Zeszyty Problemowe Postępów Nauk Rolniczych
nr 600, 2020, 31–41
DOI 10.22630/ZPPNR.2020.600.4

ANALYSIS OF PHYSICOCHEMICAL PROPERTIES OF BEETROOT DRIED BY MICROWAVE-VACUUM METHOD

Aleksandra Matys✉, Artur Wiktor, Dorota Witrowa-Rajchert
WULS-SGGW, Institute of Food Sciences

Summary. The aim of the work was to analyze the properties of dried beetroot obtained by the microwave-vacuum method, using various drying parameters (pressure, microwave power, time), and by the convective method, using air at a temperature of 70°C and a flow velocity of 2 $m \cdot s^{-1}$. Beetroots of the "Wodan" variety were used in the research. The dry matter content, density, hygroscopicity, color, content of betalains and polyphenols were determined in the dried material. Based on selected quality indicators, the process parameters, guaranteeing the obtaining dried material with the best properties, were determined. The dried material, obtained by the microwave-vacuum method, was characterized by lower density, higher hygroscopicity, darker and more similar color to the raw material, higher content of betalain and polyphenols in comparison to the dried material obtained by the convective method. The use of microwaves with a power of 200 W and a pressure of 45 hPa led to obtaining dried beetroot with the best properties.

Key words: microwave-vacuum drying, density, hygroscopicity, color, betalains, polyphenols

INTRODUCTION

Microwave-vacuum drying is one of the most effective methods of food preservation, which consists of dehydrating it using microwaves and reduced pressure [Jałoszyński et al. 2011]. This process is characterized by both fast energy exchange, resulting from the use of microwaves, and quick mass exchange at a relatively low temperature, which is associated with the use of reduced pressure [Kieca and Musielak 2010, Musielak and Kieca 2014].

Aleksandra Matys https://orcid.org/0000-0002-6473-7218; Artur Wiktor https://orcid.org/0000-0003-4058-6010; Dorota Witrowa-Rajchert https://orcid.org/0000-0002-0937-3204
✉ aleksandra_matys@sggw.edu.pl

Microwave energy is supplied to the drying material by radiation and can be absorbed, reflected or dispersed there. Food consists of polar compounds with specific dipole moments (e.g. water) on which the microwave drying mechanism is based. The occurrence of a variable electromagnetic field and the impact of microwave energy on polar compounds cause vibrations and rotations of dipoles and acceleration of ions' movements in the direction parallel to the action of the electromagnetic field. The paths of ions and dipoles and one dipoles with the other cross, hence there are numerous collisions during which kinetic energy is transferred. Intermolecular friction arises, leading to heat generation [Nowacka et al. 2012]. Heat-transferring microwaves penetrate simultaneously and directly each particle of the dried material, thanks to which the drying time is shorter and the biochemical changes are significantly limited [Bondaruk and Markowski 2005]. The heating of the dried material and the reduction of its water content take place inside it, not on the surface [Jałoszyński et al. 2008, Székely et al. 2019]. Depending on the used wave frequency, a different scale of its penetration into the material is observed. The lower the frequency, the deeper the wave penetrates [Nowacka et al. 2012]. The energy supply in the microwave-vacuum process is not affected by barriers related to specific thermal conductivity [Ambros et al. 2018]. The dynamic release of energy causes intensive water evaporation, which influences the formation of a porous structure, thanks to which the effect of material shrinkage is limited [Jałoszyński et al. 2011].

Boiling of water under reduced pressure conditions takes place at lower temperatures than in the case of this phenomenon at atmospheric pressure. By applying adequately reduced pressure, it is possible to prevent the dried material from overheating [Orikasa et al. 2018]. Reduced pressure, which increases the expansion of the dried material, contributes to the phenomenon of "puffing", which protects the material from collapsing [Piotrowski and Figiel 2005, Figiel and Michalska 2017]. The structure of "puffing" allows the acceleration of mass transfer and thus contributes to the intensification of drying.

The combination of these two factors allows to reduce the drying time and obtain products of very good quality, mainly due to the reduction of oxidation of the ingredients contained in the product [Jałoszyński et al. 2011]. Correctly adjusted conditions for the microwave-vacuum drying process, such as microwave power or pressure, can contribute to obtaining the appropriate texture and preserving a high level of important nutrients, aromas and flavors as well as the color of the obtained dried material [Jałoszyński et al. 2013]. A positive effect of this type of drying on porosity, shrinkage and reconstitution properties of dried products is also observed [Nowacka et al. 2012]. Dividing the drying time into two stages: drying and stabilization, allows to reduce the destruction of the raw material tissue, reduce the probability of the material burning, improve the quality of the obtained dried material [Piotrowski and Figiel 2005] and increase the energy efficiency of the process [Ambros et al. 2018]. For example, Musielak and Kieca [2014] studied the effect of constant, periodic and variable (two-stage) microwave power on the microwave-vacuum drying and the quality of dried beetroot. The best results were achieved in the two-stage program (initially higher microwave power, then lower), which led to the greatest reduction in drying time, a slight change in color and no damage to the dried material.

The aim of the work was to analyze the properties of dried beetroot obtained by the microwave-vacuum method, with the use of various drying parameters (pressure, microwave power, time) and the convective method.

MATERIAL AND METHODS

Material

The subject of the research were beetroots (*Beta vulgaris* L.) "Wodan" variety, from the local market. Before the research works began, they were stored in a cold store without light, where the temperature was around 5°C and the air humidity was around 90%. In order to prepare the raw material for testing, the beetroots were peeled and cut into slices 7 mm thick, from which 20 mm wide strips were then cut out. Each time about 220 g of raw material was weighed for drying.

Testing

Microwave-vacuum drying was carried out with the use of a Promis-Tech Inc. dryer (Wrocław, Poland) and was performed in six variants. The three initial dryings were carried out with the use of microwaves with the power set at 200 W. The pressure was modified, which in the first variant was 35 hPa, in the second variant 45 hPa, and in the third 55 hPa. Drying with the use of 200 W microwave power was performed in 2 main cycles, each of which lasted 22 min and was divided into the following sequences: 200 W/480 s, 0 W/180 s, 200 W/480 s, 0 W/180 s. When, after 44 min of drying, the material was still moist and the weight loss was insufficient, subsequent cycles were performed with shorter durations (usually 200 W/180 s, 0 W/60 s). The next three drying cycles were carried out with the microwave power of 300 W, and the pressure values were 35, 45 and 55 hPa. In the case of the increased microwave power of 300 W, there was one main drying cycle lasting 22 min (300 W/480 s, 0 W/180 s, 300 W/480 s, 0 W/180 s). The use of higher microwave power significantly increased the risk of burning the material. Thereby, drying to the specified humidity was performed in shortened cycles, lasting 60 s.

Due to the time required to stabilize the dried material, they were stored for a week in aluminum pouches, which were heat-sealed in order to completely reduce the access of oxygen, light and moisture. Then, analytical researches were performed, which were additionally carried out on fresh beetroot and dried beetroot obtained by the convective method, in which the drying medium was air at a temperature of 70°C, flowing parallel to the material layer at a speed of 2 $m \cdot s^{-1}$. The load on the sieve was 10.1 $kg \cdot m^{-2}$.

The dry matter content was determined using the dryer method. Comminuted dried beetroot and fresh material were dried in a convective dryer (VWR DRY-line DL 53, Leuven, Belgium) with natural air circulation until constant weight was obtained. The temperature of the drying air was 105°C, and the whole process lasted 4 h. The measurement was performed in duplicate.

The density determination consisted of determining the volume of the sample placed in a measuring cylinder with the use of hexane in the burette. On this basis, the ratio of the sample mass to its volume was calculated. The measurement was performed in triplicate.

The analysis of hygroscopic properties of the studied dried materials was performed using the method proposed by Nowacka and Witrowa-Rajchert [2010]. Samples of known mass were placed in a desiccator, at room temperature (about 25°C), over a saturated NaCl solution with a water activity of 0.75. After 1, 2, 3, 6, 9, 24, 48 and 72 h, the tested material was reweighed. The measurements were performed in triplicate.

To determine the color of the dried and fresh material a Konica Minolta CR-5 (Osaka, Japan) trichromatic colorimeter was used. The analysis was performed by the reflection method, using the CIE L*a*b* system (standard observer 2°, light source D65). The material was placed directly in the device to completely cover the hole (diameter 8 mm). The test was performed in ten replications. Based on the measured L*, a* and b* values, total color difference (ΔE – compared to the color of fresh beetroot tissue), color saturation (C) and hue (H) as well as the browning index (BI) were calculated using the following formulas:

$$\Delta E = \sqrt{\left(\Delta L^*\right)^2 + \left(\Delta a^*\right)^2 + \left(\Delta b^*\right)^2}; \quad C = \sqrt{\left(a^*\right)^2 + \left(b^*\right)^2}; \quad H = \arctan\frac{b^*}{a^*}$$

$$BI = \frac{\left[100\left(x - 0.31\right)\right]}{0.17}, \quad \text{where: } x = \frac{\left(a^* + 1.75L^*\right)}{\left(5.645L^* + a^* - 0.012b^*\right)}$$

The content of betalains in the dried and fresh material was analyzed using the chemical method according to Nilsson [1970]. The absorbance was measured in a spectrophotometer (SPECTRONIC 200E, Thermo Fischer Scientific, U.S.) at wavelengths: 476, 538 and 600 nm. The standard was the previously prepared phosphate buffer solution. The measurement was performed in duplicate.

The total polyphenols content in dried and fresh material was tested using the Folin-Ciocalteu method [Singleton et al. 1999], and the gallic acid was the reference. The absorbance of the extracts was measured in a spectrophotometer (SPECTRONIC 200E, Thermo Fischer Scientific, U.S.) at a wavelength of 750 nm. The measurement was performed twice.

Statistical methods

All results were subjected to the one-way analysis of variance in the Statistica 13.1 (StatSoft Inc., U.S.) statistical program. Using the Tukey test, the obtained results were grouped into homogeneous groups (at the significance level of $\alpha = 0.05$). Additionally, the results of the analyzes of dried beetroot obtained by the microwave-vacuum method were subjected to a two-way analysis of variance.

RESULTS AND DISCUSSION

The analyzed samples were marked as: B1 (200 W/35 hPa), B2 (200 W/45 hPa), B3 (200 W/55 hPa), B4 (300 W/35 hPa), B5 (300 W/45 hPa), B6 (300 W/55 hPa), B7 (dried by convective method) and B8 (fresh beetroot). The results of the dry matter content, density, color, content of betalains and polyphenols are presented in Table 1.

The dry matter content in all dried materials varied between 87 and 93%. The use of microwaves with a power set at 200 W led to obtaining dried materials with a higher dry matter content. At the same time, for this level of microwave radiation power, an increase in the dry matter content in the samples with increasing pressure in the dryer chamber was observed. However, the two-way analysis of variance did not show a statistically significant effect ($p > 0.05$) of any of the analyzed microwave-vacuum drying process parameters (pressure, microwave power) on the dry matter content.

The drying method significantly influenced the density of the dried materials. In the case of products obtained by the microwave-vacuum method, all results fluctuated in the range of 0.4–0.5 $g \cdot cm^{-3}$ and statistically did not differ, but their values were significantly lower than the density of the material obtained by convective method (0.688 $g \cdot cm^{-3}$) and raw material (0.898 $g \cdot cm^{-3}$). Despite the lack of statistically significant differences, it was observed that with the increase of dry matter content in the dried materials, their density decreased, which means that the porosity of the materials increased.

Dried beetroots obtained by the microwave-vacuum method were darker than the product obtained by convection and at the same time, in most cases, similar in brightness to the raw material. The value of the L* color parameter of the microwave-vacuum dehydrated materials was significantly influenced by the drying parameters. The two-way analysis of variance showed that the brightness of the microwave-vacuum dried materials did not depend on the pressure in the drying chamber and the microwave power, but it increased with increasing the microwave power level. Darkening of the color of the dried material, in reference to the raw material, was also observed by Zhao et al. [2017] during microwave-vacuum drying of lotus seeds. On the other hand, color brightening was observed in microwave-vacuum-dried tomatoes [Orikasa et al. 2018] and green coffee beans [Dong et al. 2018].

The greatest color deviation in reference to the standard – fresh material, occurred in the material dried by convective method ($\Delta E = 10.78$). Among the products of microwave-vacuum dehydration, the lowest value of total color difference was achieved by the material, the drying of which was carried out using the microwave power set at 300 W and the pressure equal to 55 hPa ($\Delta E = 1.78$). At both levels of microwave radiation, carrying out the drying process under pressure reduced to 45 hPa led to the greatest changes in the color of the dried materials. A greater color deviation of the convective dried material, compared to the products of microwave-vacuum dehydration, was also noted by Orikasa et al. [2018] when removing water from tomatoes and Song et al. [2017] during the dehydration of a pumpkin. In another research, Musielak and Kieca [2014] studied the effect of constant, periodic and variable (two-stage) microwave power on the color of the dried beetroot. In the case of the program with constant microwave power, the

Table 1. Selected properties of dried and fresh material

Tabela 1. Wybrane właściwości materiału suszonego oraz świeżego

Sample Próbka	Dry matter content Zawartość suchej masy [g·g^{-1}]	Density Gęstość [g·cm^{-3}]	L*	ΔE	C	H	BI	Betalains content Zawartość betalain [mg·100 g^{-1} d.m.]		Total polyphenols content Całkowita zawartość polifenoli [mg GA·100 g^{-1} d.m.]
								betanin betanina	vulgaxanthin wulgaksantyna	
B1	0.8975 ±0.0023abc	0.497 ±0.022a	22.89 ±2.19a	3.16	21.72 ±4.15a	15.85 ±3.16ab	56.60 ±9.51b	212.35 ±1.70b	268.08 ±7.89cd	1 332.60 ±125.51a
B2	0.9105 ±0.0101cd	0.453 ±0.007a	21.81 ±3.47a	4.95	19.89 ±5.71a	16.00 ±1.24ab	53.83 ±10.97b	333.55 ±4.18d	367.98 ±4.22f	2 153.68 ±213.87b
B3	0.9306 ±0.0054d	0.397 ± 0.052a	25.45 ±2.57ab	3.72	22.74 ±4.34a	16.80 ±1.51b	53.25 ±6.44b	210.35 ±1.44b	328.74 ±1.62e	1 760.78 ±144.80ab
B4	0.8852 ±0.0009ab	0.473 ± 0.005a	27.28 ±3.73b	5.02	25.64 ±4.90ab	15.94 ±0.94ab	56.12 ±8.10b	247.08 ±11.27c	295.82 ±13.31de	1 854.26 ±126.31ab
B5	0.9162 ±0.0075cd	0.425 ± 0.001a	27.37 ±2.76b	9.08	31.30 ±5.44b	24.72 ±3.26c	62.82 ±5.50b	178.52 ±2.77a	282.68 ±3.78d	1 844.29 ±139.70ab
B6	0.8738 ±0.0002a	0.500 ± 0.086a	23.82 ±2.03ab	1.78	24.03 ±7.25a	15.32 ±2.42ab	58.80 ±12.88b	272.20 ±13.33c	244.42 ±14.29bc	1 208.96 ±108.19a
B7	0.9096 ±0.0085bcd	0.688 ± 0.090b	32.34 ±3.55c	10.78	21.10 ±3.89a	12.94 ±2.86a	40.93 ±4.00a	271.63 ±8.67c	222.31 ±13.56ab	1 074.39 ±437.56a
B8	0.1058 ±0.0006	0.898 ± 0.037	22.35 ±1.09a	–	24.79 ±3.77ab	16.96 ±0.85b	64.02 ±5.67b	310.99 ±1.40d	199.86 ±0.72a	1 263.01 ±140.56a

a, b, ... – the same letters in the columns represent homogeneous groups in statistical terms ($\alpha = 0.05$).

a, b, … – te same litery w kolumnach oznaczają grupy homogeniczne w ujęciu statystycznym ($\alpha = 0{,}05$).

smallest color difference was noted at the lowest microwave power (9.40). In the program with periodic change of microwave power the values of ΔE parameter ranged from 4.83 to 22.01. In the case of the two-stage program, the smallest difference in the color of the dried beetroot was achieved when initially higher microwave power was used, which was then lowered in the final stage. Among atmospheric, vacuum and microwave-vacuum drying, dried beetroot obtained by the last method showed the greatest color difference (5.06–13.39) [Székely et al. 2019].

The saturation of the color (C) of all the dried beetroots did not differ significantly from the value of this parameter recorded for the raw material. The method of drying did not significantly affect the color saturation, but a greater saturation of the dried materials obtained with the use of higher microwave power was observed. However, the two-way analysis of variance did not show a statistically significant influence of the analyzed variable parameters of drying.

The product of microwave drying with a power of 300 W, at a pressure of 45 hPa, was distinguished by the highest value of the color hue, analogically as in the case of saturation. Increasing the pressure in the drying chamber at the microwave power of 200 W resulted in a statistically insignificant increase in the value of the color hue of the microwave-vacuum drying products. Also, the two-way analysis of variance showed that both the microwave power and the pressure value did not statistically significantly affect the hue of the color of the dried material.

Convective drying led to the obtaining of dried material with a statistically significantly lower browning index compared not only to microwave-vacuum dried materials, but also to the raw material. The performed two-way analysis of variance did not find statistically significant differences in the values of the browning index of microwave-vacuum-dried materials, regardless of the microwave radiation power level or the pressure in the dryer chamber. Despite the lack of statistically significant differences, in the case of microwave power of 200 W, the value of the browning index decreased with increasing pressure.

In most materials, drying resulted in a reduction of the betanin content. The content of this pigment in the dried material obtained by the microwave-vacuum method at the microwave power of 200 W and the pressure of 45 hPa was comparable to the amount of betanin in fresh beetroot. The two-way analysis of variance did not show any significant influence of the analyzed process parameters on the betanin content. Székely et al. [2019] noted a higher content of betacyanin and betaxanthin in dried beetroot obtained by the microwave-vacuum method than after using convective drying. Removal of water from the beetroot samples increased the content of yellow pigment in all dried materials. The largest amount of vulgaxanthin was observed in the dried material obtained by the method using microwaves with a power of 200 W, with the pressure reduced to 45 hPa. At the same time, this material had the highest betanin content. The product of convective drying was characterized by the lowest content of yellow pigment among all the dried materials. Increasing the pressure in the drying chamber with the microwave radiation power equal to 300 W, led to a reduction in the amount of yellow pigment in the dried material. The two-way analysis of variance did not show any significant influence of microwave power and pressure on the content of vulgaxanthin. The increase in the amount of yellow and red pigments in dried materials, compared to the raw material, could be caused, among others, by the non-selectivity of the method of their determination, which also results in the determination of other substances with antioxidant properties [Pękal 2014], as well as the increased efficiency of extraction of pigments caused by tissue damage during drying. An increase in the content of red and yellow pigments in beetroot tissue dried by convective method and with using infrared radiation in comparison to fresh tissue was also observed by Nowak et al. [2008].

The vast majority of microwave-vacuum drying products was characterized by a greater amount of polyphenols expressed as gallic acid equivalents than the raw material. The highest content of these compounds was noted in dried material obtained under pressure reduced to 45 hPa, with the use of 200 W microwaves. Similarly, the content of red and yellow pigments in this dried material was the highest. Probably, the above drying parameters contributed to the increase in the extractability of bioactive compounds contained in the beetroot tissue. The lowest content of polyphenols was determined in the dried material obtained by the convective method. Among atmospheric, vacuum and microwave-vacuum drying, the last one, carried out with the microwave waves in a vacuum, resulted in dried beetroot with the highest content of polyphenols [Székely et al. 2019]. Increasing the pressure in the drying chamber at the microwave radiation power of 300 W led to a reduction in the retention of polyphenols in the dried material, but it was not statistically significant. The two-way analysis of variance showed that both the microwave power and the pressure did not statistically significantly determine the polyphenols content in the dried materials. The phenomenon of obtaining dried beetroots with a higher

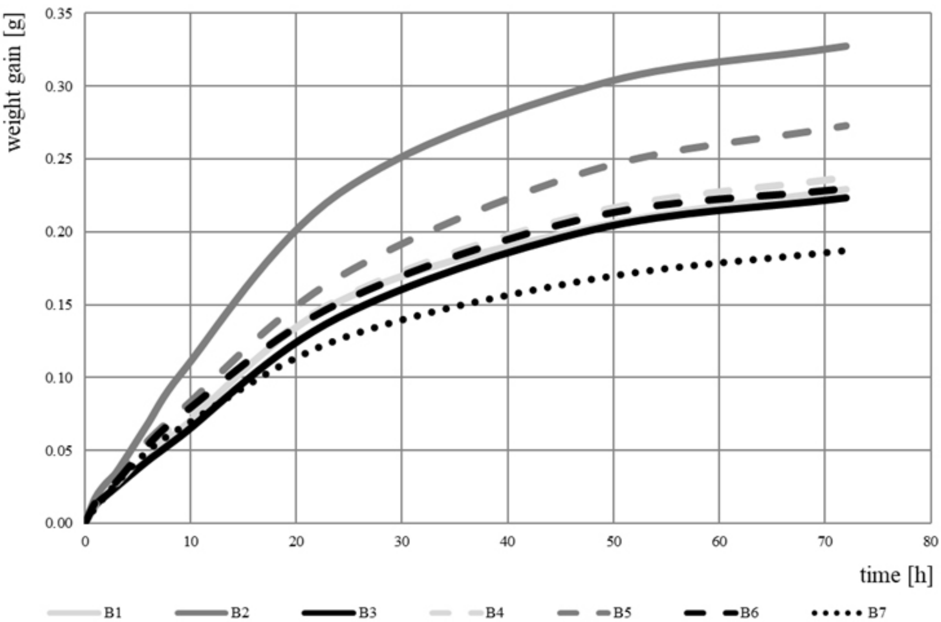

Fig. Weight gain of dried material depending on time

Rys. Przyrost masy materiału suszonego w zależności od czasu content of polyphenols compared to the raw material could result, among others, from the determination of not only polyphenols, but also other antioxidant substances reacting with the Folin-Ciocalteu reagent [Pękal 2014].

The products of microwave-vacuum drying absorbed moisture more intensively than the dried material obtained by the convective method (the figure, p. 38). In the initial stage of the determination, the weight gain of the samples was more intense, and the phenomenon was gradually slowing down over time. In Table 2 there was presented the weight gain of the samples after 24 and 72 h. Both after 24 and 72 h of measurement, the highest weight gain was recorded for the product of microwave drying with a power of 200 W, at a pressure of 45 hPa, and these values were statistically significantly different from those obtained for majority of the other dried materials.

It was observed that the lowest hygroscopicity was characteristic for the dried beetroots with the highest density (Table 1), that is with the lowest porosity at the same time. The two-way analysis of variance did not show any significant influence of any of the analyzed process parameters on the hygroscopicity of dried materials.

Table 2. Weight gain of samples after 24 and 72 h

Tabela 2. Przyrost masy próbek po 24 i 72 h

	B1	B2	B3	B4	B5	B6	B7
Weight gain after 24 h Przyrost masy po 24 h	0.1520 ±0.0288a	0.2263 ±0.0475b	0.1412 ±0.0135a	0.1533 ±0.0232a	0.1688 ±0.0112ab	0.1509 ±0.0095a	0.1261 ±0.0080a
Weight gain after 72 h Przyrost masy po 72 h	0.2290 ±0.0176ab	0.3276 ±0.0548c	0.2232 ±0.0192ab	0.2370 ±0.0386ab	0.2726 ±0.0221bc	0.2295 ±0.0094ab	0.1870 ±0.0084a

a, b, c – the same letters in the row represent homogeneous groups in statistical terms (α = 0.05).

a, b, c – te same litery w wierszu oznaczają grupy homogeniczne w ujęciu statystycznym (α = 0,05).

CONCLUSIONS

Microwave-vacuum drying resulted in obtaining materials of lower density than the dried material obtained by the convective method. Moreover, it was noticed that the higher the dry matter content in the obtained dried beetroots, the lower was their density, which also means the greater the porosity at the same time. The products of drying under reduced pressure using microwave radiation were darker than the material dried with hot air. At the same time, compared to the convective method, microwave-vacuum drying led to a smaller color change of the obtained products compared to fresh beetroot. When analyzing the nutrient content, microwave-vacuum drying led to greater reduction of betanin and greater retention of vulgaxanthin in the dried materials than convective drying. Due to the highest content of betalains (both red and yellow pigments) and polyphenols, the most optimal microwave-vacuum drying process parameters were: microwave power of 200 W and pressure of 45 hPa. However, the material dried under these conditions was distinguished by the highest capacity of water vapor adsorption, which may suggest its inferior storage stability compared to other dried materials.

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ANALIZA SUSZENIA MIKROFALOWO-PRÓŻNIOWEGO BURAKA ĆWIKŁOWEGO

Streszczenie. Celem pracy była analiza właściwości suszy buraka ćwikłowego otrzymanych metodą mikrofalowo-próżniową, przy zastosowaniu różnych parametrów suszenia (ciśnienie, moc mikrofal, czas), oraz metodą konwekcyjną, przy zastosowaniu powietrza o temperaturze 70°C i szybkości przepływu 2 $m \cdot s^{-1}$. W badaniach wykorzystano buraki ćwikłowe odmiany „Wodan”. W suszach dokonano oznaczenia zawartości suchej substancji, gęstości, higroskopijności, barwy, zawartości barwników betalainowych oraz zawartości polifenoli. Na podstawie wybranych wskaźników jakości określono procesowe parametry gwarantujące uzyskanie suszu o najlepszych właściwościach. Uzyskany metodą mikrofalowo-próżniową suszony materiał charakteryzował się mniejszą gęstością, większą higroskopijnością, ciemniejszą i bardziej zbliżoną barwą do surowca, większą zawartością barwników betalainowych i polifenoli w porównaniu z suszem otrzymanym konwekcyjnie. Zastosowanie mikrofal o mocy 200 W oraz ciśnienia wynoszącego 45 hPa doprowadziło do uzyskania suszy o najkorzystniejszych właściwościach.

Słowa kluczowe: suszenie mikrofalowo-próżniowe, gęstość, higroskopijność, barwa, betalainy, polifenole