SELECTED MECHANICAL PROPERTIES OF OSMOTICALLY DEHYDRATED APPLES IMPREGNATED WITH CALCIUM IONS

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A B S T R A C T

The effect of osmotic dehydration parameters on selected mechanical properties of apples was analyzed by applying an experimental design for four factors (comprising 27 measurement points) and three levels of independent variables. A compression test was performed using texture meter TA-XT2i at 1 mm·s^{-1} cross-head speed. Mechanical properties of apples were measured as to time, after that the compression force achieved 20 N.

Apples treated by mild conditions of osmotic dehydration were characterized by a shorter time to obtain a compression force of 20 N. Mechanical properties of apples after osmotic dehydration were significantly influenced mainly by temperature. Influence of time and concentration of sucrose solution was also significant. Thickness of samples did not affect the studied mechanical properties of osmodehydrated apples.

Keywords: compression force, calcium impregnation, experimental design

INTRODUCTION

Food is characterized by many important sensory features (colour, texture properties, taste etc.), which are accepted more or less by people. Food processing can greatly change product quality. Osmotic dehydration has an insignificant effect on physical and chemical changes of treated plant tissue in comparison with other processing methods used in food technology. Osmotic dehydration can be carried out to obtain several kinds of fruit products or food ingredients such as minimally processed or intermediate moisture fruit or as a pre-treatment before drying or freezing. Mild conditions of osmotic dehydration protect characteristic features of plant tissue, such as the plant tissue of fresh fruit and vegetables. Simultaneously typical semi permeability of the cell membrane can make it possible to obtain product with controlled water content, solid gain or eventually other added substances (vitamins, minerals, probiotics) (Kowalska, 2006). It could affect the mechanical properties of treated apples.

The decrease in water content during osmotic dehydration of plant tissue helps increase the shelf life of food and makes it possible to continue the next technological processing. Osmotically treated fruit and vegetables can make up one of the essential stages of production and allow for the possibility of creating new food products (Barrera et al., 2004). Relatively low energy is involved in the production and the end-product is of high-quality (Barrera et al., 2004). The presence of calcium ions in osmotic solution can have an influence on the decrease of food microflora activity. Chardonnet et al. (2001) affirmed that calcium chloride impregnated apples had a smaller susceptibility to pathogen infection than apples which were only osmotically dehydrated.

Food enhancement by adding nutritional substances during osmotic dehydration is possible. Kowalska and Gierada (2005) studied changes in osmotically dehydrated apples in an osmotic solution which included ascorbic acid in the temperature range of 20-40˚C. They affirmed that the presence of ascorbic acid in the osmotic solution caused a lowering of the water content and solid gain of apples in comparison to samples without ascorbic acid impregnation. Betoret et al. (2003) showed the possibility of probiotic impregnation to the tissue while vegetables were going through the osmotic process. Rodrigues et al. (2003) studied the influence of calcium ions on the enhancement of papaya during osmotic dehydration, too. The addition of calcium chloride caused a higher solid gain and a more stronger tissues structure.

Sacchetti et al. (2001) describe mass transfer kinetics using Peleg's model of osmotically dehydrated apples in sucrose and salt solution. They stated the suitability of the second degree polynomial model considering these factors: sucrose concentration (44.6-64.6%), NaCl salt concentration (0-2%) and temperature (14-30°C), with five levels of their value. They also found a significant influence of all these factors on analyzed indicators of mass transfer.

Few publications concerning osmotic dehydration of vegetables and fruit tissue in the presence of additional components are available. With the attention on the propriety of apple tissue as well as the easiness of the impregnation component, the test of applying osmotic dehydration of apples in the presence of calcium ions becomes purposeful. The test is also considered purposeful for examining chosen mechanical properties.

The aim of this work was optimization of the parameters of the osmotic dehydration of apples which were simultaneously impregnated with calcium ions on selected mechanical properties.

MATERIAL AND METHODS

Apples cv. 'Idared' were washed, peeled and cut into pieces of 25 x 25 mm and from 5 mm to 15 mm thick. Osmotic dehydration was carried out in special equipment included a container which allowed for sample separation and solution flow (flow stream 1 dm^3/min). The temperature of dehydration varied from 20°C to 60°C and the time from 1 to 5 hours. The concentration of sucrose solution was from 20% to 60%. Impregnation of apples with calcium ions was performed by adding calcium chloride to aqueous sucrose solution in the amount of 2% in relation to mass of osmotic solution (Torres et al., 2008). The ratio of solution mass to sample mass amounted to 20:1. After osmotic dehydration samples were rinsed with water for about 5 s, blotted and weighed. Determination of dry mass content was conducted using the drying method according to the standard PN-90/A-75101/03. The compression test was performed in texture meter TA-XT2i (Stable Micro Systems) at 1 $mm \cdot s^{-1}$ cross-head speed. Mechanical properties of apples were measured as a time after which the compression force achieved 20N (τ_{20N},). Osmotic dehydration was analyzed by applying an experimental design of Box-Behnken for four factors (comprising 27 measurement points) and three levels of independent variables (Tab. 1) (Achnazarowa and Kafarow, 1982; Walkowiak-Tomczak and Czapski, 2007). Effects of interaction between different factors were also checked.

Equation parameters for the response plane were determined (second degree polynomials) as functions of time. After that time, the compression force achieved 20N ($Y = f(\tau_{20N})$) in osmotically dehydrated apples taking into consideration the analyzed factors X_i (A, B, C, D) (Tab. 1) (Sacchetti et al., 2001):

$$Y = \Sigma a_i X_i + \Sigma a_{ii} X_i^2 + \Sigma a_{ij} X_i X_j$$

where: a_i – linear parameter, a_{ii} – polynomial parameter, a_{ij} – interaction parameter.

The model choice was determined by the "lack-of-fit" test at $p > 0.05$. A leap procedure was carried out to exclude those equation parameters having the least significant influence ($p > 0.05$) on a dependent variable (τ_{20N}).

The results were worked out statistically by multifactor variance analysis (ANOVA). The choice of model (response plane) was verified on the basis of correlation and adjustment coefficient and the "lack-of-fit" test at $p > 0.05$ with help of the Statgraphics Plus v. 4.1 program.

RESULTS AND DISCUSSION

The results were analyzed as the effect of the determined factors on the examined responses as well as the interaction occurring between the examined factors. The fitting of the model to data was carried out using the "lack-of-fit" test. Correlations between the examined factors were expressed as equations of the response surface (Tab. 2).

In this study, second order polynomial models were fitted to describe

Table 1. Experimental design: levels of examined factors according to Box-Behnken plan

Coded Factors No	A Temperature [°C]	B Dehydration time [h]	C Thickness [mm]	D Sucrose concentration [%]
1	60	3	10	60
2	40	3	10	40
3	20	3	10	20
4	20	5	10	40
5	40	3	10	40
6	60	1	10	40
7	20	3	5	40
8	40	1	15	40
9	60	3	5	40
10	20	3	10	60
11	40	1	10	20
12	60	3	15	40
13	40	1	5	40
14	20	1	10	40
15	40	3	10	40
16	40	3	15	20
17	40	5	10	60
18	40	1	10	60
19	40	5	10	20
20	40	5	15	40
21	40	3	5	60
22	40	3	15	60
23	40	5	5	40
24	60	3	10	20
25	20	3	15	40
26	40	3	5	20
27	60	5	10	40

compression time (τ_{20N}) studied in osmodehydrated apples with or without calcium chloride impregnation depending on the examined factors (Tab. 1) and their interactions (Tab. 2).

Fresh apples and those dehydrated under mild conditions were characterized by a shorter time needed to obtain compression force of 20 N. These samples were less mechanically changed (they were harder) then those for which a longer time of compression was needed. In raw material, the time was about 0.46 s (Fig. 2).

The time of the compression test needed to obtain compression force of 20 N (softness of samples) of osmotically dehydrated apples depended on all factors, but mostly on temperature and sucrose concentration (Fig. 1; Tab. 2). The highest significant interaction effect of factors on the compression test of impregnated

T a b l e 2 . Parameters of response equations describing changes of compression time τ_{20N} of osmotically dehydrated apples, and apples saturated with calcium ions

Factors	Parameters of equation	p-value	Parameters of equation	p-value
Constants	2.2795		5.9567	
A – temperature	-0.1828	0.0005*	-0.2623	0.0006*
B – time	1.0606	0.0056*	0.4863	0.0038*
C - thickness	0.1183	0.0452*	0.0530	0.0528
D - concentration	-0.0565	0.0035*	-0.0968	0.0017*
A^2	0.0023	0.0061*	0.0025	0.0049
A·B	0.0044	0.1761	0.0105	0.0362*
A·C	0.0026	0.0925	0.0034	0.0553
A·D	0.0017	0.0159*	0.0022	0.0085*
B^2	-0.1105	0.0267*	-0.0873	0.0392*
B·C	-	-	-	-
B·D	-0.0061	0.1025	-	-
C^2	-0.0089	0.0948	-0.0074	0.1226
D^2	0.0006	0.0789	0.0008	0.0433*
Lack-of-fit	0.0538		0.0532	
R^2	0.9300		0.9254	
R^2(adjusted) [%]	0.8700		0.8706	

*Significant differences

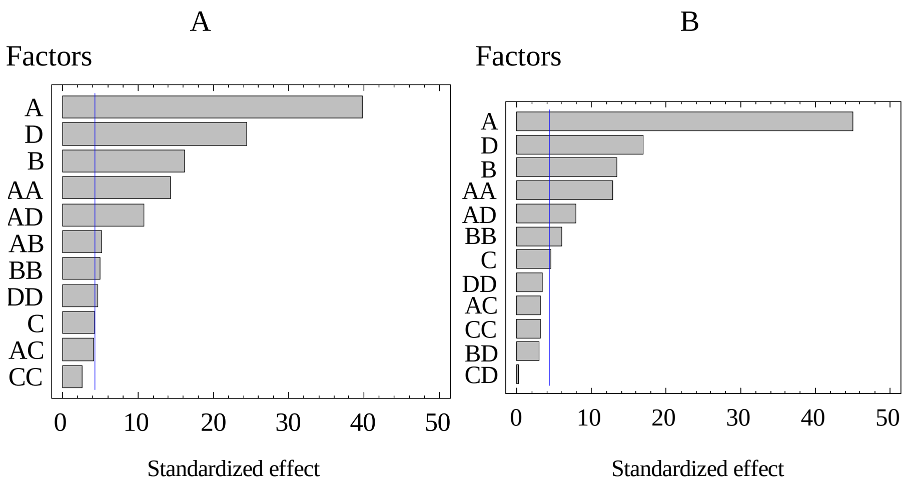

Figure 1. The effect of osmotic dehydration parameters (factors) on mechanical properties (τ_{20N}) of apples: a) osmotic dehydration; b) osmotic dehydration with calcium impregnation

apples took into account: the temperature and sucrose concentration (A-D) (Fig. 1; Tab. 2).

Mechanical properties of osmotically dehydrated apples which were simultaneously calcium impregnated mainly depended on dehydration temperature (Fig. 1; Tab. 2). The influence of the sucrose solution concentration on mechanical properties of dehydrated apples was also significant. The thickness of the samples did not have an influence as far as the mechanical properties of the apples were concerned, although the p-value was lower than the significant limit (Fig. 1; Tab. 2). The highest significant interaction effect of factors on the time of the compression test in dehydrated apples concerned the temperature–sucrose concentration pair (A-D) and a slightly less effect was found in the temperature-time pair (A-B) (Tab. 2; Fig. 1, 2).

As a result of the higher temperature of osmotic dehydration of apples, the loosened and partially damaged tissue was a particular consequence involved in the longer time of compression needed to obtain 20 N force. The temperature increase from 20˚C do 60˚C during 3 hours of osmotic dehydration of 10 mm-thick samples in 20% and 60% sucrose solution caused compression time to last almost 3 times longer. When this process was carried out with calcium ions, the compression time lasted almost 4–5 times longer (Fig. 2). A similar effect took place when the sucrose concentration was increased to the range of 20-60% (Fig. 2). Apple samples after osmotic dehydration at 20˚C were characterized by harder tissue. The harder tissue meant that a shorter time was needed to compress them to 20 N. This was a result of water content reduction. Furthermore, the addition of calcium ions to the osmotic solution caused higher osmotic pressure. As a consequence, water content decreased, the tissue lost turgidity and samples were softer.

Osmotic dehydration of 5 mm--thick samples of apples during 3 or 5 h at a temperature of 40˚C in 40% sucrose solution with or without calcium addition, did not significantly effect the time factor of the compression test. But shorter time of dehydration caused the samples to be softer and therefore about a 50% more time was needed for the compression test than osmotic dehydration within a 1 h time span (Fig. 3).

The effect of the thickness of apple samples after osmotic dehydration was significant in apples subjected to additional calcium impregnation (Tab. 2; Fig. 1). For example apples osmodehydrated at a temperature of 20˚C in 40% sucrose solution needed a slightly shorter (by up to 30%) time of compression force (τ_{20N}) for 5 mm-thick samples then for 15 mm-thick ones, especially at the higher temperature of 60˚C (Tab. 3). The addition of calcium ions to the osmotic solution during the dehydration of apples had a slightly worse influence on the mechanical properties. The parameters needed to obtain the average mechanical properties were: temperature 20-35˚C (regarding osmotic dehydration

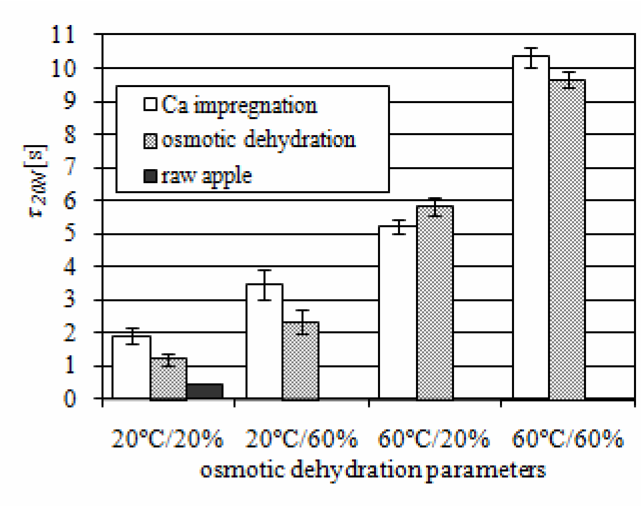

Figure 2. The effect of temperature and sucrose concentration on mechanical properties (τ_{20N}) 10 mm-thick apple samples dehydrated osmotically for 3h

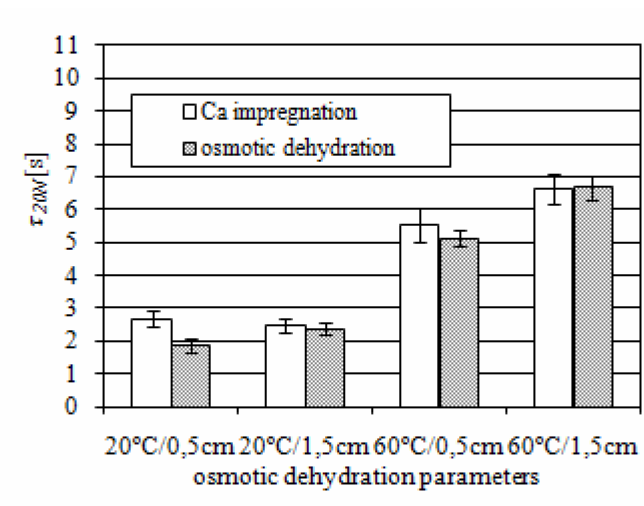

Figure 3. The effect of thickness of samples on the mechanical properties (τ_{20N}) of apples after osmotic dehydration during 3 h in 40% sucrose solution

T a b l e 3. The conditions of osmotic dehydration and calcium chloride impregnation of apples required to obtain minimum or maximum value of compression time

Factors	Value of factors			
	Osmotic dehydra-tion impregnation		Osmotic dehydration with calcium chloride	
	Min	Max	Min	Max
Temperature [°C]	20	60	36	60
Time of immersion [h]	1	4	1	5
Thickness of sam-	15	15	5	15
Sucrose concentration	25	60	23	60

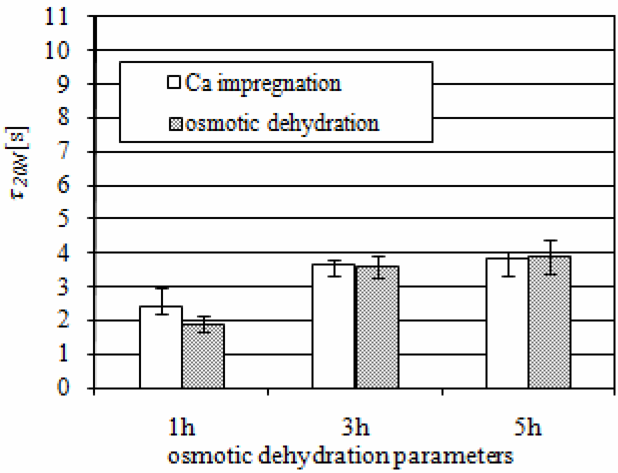

Figure 4. The effect of osmotic dehydration time on the mechanical properties (τ_{20N}) of 5 mm-thick apple samples dehydrated osmotically in 40% sucrose solution at temperature 40˚C

with and without calcium ions), time of dehydration (independently of processing with or without calcium ions impregnation) 1 h, thickness of samples 5 and 15 mm (regarding osmotic dehydration with and without calcium ions) and a low sucrose concentration of about 25% (Tab. 3). These results were in variance with other results in literature (Abud-Achila et al., 2008).

Rodrigues et al. (2003) affirmed that the addition of calcium ions to the osmotic solution at a temperature above 50°C caused hardness and enlargement of papaya. Del Valle et al. (1998) showed a similar effect.

Sereno et al. (2001) was concerned with the analysis of mass transfer in apples at variable temperatures from 5˚C to 60˚C and osmotic solution concentration (sucrose within a 40-60% range, sodium chloride within a 15-26.5% range or their mixture), taking into consideration Fick's second law. They observed that their analyzed model, taking into consideration the influence of temperature and osmotic solution concentration upon mass transfer indicators, can also be implemented in a broader range of related dehydration conditions.

Abud-Achila et al. (2008) used the orthogonal experimental design with osmotic dehydration of the yam bean in order to analyze the influence of four factors: osmotic solution concentration, temperature, dehydration time and thickness of samples at three levels of their value. They proved that the highest water loss from the yam bean can be achieved using samples with a thickness of 10 mm, 60% sucrose solution, temperature 60°C and time of 2 h. The highest increase in dry mass content took place in samples of smaller thickness (5 mm) dehydrated in less concentrated solution (50%) and after a longer dehydration time (6 h).

In order to clarify the problem, if calcium addition to osmotic solution during dehydration of apples causes improvement or worsening of mechanical properties, another experiment(s) should be done with stricter analyses.

CONCLUSIONS

Temperature, sucrose concentration and time of osmotic dehydration had a significant influence on the mechanical properties of osmodehydrated apples which were simultaneously impregnated with calcium. Sample thickness was significant also, but only in osmodehydrated apples without calcium impregnation.

By using second order polynomial models fitted in this study, interactions between temperature and time/sucrose concentration on compression time of osmodehydrated apples with or without calcium chloride impregnation were achieved.

The surface responses methodology of mechanical properties of apples measured at the time of the compression test indicated that apples osmodehydrated with calcium ions in mild conditions had worse mechanical properties then apples without calcium impregnation. Apples osmodehydrated in sucrose solution with calcium chloride were softer, especially at temperatures higher than 40˚C.

A short time of osmotic dehydration of apples (1 h) at ambient temperature 20-35˚C in 25% sucrose solution caused a lowering of compression time but after a longer time (4-5 h) the highest values were achieved. The addition of calcium ions to osmotic solution during dehydration of apples had an influence on the mechanical properties mainly at low temperatures (20˚C).

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WYBRANE WŁAŚCIWOŚCI MECHANICZNE JABŁEK ODWADNIANYCH OSMOTYCZNIE I NASĄCZANYCH JONAMI WAPNIA

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S T R E S Z C Z E N I E

Wpływ parametrów odwadniania osmotycznego w roztworze sacharozy na wybrane właściwości mechaniczne jabłek były analizowane z zastosowaniem planu eksperymentów z czterema czynnikami (27 punktami pomiarowymi) i trzema poziomami wartości zmiennych niezależnych. Badanie przeprowadzono za pomocą teksturometru TA-XT2i z prędkością przesuwania głowicy 1 mm·s^{-1}. Właściwości mechaniczne jabłek były analizowane na podstawie czasu potrzebnego do osiągnięcia siły ściskania próbek o wartości 20 N. Jabłka poddane łagodnej obróbce osmotycznej charakteryzowały się krótszym czasem, po którym uzyskano założoną wartość kompresji próbek (20 N). Wykazano największy wpływ temperatury odwadniania na właściwości mechaniczne jabłek. Inne parametry osmotycznego odwadniania jabłek, jak czas i stężenie roztworu sacharozy były również istotne, natomiast grubość próbek nie wpływała na właściwości mechaniczne badanych jabłek.

Słowa kluczowe: test ściskania, nasączanie wapniem, planowanie eksperymentów