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# Colour, moisture adsorption, and antioxidant properties of oven-dried chokeberry powder obtained after ultrasound-assisted osmotic dehydration

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*Osmotic dehydration (OD) of chokeberry samples in erythritol (ERT) and xylitol (XYL) solutions enhanced by ultrasonication (US) has been examined in terms of moisture adsorption, colour and antioxidant properties. After air-forced drying, the powder subjected to OD in ERT solution exhibited higher equilibrium moisture contents (EMC) in 0.20–0.45 $a_w$ than those in XYL solution. On the other hand, EMC values increased with the prolongation of US time from 5 to 30 min in the case of XYL solution. CIEL*a*b colour system was used for the determination of colour changes. While L* (the colour coordinate represents lightness (L* ~ 100) or darkness (L* ~ 0) of the sample) values decreased with the prolongation of US time from 5 to 30 min for both osmotic agents, only XYL solution caused the increase of a* (the colour coordinate represents green (−a*) or red (+a*) colour of the sample) and b* (the colour coordinate represents blue (−b*) or yellow (+b*) colour of the sample) to their maximum values at 30 min of sonication. A sample of powder subjected to OD in ERT solution has shown a higher total phenolic, total anthocyanin and antioxidant capacity. We may conclude that OD of chokeberries coupled with 30 min of sonication has resulted in chokeberry powder with the highest content of bioactive substances.*

**Keywords:** Aronia; Equilibrium moisture content; Sweetener; ANOVA

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## Introduction

Black chokeberry (*Aronia melanocarpa* L.) is a perennial shrub of the *Rosaceae* family. The fruits are edible possessing many health-promoting effects on cardiovascular diseases, hyperlipidemia, hypertension, and diabetes [1]. Choke-berry fruits are an excellent source of various bioactive compounds including anthocyanins, flavonols, flavanols, proanthocyanidins, and phenolic acids [2].

To prepare the powder with extension stability, a few drying techniques have been examined. It was found that antioxidant activity, phenolic and anthocyanin contents decreased with the increasing of drying temperature from 50 °C to 70 °C [3]. Freeze-drying or spray drying appeared to be excellent in the retention of bioactive substances when compared with convective drying [4,5]. Osmotic dehydration (OD) is a nonthermal process, which allows water removal from plant tissue, and conversely, an impregnation of the tissue with the solutes presented in the osmotic solution. OD is usually applied as a pretreatment step prior to further drying process. Mass exchange occurs during the OD process, which changes the chemical composition of the dehydrated food. The water content decreases, dry matter increases, accompanied by the leakage of low-molecular compounds [6]. The application of ultrasound technology in the OD of plant tissues may cause both, water loss and water gain, with respect to the ultrasound parameters (sonication time, amplitude and ultrasound power) and products [7]. Ultrasound-assisted OD pretreatment was applied to various plant tissues, where both loss and gain of bioactive compounds have been observed together with the change of moisture sorption behaviour and colour [8–18]. There is scarce data available on the effect of sonication during osmotic dehydration of chokeberry fruits. Bae et al. examined the effect of OD with two osmotic agents followed by different drying techniques on the quality of chokeberry dried powder [11]. In this study, the effect of OD with the combination of various sonication times on the moisture adsorption properties, colour, and some antioxidant properties of chokeberry powder was investigated. In addition, xylitol and erythritol were used as osmotic agents during the OD process.

## Materials and methods

### Sample preparation

Fresh chokeberry fruits (*Aronia melanocarpa* L.) were harvested in Krakow (Poland) at processing maturity. The fruits were sorted, washed, and inedible parts removed. Then, the chokeberry fruits were stored at 8 ºC before further processing.

## Ultrasonic pretreatment and osmotic dehydration

Osmotic dehydration was carried out in 30% (*w/w*) of xylitol (XYL) and erythritol (ERT) solutions according to the procedure of Nowacka et al. [19]. Solutions were prepared by dissolving the solutes in distilled water. Fruits (50 g) were placed in 250 mL beakers containing the osmotic solution. The weight ratio of osmotic medium to fruit sample was 4:1. The ultrasonic pretreatment was carried out at 40 ºC in an ultrasonic bath SONIC 14 (PolSonic, Warsaw, Poland) without mechanical agitation, using frequency 40 kHz and the total power of 400 W generated by sonotrodes, which corresponds to an intensity of 8 W/g. Samples were subjected to ultrasonic waves (US) for time periods of 5 to 45 min (US-5 to US-45). Afterwards, to continue the osmotic dehydration process for an additional 3 h, bakers with the tested samples were transferred to a rotary shaker at a speed of 120 rpm. After the treatment, the fruits were rinsed with distilled water for 10s and dried with absorbent paper. To evaluate the effect of ultrasound, the same procedure was carried out in the ultrasonic free environment (US-0). The treatment was conducted in three replicates for each osmotic solution.

## Preparation of dried powder

Each berry was cut in four parts and dried in a forced-air oven for 22–23 hours at 45 °C. Dried material was manually homogenised in a mortar to obtain fine powder. Desiccator with freshly dried silica gel had been used for the storage of chokeberry powder until analysis was performed.

## Moisture adsorption of chokeberry powder

Moisture adsorption in various relative humidities (0–80 %) was carried out in a device DVS Intrinsic Plus (Surface Measurement Systems Ltd., London, UK) monitoring the change in mass of the sample subjected to various levels of relative humidity (RH). Briefly, approximately 25 mg of dried sample was placed on an aluminium dish hanged on a sensitive analytical microbalance (mass resolution ±0.1 μg) in a closed chamber. Relative humidity of the surrounding space was controlled by the air stream (200 ccm) passing through the reservoir of re-distilled water at 10 % steps until the change in mass was lower than 0.002 mg/min within 10 min for each RH level. The results were expressed as equilibrium moisture content (EMC, mg/g of dry mass).

## Colour determination

Transmission spectrum of chokeberry powder samples was measured in the reflectance mode using a benchtop UltraScan VIS spectrophotometer (HunterLab, Reston, USA) with a d/8° geometry and standard illuminant D65. The spectra were measured in the range from 400 to 700 nm (with 10 nm reporting interval) and the colour expressed in a CIELab three-dimensional colour system, where $L^*$-axis represents a lightness (0-black, 100-white), $a^*$-axis is extended from green ($-a^*$) to red ($+a^*$), and $b^*$-axis from blue ($-b^*$) to yellow ($+b^*$). White tile was used as a standard for colour measurement. Each value was measured in five replicates.

## Determination of antioxidant properties

### *Extraction procedure*

Chokeberry powder (0.5 g) was placed in a glass tube with 10 mL of 90% methanol solution and 30 μL of formic acid followed by extraction for 30 min in an ultrasonic bath [20]. Supernatant was removed from the pellet after centrifugation at 3000 rpm for 5 min and stored at −18 °C prior to analysis. Each extract was prepared in duplicate.

### *Spectrophotometric assays*

The procedures for the determination of total phenolic content (TPC), total flavonoid content (TFC), and antioxidant capacity (TEAC) were adopted from our previous study [21]. Briefly, TPC was determined by the reaction of phenolics with Folin-Ciocalteau reagent. The product of the reaction was monitored at 765 nm and the results expressed as gallic acid equivalent in dry mass (mg GAE/g d.m.). Aluminium chloride assay was used for the determination of the total flavonoid content (TFC) where increase in absorbance at 425 nm was proportional to the increase of flavonoid content. The results were expressed as the quercetin equivalent (mg QRT/g d.m.). DPPH (2,2-diphenyl-1-picrylhydrazyl) assay was applied to the determination of antioxidant capacity, where the discoloration of DPPH· methanol solution in the presence of compounds with antioxidant activity was monitored at 517 nm. Results were expressed as Trolox equivalent antioxidant capacity (TEAC) in mg/g d.m. Total anthocyanin content (TAC) was determined by the pH-differ method [22]. Briefly, the extract was mixed with two buffer solutions (1.0 and 4.5 pH) and the absorbance was observed at two wavelengths (510 and 700 nm). TAC values were calculated using the molar absorption coefficient of cyanidin-3-glucoside (C3G) and expressed as mg C3G/g d.m.

Statistical analysis

The normality of all variables was evaluated using Shapiro-Wilks test. The effect of osmotic agent (Factor A) and time of ultrasonic pretreatment (Factor B) was determined using one-way analysis of variance (ANOVA) for variables with the normal distribution. In other words, the null hypothesis assumes that the means are equal. For multiply comparison between the means, post hoc Duncan´s test was applied. The results were expressed as an arithmetic mean and standard deviation. If the normality of variables was not confirmed, nonparametric Kruskal-Wallis ANOVA was used to study the effect of osmotic agents and US pretreatment time. Median with its average absolute deviation (AD) was used for the estimation of the mean in case of non-normal distribution. All statistical analysis was performed with Statistica 12 (Tibco Software Inc., Palo Alto, CA, USA) at the probability level $p = 0.05$.

## Results and discussion

Moisture adsorption of chokeberry powder

Moisture adsorption isotherms of all chokeberry powder samples (Figure 1) followed the type 3 according to BET classification [23]. The respective shape of the isotherm curve is characteristic for products with high soluble sugar content, such as osmotically dehydrated cambuci slices [8]. The gradual increase of EMC with the increase of $a_w$ (absence of sigmoid shape) refers to the multilayer sorption of water molecules on the surface of solids. As seen, there were no differences among the equilibrium moisture content of chokeberry powder pretreated with erythritol in the whole $a_w$ range, suggesting that ultrasound treatment did not affect the moisture adsorption properties. On the other hand, the moisture adsorption of powder previously dehydrated in xylitol solution increased with the increase of ultrasound treatment time from 5 to 30 min, particularly above 0.60 $a_w$. For example, the gradual increase of EMC from 191.5 to 228.7 mg/g d.m. at 0.70 $a_w$ was observed with the prolongation of US time from 5 to 30 min. EMC was similar for chokeberry powder pretreated with OD-US for 45 min and those without US. In contrast to our findings, osmotic dehydration of quince in sucrose solution combined with sonication decreased the EMC in comparison with that of US untreated samples [9]. In the lower $a_w$ region (0.20–0.45), apparently lower EMC for the powder pretreated with xylitol was observed without respect to the ultrasound treatment in our study. This is in agreement with a study by Cichowska-Bogusz et al., where ultrasound pretreatment of dried apples caused the lower moisture adsorption at 0.75 $a_w$, most intense when apples had previously been dehydrated in xylitol solution [10]. In our study, EMC of powders pretreated with erythritol (without respect of sonication) was at the same level as that for XYL-US-30 above 0.60 $a_w$.

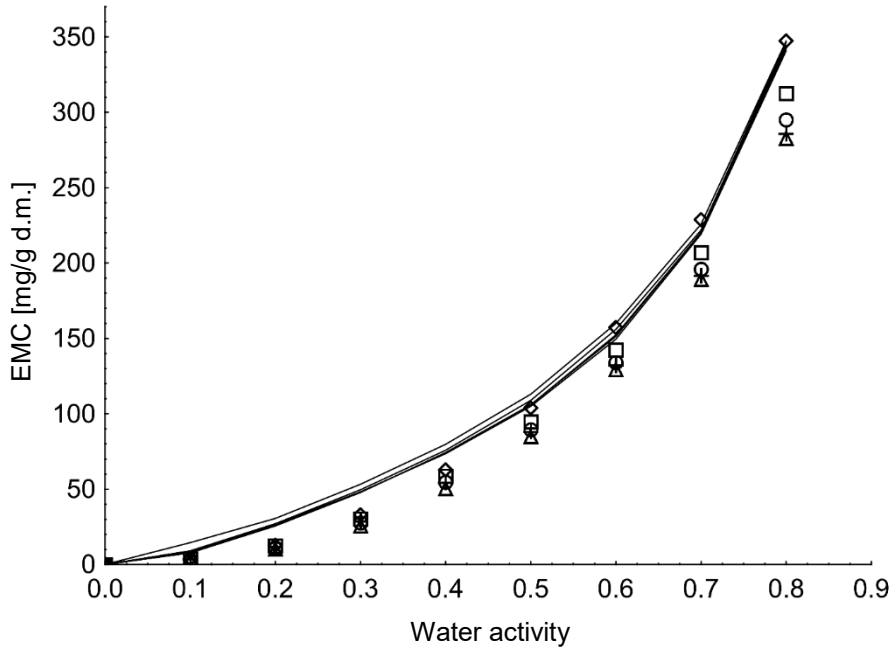


**Fig. 1** Equilibrium moisture content (EMC) vs. $a_w$ (at 25°C) of chokeberry powder pretreated by osmotic dehydration in erythritol (lines) and xylitol (symbols) with the assistance of ultrasound for 0 (+), 5 (○), 15 (□), 30 (◊), and 45 (Δ) min

## The change of colour

Chokeberry powder pretreated with xylitol, not subjected to US treatment, showed higher $L^*$ values, although not significant ($p$ = 0.072), and lower $a^*$ ($p$ = 0.001) and $b^*$ values ($p$ = 0.007), which represents a lighter, less red, and less yellow colour in comparison with those of erythritol. The effect of glucose, sucrose, and xylitol during osmo-dehydration pretreatment of chokeberry fruits followed by air-forced drying was studied by Bae et al. [11]. They had found that lightness did not depend on the osmotic solution, but lower both $a^*$ and $b^*$ values were observed when xylitol was used as the osmotic agent.

One-way ANOVA indicated a significant impact of the US pretreatment on all $L^*$ ($p$ = 0.001), $a^*$ ($p$ = 0.028), and $b^*$ ($p$ = 0.025) values; however, a strong interaction between osmotic agent and US treatment time was identified for all colour coordinates ($p$ < 0.01). As can be seen from Figure 2A, $L^*$ values have decreased with the increase of US treatment time from 5 to 30 min for both erythritol and xylitol osmotic agents. Figure 2A also showed that lighter powder was obtained after osmotic dehydration in xylitol solution. The study of the effect of various osmotic agents on the lightness of dried apple slices revealed that the parameter $L^*$ was significantly lower for US-treated samples, however, no differences among $L^*$ values were obtained for erythritol, xylitol and sucrose [10].

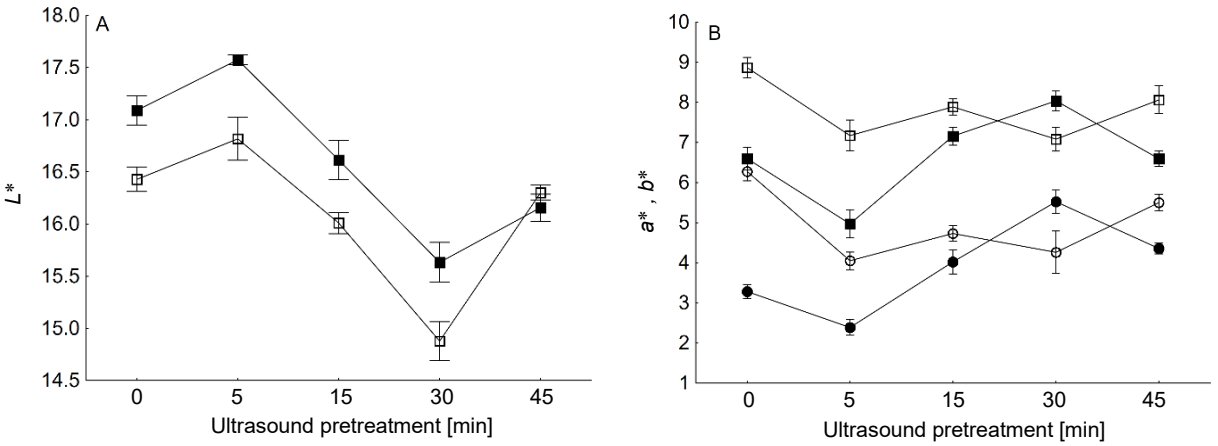


**Fig. 2** The effect of ultrasonic time on A) lightness/darkness (L*) and B) green/red (a*, squares) and blue/yellow (b*, circles) colour coordinates of chokeberry powder during osmotic dehydration pretreatment process in erythritol (open symbols) and xylitol solution (closed symbols)

Increase of lightness was observed for freeze-dried strawberries previously treated by osmotic dehydration enhanced by ultrasound [12]. However, different intensity of ultrasound was applied in their study. It was recently described that the change in colour of dried plums was affected by various ultrasound intensities (0.45–1.35 W/g) [13]. Although a strong interaction between osmotic agent and US treatment time was identified for *a** and *b** colour coordinates ($p < 0.001$), different trends have been observed for samples pretreated with xylitol and erythritol (Figure 2B). Significant decrease of *a** and *b** values was determined after 5 min of US pretreatment, followed by their gradual increase to the maximum values for US-30 in xylitol solution. On the other hand, maximal values of *a** and *b** were determined for the powdered form using erythritol as a dehydrating solution, without ultrasound pretreatment prior oven-drying at 45 °C. In that case, we may conclude that application of ultrasound have just caused the decrease of both *a** and *b** values regardless of time of sonication pretreatment. Various effects on the overall colour changes of kiwi fruit after 30 min of ultrasound-assisted osmotic dehydration in XYL and ERT solutions for subsequent convective drying have been observed in the study of Kroehnke et al [14]. The sample subjected to OD-US pretreatment with ERT solution showed higher colour changes in comparison with that of OD without applying US waves. Using XYL, ultrasound caused smaller colour changes in kiwi fruit powder. In our study, the overall changes in colour did not significantly differ between chokeberry samples pretreated with XYL and ERT solutions (data not shown).

## Antioxidant properties

Total phenolic, flavonoid and anthocyanin contents of chokeberry powder obtained by ultrasound-assisted osmotic dehydration with the subsequent air-forced drying at 45°C were in the range of 19.60–42.65 mg GAE/g d.m., 3.19–8.09 mg QRT/g d.m., and 0.20–0.57 mg C3G/g d.m., respectively. Those contents are in accordance with convectively dried chokeberry fruits at 50–70 °C [3], but being much lower than those obtained from chokeberry powder prepared by spray-drying or freeze-drying processes [4,5]. In the case of samples subjected to OD-US in erythritol and xylitol solutions, TFC values were similar as confirmed by Mann-Whitney test (Table 1). Therefore, we may say that chokeberry powder samples subjected to ultrasound-assisted osmotic dehydration in erythritol solution have exhibited significantly higher values for TPC ($p < 0.05$), TAC ($p < 0.05$) and TEAC ($p < 0.001$).

**Table 1** The effect of erythritol (ERT) and xylitol (XYL), and ultrasound pretreatment on the total phenolic content (TPC), total flavonoid content (TFC), total anthocyanin content (TAC), and antioxidant capacity (TEAC) of chokeberry powder

| | Effect of US pretreatment | | Effect of osmotic agent | |
|---|---|---|---|---|
| | K-W ANOVA | | M-W test | |
| | erythritol | xylitol | | |
| TPC | rejected* | rejected** | rejected* | ERT > XYL |
| TFC | rejected** | confirmed | confirmed | |
| TAC | rejected** | rejected** | rejected* | ERT > XYL |
| TEAC | confirmed | rejected* | rejected*** | ERT > XYL |

Null hypothesis means that all means are equal against; US, ultrasound; K-W, Kruskal-Wallis; M-W, Mann-Whitney; * $p < 0.05$; ** $p < 0.01$, and *** $p < 0.001$

Similar results were obtained from US-assisted osmotic dehydration of kiwi fruit, where a higher retention of polyphenolic substances was achieved by applying erythritol and sorbitol in contrast to that of sucrose solution [14]. On the other hand, chokeberry fruit powder pretreated with sucrose, glucose or xylitol solutions did not significantly differ in TPC, but it has shown lower proanthocyanidin content and antioxidant activity (evaluated using FRAP and ABTS assays) for the samples pretreated with xylitol [11]. It should be noted that the retention of bioactive compounds depends upon both osmotic solution and ultrasound time as was concluded for dried plums. While application of US for 30 min gave higher phenolic content for samples pretreated with glucose solution, the extension of US time resulted in the opposite effect; i.e., higher phenolics in samples pretreated with sucrose solution [15]. The effect of ultrasound applied

during OD on the antioxidant properties of chokeberry powder is illustrated in Table 1. The null hypothesis that all the means are equal was not confirmed for TPC ($p < 0.05$), TFC ($p < 0.01$), and TAC ($p < 0.01$) when ERT had been used as osmotic substance during ultrasound-assisted osmotic dehydration. It means that those antioxidant properties were affected by the time of ultrasound pretreatment. Multiplied comparison has revealed that there is no significant difference between TPC values, but the highest total flavonoid content was found for samples pretreated with ultrasound for 30 min (7.38 ± 0.03 mg QRT/g d.m.) in comparison with samples not subjected to ultrasound (6.36 ± 0.17 mg QRT/g d.m.; $p < 0.05$) or ultrasonicated for 5 min (6.12 ± 0.07 mg QRT/g d.m.; $p < 0.01$).

Similarly, chokeberry powder subjected to OD-US-30 showed significantly higher total anthocyanin content with a median 0.52 ± 0.01 mg C3G/g d.m. ($p < 0.05$) when compared to TAC value 0.38 ± 0.02 mg C3G/g d.m. after OD-US-5 process. Using xylitol as the osmotic agent, the effect of ultrasound pretreatment time was confirmed for TPC ($p < 0.01$), TAC ($p < 0.01$) and TEAC ($p < 0.05$) values. Similar trends were obtained for chokeberry powder samples pretreated by osmotic dehydration in xylitol solution showing the highest TPC (41.43 ± 3.36 mg GAE/g d.m.) and TAC (0.54 ± 0.01 mg C3G/g d.m.) values for ultrasound-assisted OD for 30 min. It was previously observed that ultrasonic treatment time during osmotic dehydration affected the release of phenolic substances from plant cells. For instance, sonication of cashew apple bagasse for 5 min increased the total phenolic content and antioxidant capacity in comparison with 2 min treatment [16]. Although our results are not consistent in all spectrophotometric assays, application of ultrasound for 30 min during OD of chokeberries seems to preserve more antocyanins when both xylitol and erythritol were used. Ultrasound may cause plasmolysis of cells; therefore, an enhanced release of phenolic compounds from plant cells can be observed in various products [16–18]. In addition, a formation of many microscopic channels by ultrasound during OD was responsible for the loss of structural integrity of pomegranate seeds [17]. We may hypothesize that US treatment for 45 min during OD of chokeberry caused a disruption of the cell walls, which facilitated the release of bioactive substances, exposing them for further oxidation. Similarly, a decrease of phenolic content and antioxidant capacity of Sanhua plum was observed with the increase of ultrasound power [13].

## Conclusion

The osmotic dehydration coupled with the ultrasonication process was found to be suitable for the preparation of chokeberries prior to oven-air drying. OD in erythritol solution has resulted in chokeberry powder with higher EMC, particularly at the low $a_w$ region. Various sonication times during OD were

reflected in different EMC for xylitol, but not for erythritol. Based on our results, OD in erythritol solution enhanced by sonication for 30 min can be recommended since the higher content of phenolic and anthocyanin substances has been observed. Finally, the powder samples exhibited also a lighter tone of the respective colour.

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