·kg/kg aer uscat)	Drying coefficient $K_{II} \cdot 10^4$, s^{-1}	Drying time in the first period τ_I, min	Drying time in the second period τ_{II}, min	Total drying time τ_{total}, min
60	0.66	45.8	3.32	65	165	230
70	0.72	46.5	3.68	60	130	190
80	0.90	47.7	4.81	55	90	145

**p≤0.05*

Apple pomace is a rich source of bioactive compounds and beneficial nutrients for health, exhibiting antioxidant, anti-inflammatory, antibacterial, and antiviral activities. Daily consumption of apple pomace in the diet has a beneficial influence on digestion enzymes, intestinal microbiota, and plasma cholesterol and triglyceride levels [27].

The content of bioactive compounds in dried apple pomace samples at different drying agent temperatures was determined, as shown in Table 2. It was found that increasing the drying agent temperature from 60 to 80 °C resulted in the following values of TPC: 611.44 mg GAE/100 g DW (60°C), 728.82 mg GAE/100 g DW (70°C), and 589.93 mg GAE/100 g DW (80°C), demonstrating the highest extraction yield of TPC at the temperature of 70°C. High temperatures may have led to cellular wall damage, triggering the release of enzymes such as polyphenol oxidase and peroxidase, contributing to the reduction in phenolic compound content. High temperatures can cause irreversible changes in the chemical structure of heat-sensitive phenolic compounds. These changes affect the reactivity of aromatic rings, which interact with the Folin-Ciocalteu reagent, explaining the lower retention of TPC observed at the highest tested temperature [16]. Similarly, Vega-Galvez et al. [28] reported that drying Granny Smith apple pieces at temperatures of 40, 60, and 80°C and at a drying agent velocity of 1.5 m/s caused a decrease in polyphenol content from 44.82 GAE/100 g DW (60°C) to 27.04 GAE/100 g DW (80°C). Gumul et al. reported TPC in apple pomace as 89.39 mg GAE/100 g DW [29]. Persic et al. reported total phenolic content in the range of 19–50 mg GAE/100 g DW [30]. Similar trends have been noted for CTC with values of 66.14 mg TAE/100 g DW (60°C), 78.91 mg TAE/100 g DW (70°C), and 63.54 mg TAE/100 g DW (80°C). As with polyphenols, the highest yield is recorded for the temperature of 70°C, and the lowest for 80°C. The concentration of tannins determined in apple pomace corresponds to literature data, which mention tannin contents ranging from 29.11 to 73.4 mg TAE/100 g DW, depending on the tested apple variety [31]. Tannins can be found in many fruits, with reported values ranging between 238-275 mg TAE/100 g product, varying depending on the variety, fruit parts used, maturity stage, processing conditions, presence of other compounds, as well as the types and tests for tannin analysis [32,33]. In our opinion, TPC and TC in apple pomace can be influenced by extraction conditions and the different ways in which the results were expressed [34].

The TCC registered values of 3.19 (60°C), 4.93 (70°C), and 3.66 mg/100 g DW (80°C), as shown in Table 2. Based on the presented results, the highest yield of bioactive compounds was recorded at the temperature of 70°C. In a study by Raut et al., it was demonstrated that TCC in dried carrot slices through the convective method had the following values: 0.63 mg/100 g DW (50°C), 0.69 mg/100 g DW (60°C), and 0.55 mg/100 g DW (70°C). It was mentioned that the carotenoid content decreases if the samples are exposed to prolonged drying or higher drying temperatures of over 70°C. This phenomenon can be explained by the fact that convective drying at lower temperatures over an extended period exposes carotenoids to oxygen, causing extensive degradation. Additionally, lipoxygenase and peroxidase enzymes are responsible for oxidative degradation of carotenoids [35]. The same values of total carotenoid concentration in apple pomace were recorded by other authors, with values of 14.5 mg/100 g for Berzukroga yellow apples and 5.1 mg/100 g for Bernu Prieks apples [36]. In the case of carotenoid content, for most vegetables and fruits, drying usually results in a loss of 10-20%. Exposure to air or high temperatures facilitates oxidation and isomerization reactions of trans-carotenoids, and lipoxygenase enzymes form reactive radicals that destroy carotenoids [37].

Table 2

The content of biologically active substances in apple pomace

Temperature, °C	Total polyphenol content, mg GAE/ 100 g DW	Tannin content, mg TA/ 100 g DW	Total carotenoid content, mg/100g DW	Antioxidant activity (DPPH), μmol TE/g DW
80°C	586.9±22.4	63.54±7.40	3.66±0.64	62.45±21.2
70°C	728.8±28.5	78.91±9.28	4.93±0.43	62.90±18.05
60°C	611.4±26.4	66.14±9.18	3.19±1.20	74.94±18.06

The AA of apple pomace is directly influenced by the presence of bioactive compounds. The results of the analysis of dried apple pomace extracts in the temperature range of 60-80°C recorded values ranging from 74.94 to 62.98 μmol TE/g DW of DPPH inhibited free radicals. Other authors have demonstrated that in Malus Domneasca apple pomace, the AA values ranged between 0.77 and 0.91 mg Trolox/g [38]. In another study, it was shown that the DPPH radical scavenging activity was significantly reduced in potato waste with increasing drying temperature, with values of 14.26 ± 0.06 μM TE/mg DW at 40°C compared to 08.61 ± 0.01 μM TE /mg DW at 120°C [39]. Similarly, the influence of temperature on AA was demonstrated in lemon residues exposed to drying temperatures from 50°C to 110°C, resulting in a significant increase ($p<0.05$) in the percentage of DPPH radical inhibition. Higher inhibition percentages were recorded at a drying temperature of 50°C, with maximum inhibition values of 73.73±1.12% and 50.07±5.15% at 110°C. Both values obtained at the end of the drying process are characterized in that at 50°C they are higher based on the compounds eriocitrin and hesperidin, while at 110°C they are based on hesperidin compounds [40]. Additionally, Gorjanović et al. reported high values of antioxidant activity in dried apple pomace, with results ranging from 0.1 to 4.5 mmol TE/100 g DW. It should be noted that the antioxidant potential of apple pomace is not solely the result of the presence of polyphenols but is also influenced by other compounds with antioxidant properties (such as vitamin C, E, β-carotene, etc.) [41].

Thus, the convective drying of apple pomace at different temperatures exhibits a significant influence on the composition of bioactive compounds and antioxidant activity.

The research results have demonstrated that by increasing the drying temperature from 60 to 80°C, the drying time is reduced by 1.6 times. An increase of 1.4 times in the drying rate was observed in the first drying period. Drying rate curves showed the presence of a constant drying rate period (first period) and a variable drying rate period (second period). It was found that increasing the drying temperature from 60 to 80°C led to a reduction in the drying time in the first period by 15.38% and in the second period by 45.45%. The increase in the drying agent temperature led to an increase in the values of kinetic characteristics. For the K_I coefficient, the values increased by 4%, and for K_{II}, by 44%. It was demonstrated that the drying temperature influenced the biological value and antioxidant activity of dried apple pomace. The highest values of the total carotenoid content were obtained for higher drying temperatures, decreasing at lower temperatures. This dependence is due to the prolonged drying time and the influence of factors that destroy carotenoids. The same dependence was observed for the total polyphenol and tannin contents. The highest extraction efficiency of bioactive substances was recorded at a drying temperature of 70°C: TPC - 728.82 mg GAE/100 g DW, TC - 78.91 mg TAE/100 g DW, TCC - 4.93 mg/100 g DW. Antioxidant activity values ranged from 62.45 to 74.94 μmol TE/g DW. Therefore, the parameters of convective drying need to be chosen considering the influence of both the drying agent temperature and the drying time on the yield of bioactive substances. Drying at a temperature of 70°C may represent a compromise, providing a good balance between drying time and maintaining a high yield of polyphenols, carotenoids, and tannins in apple pomace. The dried pomace obtained while preserving bioactive substances can be used in the food industry to manufacture various new products with high biological value.

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