The influence of temperature on rehydration and sorption properties of freeze-dried strawberries

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Summary

The aim of this work was to investigate the influence of heating shelf temperature on selected physical properties of freeze-dried strawberries. Frozen strawberries were freeze-dried at heating shelf temperature of 10, 30, 50 and 70°C for 24 hours. Rehydration, adsorption rate and sorption isotherms were determined for freeze-dried strawberries. With increase the temperature of heating shelves in the range 10-50°C, rehydration capacity was increased. After 30 minutes of the rehydration process there was a decrease in water content, which is probably connected with changes in structure. Sorption isotherms were included to II isotherms type and have a sigmoidal shape characteristic for most food products. For the mathematical description Peleg's model was used. For freeze-dried strawberries obtained in the range from 10 to 50°C isotherms have the same courses. Increase of temperature to 70°C resulted in obtaining the lowest water content in the water activity range from 0.113 to 0.648. There is a statistically significant influence of temperature of heating shelf on water vapour sorption for freeze-dried strawberries. For fruit freeze-dried in 30°C there was the highest water vapour sorption rate.

Keywords: strawberries, freeze-drying, water sorption, kinetic, rehydration

Introduction

Strawberries are esteemed for their taste, flavour and nutritional value. Fresh fruit are not only tasty, but also contain high levels of vitamins A, B_1, B_2 and C as well as considerable amounts of chemical ingridients such as iron, phosphorous, magnesium and calcium. In Poland commercial varieties of these fruit are produced on a large scale. Their presence on the global market assures strong domination in cultivation of one variety – Senga Sengana. Because fresh fruit appear at the turn of May and June, it is very important to obtain suitable preservation technology.

In recent years different methods of drying have been analysed and much attention was focussed on the quality of food preserved by these methods (Jena and Das, 2005). The high temperature used in traditional drying caused a decrease in quality, which consists in large changes in structure and crispness losses (Maskan, 2001; Akanbi and Oludemi, 2004). Dried strawberries prepared by these methods are significantly different from the raw material. Continuous "improvement" of the convective drying process is targeted at obtaining high quality of dried strawberries (Alvarez et al., 1995) and berries (Lim et al., 1995), which leads to elaborate freeze-drying technology. Freeze-drying is a method in which water is removed from the material by sublimation. The process is long and done in a more expensive equipment than that used in atmospheric drying but the quality of the product is considered as the highest of any dehydration techniques (Lewicki, 2006). The porous microstructure of freeze-dried materials is characterized by high hygroscopicity, small resistance to mechanical damage and thin cellular walls, which easily undergo collapse after crossing the glass transition temperature. Well-chosen parameters of freeze-drying are very important not only because of the quality attributes of the final product, but also because of the high cost and time necessary to obtain the dry product. Knowledge of the characteristic phase change temperatures for nutritional components allows proper planning of the sublimation process and lately freeze-dried product storage, which ensures high quality (Shishehgarha et al., 2002, Chakraborty et al., 2006). It is expected that the high porosity developed in freeze-dried products plays a significant role in both rehydration capacity and rate. Pre-drying treatments as freezing and rehydration itself can induce structural and compositional changes in the food tissue which affect freeze-dried product quality (Giraldo et al., 2006).

Physical, chemical, biochemical and microbiological changes which determine nutritional stability are closely connected with the status of water in food products. A great deal of valuable information about these sorption isotherms is available regarding the

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dependence between water activity and water content in the product (Gondek and Lewicki, 2005).
The aim of this work was to investigate the influence of heating shelves temperatures on chosen physical properties of freeze-dried strawberries.

Materials and methods

Frozen strawberries of the variety Senga Sengana – without leaf stalk, calibrated (25-30 mm) – were investigated. Fruit were stored in plastic pouches of 500g each, in the freezer at temperature -18°C. Time of storage was 6 months.
Frozen strawberries were dried in an ALPHA1-4 LDC-1m freeze-dryer, Christ company (Osterode am Harz, Germany), with contact heating of material with parameters: pressure 63 Pa, safety pressure 103 Pa, time 24 hours, temperature of heating shelf 10, 30, 50 and 70ºC. Control of fruit temperature during drying was done with the use of thermoelements. After freeze-drying, fruit were enclosed in jars and were stored in a dark place at a temperature of 25±3ºC until the time of investigation.
Rehydration properties were estimated on the basis of freeze-dried fruit mass increase during a specified time of being kept in water (Witrowa-Rajchert, 1999). The measurement was done at room temperature, for every type of freeze-dried strawberries, in four repetitions for four different times. 100 ml of distilled water were poured into four beakers and one whole strawberry (about 1 g) previously weighed on a technical scale with accuracy to ±0.001 g was put in. After times of 5, 30, 60 and 120 minutes, fruit were drained, weighed and dry matter content and water content were determined (Drzazga, 1995).
To determine the isotherms of water vapour sorption (Gondek and Lewicki, 2005), weighed strawberries (about 1 g) as whole fruit were put in 7 chambers with water activity (a_w) from 0.113 to 0.903 for 3 months. After that time they were weighed again and water activity was determined.
The measurement of water vapour sorption kinetics (Domian et al., 1996) was done in four repetitions for every type of strawberry using a stand which could ensure permanent measurement of mass increase in conditions of constant temperature and relative humidity. Saturated $NaNO_2$ solution was used to obtain constant water activity of environment (0.648). The measurement was done at a temperature of 25±1°C for 20 hours. The sample for investigations was whole dried strawberries. The mass increase was registered by means of the computer program “Measurement for DOS”.

An exponential equation (Kowalska et al., 2006) was used for mathematical interpretation of the obtained results:

$$u = a + b * \left(1 - \exp^{(-c*\tau)}\right) \quad (1)$$

where:
u-water content [gH_2O/g d.m.]
a, b, c-constant of equation
t-time [h]

For interpretation of rehydration capacity and water vapour sorption data in water content (u) in different times (t), correlation coefficient R^2; mean relative error MRE (Jamali et al., 2006); error of water content estimation SEE (Jamali et al., 2006); residual squares sum RSS (Pagano and Mascheroni, 2005); and root mean square RMS (Lewicki, 2000) were determined on the basis of equations:

$$u = \frac{(1-s)}{s} \quad (2)$$

$$MRE = \frac{100}{n} * \sum \left| \frac{u_e - u_o}{u_e} \right| \quad (3)$$

$$SEE = \sqrt{\sum (u_e - u_o)^2} \quad (4)$$

$$RSS = \sum (u_e - u_o)^2 \quad (5)$$

$$RMS = \sqrt{\frac{\sum \left(\frac{u_e - u_o}{u_o} \right)}{n}}^{2} * 100\% \quad (6)$$

where:
s-dry matter content in samples–fraction, g d.m./g
n-number of data
e-experimental
o-calculated

Statgraphics Plus, version 3.0 (Microsoft), Excel 2000 (Microsoft), Table Curve 2D v. 3 (Jadel), and Statistica 5.0 (StatSoft) were used for statistical analysis. Standard deviations (SD) were determined for obtained mean results. Fisher’s test for verification of the hypothesis of equality of means for analysed coefficients in measured samples and Pearson’s correlation coefficient were used. The least significant difference (LSD) between mean values was calculated for analysed technological coefficients in investigated pairs in dependence on the applied variable using Fisher's test (multiple range test). The analyses were done with the significance level of 0.05.

Results and discussion

Rehydration properties of freeze-dried strawberries

There was found to be a statistically significant influence of temperature of heating shelves on rehydration capacity of freeze-dried strawberries between I (10°C) and III (50°C), II (30°C) and IV (70°C), as well as III (50°C) and IV (70°C) (Fig. 1). After 30 minutes of rehydration, water content of freeze-dried strawberries decreased except in the case of dried fruit which were obtained at heating shelf temperature of 10°C (I), for which water content slightly increased. Probably after 30 minutes there was intensive diffusion of soluble solids outside, much more than the quantity of water that penetrated into the tissue. This phenomenon has been described by Witrowa-Rajchert (1999). Tzee Lee et al. (2006) also confirmed dependence of rehydration capacity on plant material structure. They found that freeze-dried potatoes and avocado during 3 minutes of rehydration showed a high degree of water adsorption and in the case of bananas five times less, in spite of the same conditions of freeze-drying.

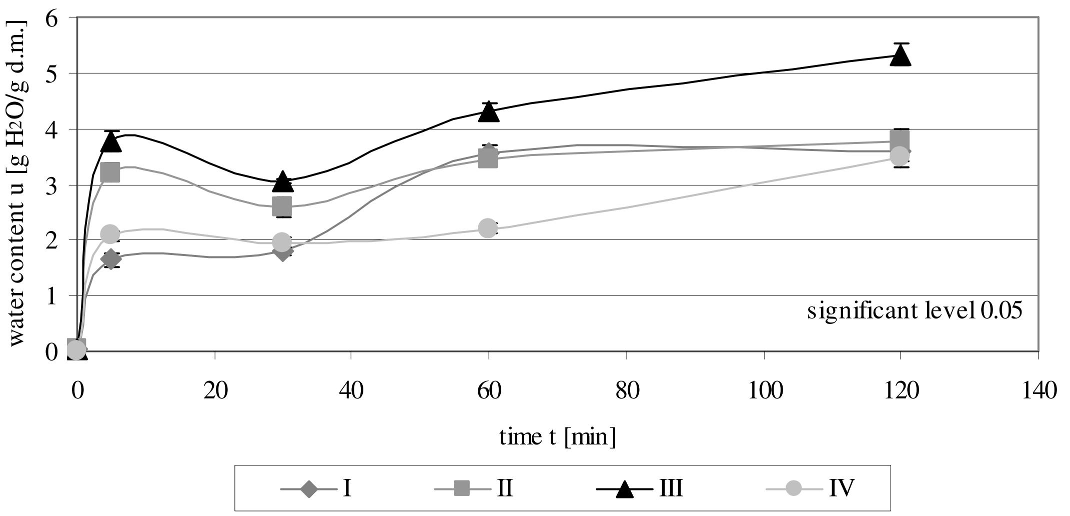

Fig. 1. The influence of of heating shelf temperature on water content as a function of rehydration time, for freeze-dried strawberries without osmotic dehydration. Temperature: I-10°C, II-30°C, III-50°C, IV-70°C

It was found that with increase of heating shelf temperature in the range 10-50°C, rehydration capacity was increased. However, for strawberries freeze-dried at 70°C it was decreased, as a result of unfavourable changes in structure. Also Marques et al. (2009) showned that water uptake capacity for freeze-dried tropical fruits was affected not only by injuries during moisture removal but also by structure changes induced by the rehydration process.

Also Gawałek (2005) found that generally freeze-dried materials have very good rehydration capacity, but an increase in the temperature of heating shelves in the range 40 - 90°C caused a decrease in quality and water imbibition capacity. Lis and Lisowa (1999), who investigated the rehydration capacity of apples found that rehydration capacity decreased with increase of temperature.

Freeze-dried strawberries obtained at heating shelf temperature of 50°C had the highest water content after immersing in water for 5, 30, 60 and 120 minutes. While the smallest water imbibition capacity was for strawberries dried at a temperature of 70°C (Fig. 1). This may be connected with changes in the structure of freeze-dried strawberries.

When the temperature of sublimation is too high (60°C) the temperature of the raw material untimely increase not only on the surface, but also inside (Lis and Lisowa, 1999). For fruit obtained at heating shelf temperature of 70°C the glass transition temperature was exceeded considerably, which influenced reconstitution capacity. Strawberries dried at heating shelf temperature of 50°C showed lower, but similar properties to freeze-dried material obtained at a temperature of 30°C. The glass transition temperature

probably was not exceeded and they imbibed water more than those obtained at a temperature of 70°C.
Shishegharha et al. (2002) confirmed that temperature of heating shelves above 50°C caused dry material to reach a temperature above the glass transition temperature, which increases probability of collapse. They defined that for freeze-dried strawberries the glass transition temperature was about 38°C.

Sorption isotherms of freeze-dried strawberries

Sorption isotherms of freeze-dried strawberries have a sigmoidal shape characteristic for most food products (Fig. 2). On this basis sorption isotherms can be included in type II isotherms (Brunauer et al., 1940). It reflects the mechanism of water binding and properties of the material. Also, Palou et al. (1997) made the same classification for vacuum-dried cookies and crisps, while Swami et al. (2005) did the same for convective dried nuggets. Also, Moraga et al. (2004) for freeze-dried strawberries obtained a sorption isotherm course typical for products with high sugar content. This is connected with the slow changes in equilibrium water contents at low water activity and rapid increase above water activity 0.5. At that water activity level interactions between solvent (water) and soluble solid are connected with sugar dissolution.

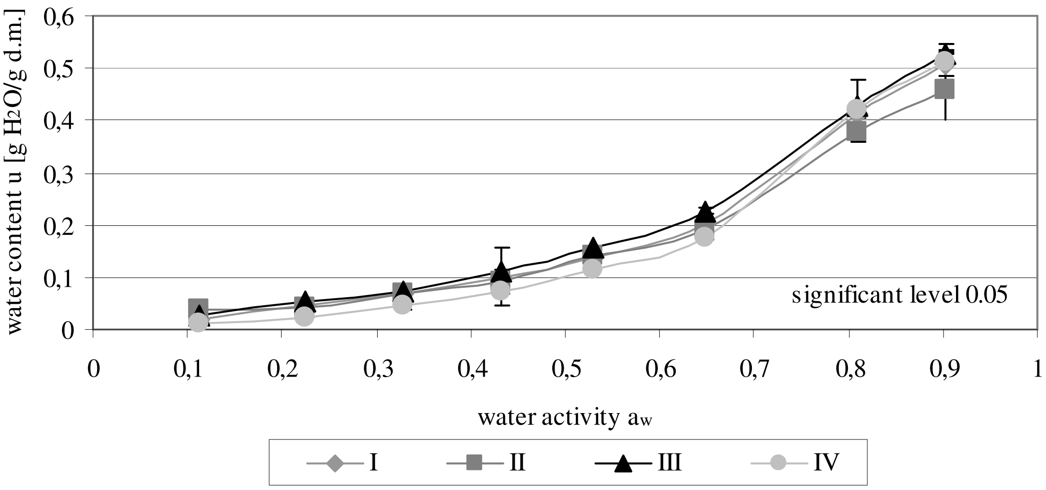

Fig. 2. The influence of temperature of heating shelf on water content as a function of water activity, for freeze-dried strawberries without osmotic dehydration. Temperature: I-10°C, II-30°C, III-50°C, IV-70°C

There were no statistically significant differences between strawberries which were obtained at heating shelf temperature of 10°C (I), 30°C (II) and 50°C (III) in sorption isotherms course at significance level of 0.05, while after the temperature of heating shelves increased to 70°C (IV) there were statistically significant differences (Fig. 2). Freeze-dried strawberries at 70°C have the smallest water absorption in water activity 0.113 - 0.648, while in higher water activities (0.648 - 0.903) they have water content similar to freeze-dried fruit which were obtained in the range of heating shelf temperature from 10 to 50°C (I, II, III).

A study to choose the best fitting mathematical model for the description of sorption isotherms was undertaken. 5 models were analysed: Oswin, Halsey (Akanbi et al., 2006), Iglesias-Chrife (Johnson and Brennan, 2000), Peleg (Gondek and Lewicki, 2005) and Lewicki (1998) (Table 1). A study was also undertaken to apply the GAB model (Lewicki, 1997), but the parameters of this equation did not fulfil the conditions determined by Lewicki (1997) to be permitted for experimental data description.
It was decided to choose Peleg's empirical equation (Gondek and Lewicki, 2005) on the basis of minimum MRE, RSS, SEE, RMS value and highest R^2 (Tables 1 and Fig. 3).

Table 1. Parameters of fitting water vapour sorption models for freeze-dried strawberries without osmotic dehydration. Temperature: I-10°C, II-30°C, III-50°C, IV-70°C

Model / Coefficient	Lewicki's	Peleg's	Iglesias-Chirife's	Oswin's	Halsey's
I [10°C]					
MRE%	34.26	8.24	68.97	26.87	45.55
RSS	0.015	0.001	0.022	0.008	0.012
SEE	0.123	0.035	0.147	0.922	0.109
RMS	61.60	11.14	122.28	39.57	60.68
R^2	0.990	0.994	0.904	0.963	0.947
II [30°C]					
MRE%	42.03	4.54	47.04	17.42	41.40
RSS	0.023	0.001	0.017	0.007	0.009
SEE	0.153	0.032	0.132	0.084	0.099
RMS	63.67	5.40	70.96	23.44	57.20
R^2	0.985	0.994	0.903	0.960	0.945
III [50°C]					
MRE%	35.5	8.18	58.15	25.13	49.52
RSS	0.13	0.018	0.019	0.008	0.070
SEE	0.017	0.136	0.140	0.091	0.266
RMS	65.44	11.39	98.71	34.57	59.18
R^2	0.992	0.997	0.918	0.965	0.949
IV [70°C]					
MRE%	36.01	11.35	126.16	49.35	51.57
RSS	0.009	0.023	0.030	0.014	0.008
SEE	0.094	0.152	0.174	0.116	0.092
RMS	67.32	14.24	238.04	68.67	65.91
R^2	0.990	0.990	0.880	0.946	0.967

Figure 3 shows exemplary graphic points calculated from Peleg's model adjusted to experimental water vapour sorption isotherm data for freeze-dried strawberries. Also Lewicki used models to describe (Lewicki, 1997) sorption isotherms equation: Peleg's for 27 products, GAB for 23, and Lewicki equation for 28. He showed that the highest probability of fitting experimental data and minimum mean residual error is given by Peleg's model. Palou et al. (1997) considered also that Peleg's model was the best to describe isotherms for cookies and chips, because the relative standard deviation was lower than 7%. According to Gondek and Lewicki (2005) Peleg's equation can be used to describe isotherms of type II and III.

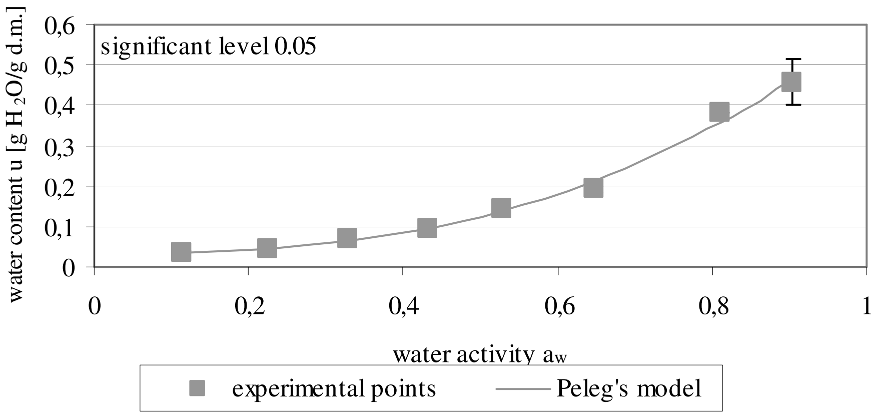

Fig. 3. Fitting of Peleg's model to describe water vapour sorption isotherms for freeze-dried strawberries without osmotic dehydration II [30°C]

Kowalska et al. (2005) affirmed that the characteristic sigmoidal shape of obtained isotherms is connected with occurrence of monomolecular sorption range in the atmosphere of low water activity < 0.3, multi-layer sorption for range 0.3 < a_w < 0.65 and capillary condensation at a_w > 0.65. This type of isotherm is often encountered for food products. The demonstrated differences in equilibrium water content may be a result of chemical composition, type of components and structure of investigated materials.

Water vapour sorption kinetics of freeze-dried strawberries

To optimize drying process and choose the best storage conditions it is important to know sorption properties (Moraga et al., 2004). Interactions between moisture in food products and surrounding air are important for drying, storage, packaging and transport. Suitable design of those operations has influence on physical properties, moisture, mass and volume changes (Raghavan and Silveira, 2001). Very often sorption properties are investigated with the use of sorption isotherms, but rare with the use as sorption kinetic.

The analysis of water content curves as a function of time showed that there is a statistically significant influence (significant level 0.05) of temperature of heating shelf on water vapour sorption for freeze-dried strawberries without osmotic dehydration (Fig. 4). Only between freeze-dried strawberries III (50°C) and IV (70°C) there was no statistically significant difference in curves' course. This may be connected with structural changes of freeze-dried strawberries. Despite having the highest water content, after 20 hours of sorption process in freeze-dried strawberries obtained at heating shelf temperature of 70°C, differences for freeze-dried strawberries are very small, lower than 0.01 g H_2O/g d.m.

Convective drying of strawberries caused 50% a decrease in water vapour sorption after 20 hours of the process in relation to freeze-dried fruit (Fig. 4). While Montes and Gerard (2004) during convective drying of bentonite showed that the drying temperature of sample does not play an important role in the water adsorption kinetics. However, when this temperature is very high, the water adsorption phenomena was severely affected, i.e. the maximal amount of adsorbed water decreases.

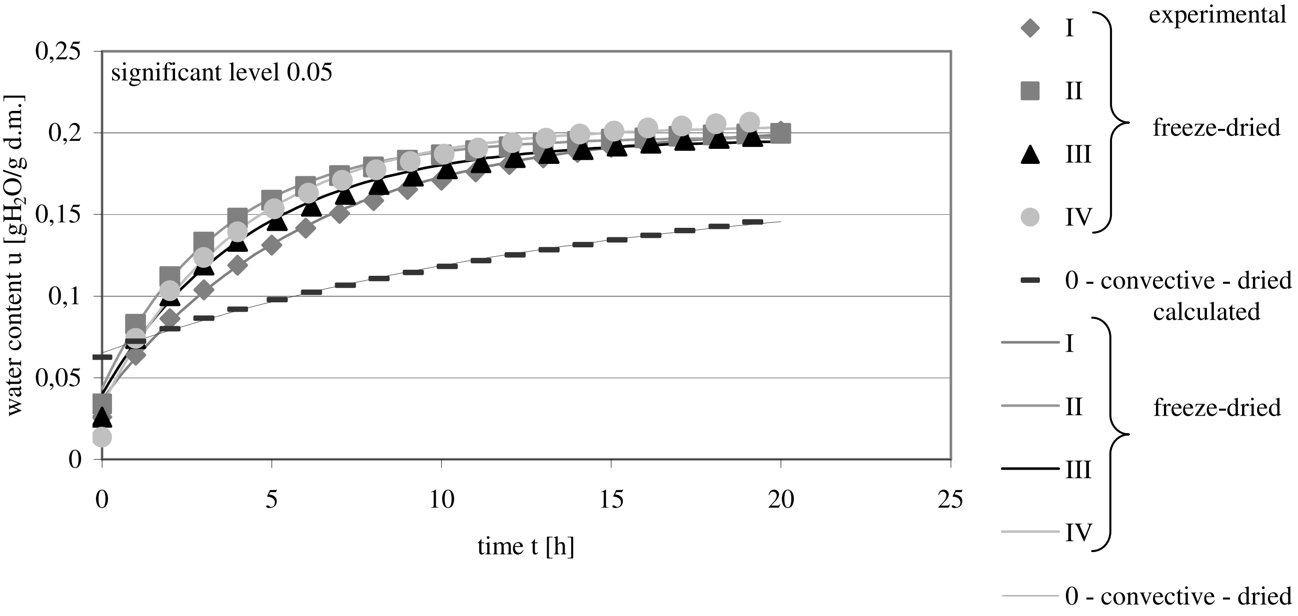

Fig. 4. The influence of temperature of heating shelf and drying method, on water content as a function of time, for freeze-dried and convective-dried strawberries without osmotic dehydration. Temperature: I-10°C, II-30°C, III-50°C, IV-70°C. Drying method: 0 - convective-drying [60°C]. a_w of environment - 0,648

Relations of changes in water content to sorption time for freeze-dried strawberries were mathematically analysed (Table 2). The high correlation coefficient (R^2) for freeze-dried strawberries, lower for most relations of mean relative error (MRE) fitting of experimental and calculated data concerning the initial water content and after 20 hours of water vapour sorption, was decisive for the choice of exponential equation (1) to describe sorption of water vapour curves in the investigated time range (Fig. 4).

Table 2. Parameters of fitting exponential equation $u = a + b * \left(1 - \exp^{(-c*\tau)}\right)$ to describe water vapour sorption kinetics for freeze-dried and convective-dried strawberries without osmotic dehydration. u_0 – initial water content, u_{20} – water content after 20 hours

Dried strawberries		Equation coefficients			R^2	MRE [%]	Experimental water content [g H_2O/g d.m.]		Calculated water content [g H_2O/g d.m.]	
		a	b	c			u_0	u_{20}	u_0	u_{20}
Freeze-dried	I	0.036	0.168	0.167	0.998	19.97	0.026	0.201	0.036	0.198
	II	0.043	0.154	0.283	0.998	13.74	0.034	0.199	0.043	0.197
	III	0.040	0.156	0.228	0.996	25.75	0.026	0.198	0.039	0.195
	IV	0.032	0.172	0.246	0.995	74.04	0.013	0.207	0.032	0.203
Convective-dried	0	0.065	0.108	0.068	0.998	3.09	0.062	0.147	0.065	0.145

For freeze-dried strawberries (Fig. 4) and also for convective drying, the correlation coefficient (R^2) of chosen exponential equation (1) is in the range 0.995 - 0.998. Mean residual error (MRE) agreement of experimental and calculated data concerning initial water content and after 20 hours of sorption process is lower for convective dried strawberries at 3.1%. In the case of freeze-dried strawberries at heating shelf temperature of 10°C (I), 30°C (II) and 50°C (III) the value of MRE is higher and is in the range 13.7 – 25.7%. The highest MRE at the level of 74% is that of freeze-dried strawberries obtained at heating shelf temperature of 70°C (IV). So considerable MRE value increase results from variances between experimental and calculated initial water content for freeze-dried fruit. Dried strawberries obtained similar experimental and calculated water contents after 20 hours of the absorption process; therefore it was decided to use an exponential equation to describe sorption kinetics despite the high MRE value for freeze-dried strawberries IV (70°C).

Analysing the course of water vapour sorption rate curves as a function of water content in common water content range 0.05 – 0.15 g H_2O/g d.m., it was found that there are statistically significant differences between freeze-dried strawberries dependently on temperature of heating shelf (Fig. 5).

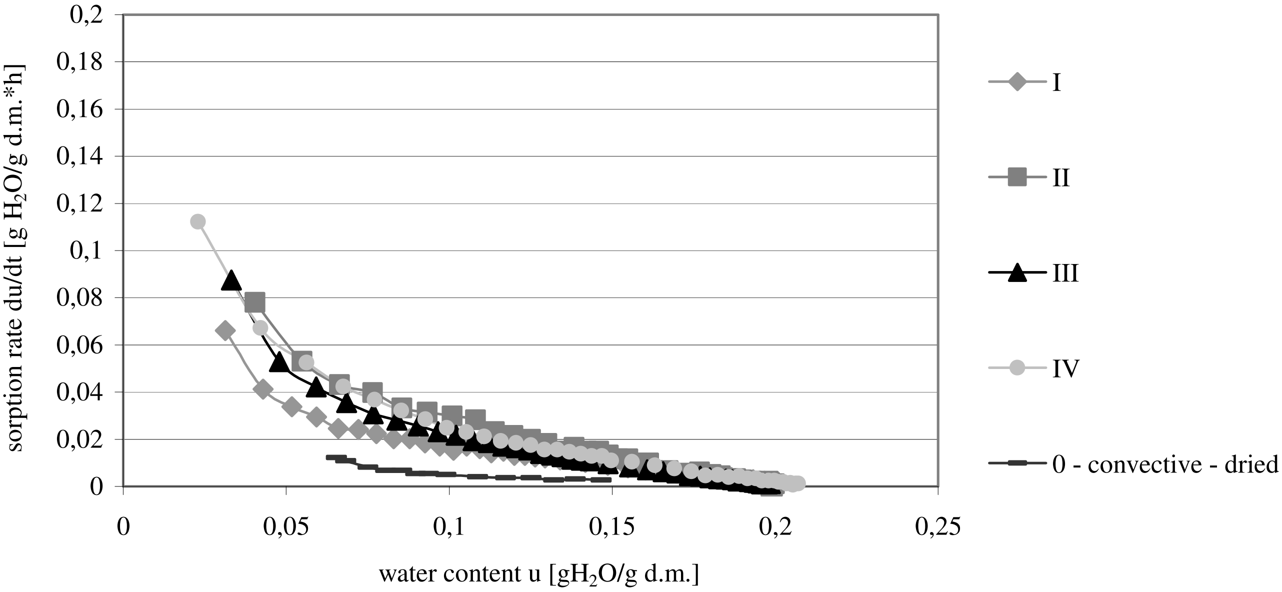

Fig. 5. The influence of temperature of heating shelves and drying method on rate of water vapour sorption as a function of water content, for freeze-dried and convective–dried strawberries without osmotic dehydration. Temperature: I-10°C, II-30°C, III-50°C, IV-70°C. Drying method: 0 - convective-drying [60°C]. a_w of environment - 0,648

Strawberries obtained at heating shelf temperature of 10°C (I) showed the lowest water vapour sorption rate from all freeze-dried strawberries. The curve of changing water vapour sorption rate for convective dried fruit in common with freeze-dried fruit in the water content range 0.05 – 0.15 g H_2O/g d.m. showed significant differences (Fig. 5). Convective drying caused a decrease in water vapour sorption rate for those fruit in relation to freeze-dried strawberries. Montes and Gerard (2004) during convective drying of bentonite shown that when this temperature is very high, the water adsorption phenomena was severely affected, i.e. the rate water adsorption decrease because the half-adsorption time was about two times more superior.

According to Domian et al. (2002) water vapour sorption capacity is characteristic for the product and is dependent on the occurring form and on the technological process, while freeze-dried material has the highest sorption rate as a function of water content.

There was found a significant influence of drying method on rate of water vapour sorption for dried strawberries (Fig. 5). Convective drying caused a decrease of water vapour sorption rate in relation to freeze-dried strawberries.

Conclusions

Application of heating shelf temperature of 30 and 50°C results in achieving the highest hydration degree during the rehydration process for freeze-dried strawberries. Between those freeze-dried fruit there were no significant differences in the course of rehydration curves. It was found that after 30 minutes of the rehydration process there was a decrease in water content, which is probably connected with changes in structure.

Isotherms of water vapour sorption for freeze-dried strawberries do not have differences in courses in the range of heating shelf temperature from 10 to 50°C. Increase of temperature to 70°C resulted in obtaining the lowest water content in the water activity range from 0.113 to 0.648.

Differentiation in temperature of heating shelf influenced the kinetics of water vapour sorption for freeze-dried strawberries without osmotic dehydration. Application of a heating shelf temperature of 30°C results in obtaining the highest water vapour sorption rate.

Sorption isotherms for freeze-dried strawberries have a sigmoidal shape characteristic for most food products. For the mathematical description Peleg's model was used.

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