OSMOTIC DRYING EFFECTS ON THE MASS TRANSFER
AND SHRINKAGE OF QUINCE TISSUE

UTICAJ OSMOTSKOG SUŠENJA NA PRENOS MASE
I PROMENU ZAPREMINJE TKIVA DUNJE

Milivoj RADOJČIN, Mirko BABIĆ, Ivan PAVKOV, Zoran STAMENKOVIĆ
University of Novi Sad, Faculty of Agriculture, 21000 Novi Sad, Trg Dositeja Obradovića 8, Serbia
e-mail: mradovic@polj.uns.ac.rs

ABSTRACT

The purpose of this paper is to examine the effects of osmotic drying on the mass transfer and shrinkage of quinces. The osmotic drying was performed in a sucrose and water solution. The temperatures of osmotic solution were 40 °C and 60 °C, and the initial concentrations were 50 °Bx and 65 °Bx. All four combinations were used in the experiment. An increase in the experimental parameter values intensified the mass transfer. According to the amount of exchanged matter, it is possible to determine the shrinkage of quince tissue. The most significant changes in quince tissue volume were measured at the highest levels of experimental parameters. After combined drying (osmotic + convective), the effects of exchanged matter on the shrinkage ceased. More notable volume changes were measured in the quince samples after convective drying (without osmotic drying) in comparison with the samples dried osmotically. The experimentally results indicate the positive effects of osmotic drying on the volume of dried quinces.

Keywords: quince, osmotic drying, shrinkage, mass transfer.

INTRODUCTION

The osmotic dehydration process involves a partial removal of water from a given food using a hypertonic solution consisting of one or more solutes (Ponting, 1966). The difference in osmotic pressure obtained from the system leads to a flow of water from the food to the solution and an opposite flow of solutes from the syrup to the product, although in smaller proportions. Furthermore, a third flow of solids may take place from the food to the solution, which, although on a much smaller scale, may lead to a meaningful loss of product quality (Raoult-Wack, 1994). One of the most important physical alterations caused by biomaterial drying is the change in geometric features, i.e. the changes in shape and volume (Frias et al., 2010). The change in the shape of materials during the drying process affects the quality of the final product. The intensity of such changes depends on the moisture transport mechanism, i.e. drying methods and conditions affect the intensity of shape and volume change.

Material volume changes occurring during osmotic drying can be observed at various structural levels which encompass micro- and macrostructural levels, i.e. cell and sample levels. Drying and water loss cause changes in the cell microstructure and volume (Koc et al., 2008). Moisture content reduction and changes in the cell membrane features cause the shrinkage and deformation of cells, plasmolysis, cell separation and disruption of intercellular links. Osmotic drying as a pretreatment of convective drying positively affects the preservation of shape and volume reduction as stated by Karathanos et al. (1996). In addition to the improvement of physical properties, other advantages of osmotic drying are the prolonged storage life with the higher final moisture content and reduced drying time to the final moisture content (Babić Lijiljana and Babić M. 2003).

The quince is a seasonal fruit mostly used for the production of brandy, juices, jams, etc. However, due to its pleasant aroma, the quince can provide an interesting material for drying. The purpose of this paper is to determine the effects of osmotic drying on the mass transfer and volume change of quince tissue.

MATERIAL AND METHOD

The quinces of the Leskovačka variety were used in the experiment. Previous researches have shown that this variety is favourable for drying from the perspective of shape and dimension (Radojčin et al., 2011). The variety is characterised by a very pleasant aroma, which is very reputable in the processing industry. The osmotic drying was performed in a sucrose and water solution in an experimental osmotic dryer. The dryer is self-designed (Babić et al., 2005). The device provides agitation of the solution. The material-solution weight ratio was higher than 1:10. The temperatures of osmotic solution were 40 °C and 60 °C, and the initial concentrations were 50 °Bx and 65 °Bx. The combinations of these factors will be referred to as treatments in the further course of the paper. The treatments will be named the first, second, third and fourth. The first, second, third and fourth treatment had the parameter combinations of 50 °Bx-40 °C, 50 °Bx-60 °C, 65 °Bx-40 °C, 65 °Bx-60 °C, respectively. The solution temperature of 60 °C was used in the experiment due to the hardness of quince fruits. The osmotic drying lasted for 180 minutes and it was used as a pretreatment. The kinetics of osmotic drying was evaluated on the quince samples with the dimension of 15x15x15 mm. The convective drying was conducted at the temperature of 50 °C and...
the air velocity of 1 m/s. The drying was conducted in a self-designed experimental convective dryer (Pavkov et al., 2009). The moisture content of fruits was determined by the standard hot air oven method keeping the fruit in the oven for 24 h at 105 °C. The solid gain (SG) was determined by using the following relation:

\[ SG = \frac{s_0 - s}{m_0} \]  

(1)

where \( m_0 \) is the initial mass, \( s \) is the dry mass after time (t) of osmotic dehydration, \( s_0 \) is the initial dry mass. The changes in the volume of quince cube samples (occurring during drying) with the dimension of 15x15x15 mm were measured. The volume measurements were conducted every 20 minutes by stopping the osmotic drying process. Each of the 10 measurements, done in total during 180 minutes of drying, were conducted on eight samples (cubes). The volume measurements were done after the combined drying (osmotic + convective) as well. The volume of the control samples, which were not osmotically dried, was also measured.

The volume of each sample was calculated on the basis of the measured values of the thrust force exerted on the water-immersed sample by the following equation (Moshenin, 1986):

\[ V = \frac{m_{li} - m_l}{\rho_f} \]  

(2)

where \( m_{li} \) is the mass of liquid and the immersed sample, \( m_l \) is the mass of liquid, and \( \rho_f \) is the liquid density.

Volume changes during osmotic drying were calculated by the following equation:

\[ SV = \frac{V_0 - V_i}{V_0} \]  

(3)

where \( V_0 \) is the sample volume prior to osmotic drying and \( V_i \) is the sample volume at a certain time of osmotic drying.

The sample mass measurement was done by an analytical balance with 0.001 g readability. The regression analysis of quince volume change during osmotic drying was conducted in relation to the sample moisture content. The modelling was done by the Statistica 12 software. The model coefficients were calculated on the basis of the experimental data by means of a nonlinear regression analysis. The accuracy of the regression models was determined on the basis of the correlation coefficient (R) and the mean percentage error (MPE). A model describes the examined value more accurately at higher values of the correlation coefficient and lower values of the mean percentage error. It is generally acknowledged that the values of the mean percentage error lesser than 10 % indicate a higher goodness of fit. The experimental data modelling was done by the following equations suggested in the literature:

Mayor and Sereno (2005): \[ SV = 1 + ao + bo^2 + co^3 \]  

(4)

Lozano et al., (1983): \[ SV = a + b \frac{\omega}{\omega_0} + c \text{exp}\left(\frac{d}{e + \omega}\right) \]  

(5)

Ratti (1994): \[ SV = a + b_0 + c \omega + d \omega^2 \]  

(6)

Mayor and Sereno (2004): \[ SV = a + b \frac{\omega}{\omega_0} + c \left(\frac{\omega}{\omega_0}\right)^2 \]  

(7)

The goodness of fit between the experimental and calculated data was determined on the basis of the correlation coefficient (R) and the mean percentage error (MPE). The correlation coefficient and the mean percentage error were calculated on the basis of the following equations:

\[ R = \sqrt{1 - \frac{\sum_{i=1}^{n} (SV_{i(cal)} - SV_{i(exp)})^2}{\sum_{i=1}^{n} (SV_{i(esp)} - SV_{i(cal)})^2}} \]  

(8)

\[ SPG = \frac{100}{n} \sum_{i=1}^{n} \frac{SV_{i(esp)} - SV_{i(cal)}}{SV_{i(esp)}} \]  

(9)

where \( SV \) is the volume shrinkage (experimental and calculated values) and \( n \) is the number of measurements.

**RESULTS AND DISCUSSION**

**Osmotic Drying**

The experimental data on the osmotic dehydration kinetics and solid gain of quinces under different process conditions are shown in Figure 1 and Figure 2. The most intensive changes in moisture content were measured in the first 20 minutes of the process. After 20 minutes, the reduction of moisture content decreases between each measurement. The samples dehydrated in the sucrose solution of 65 °Bx had lower moisture content at the end of the process. The moisture contents of the samples after 180 minutes of osmotic process in the 50 °Bx-40 °C, 50 °Bx-60 °C and 65 °Bx-40 °C; 65 °Bx-60 °C sucrose solutions were 62.1 %; 58.7 %; 53.6 % and 45.3 % (wet basis), respectively. The results indicated that the most intensive decrease in the moisture content occurred with an increase in the osmotic solution concentration. Similar results were obtained by Falade and Shogaolu (2009) when using sucrose solutions in the osmotic dehydration of pumpkins. Kadam and Dhingra (2011) also obtained similar results by using sucrose solutions in the osmotic dehydration of bananas. Higher process temperature promote faster water transfer on the surface due to lower viscosity of the osmotic medium (Dalla Rosa et al., 2011). The influence of osmotic parameters on solid gain during 180 minutes of the drying process are shown in Figure 2. The solid gain increased with an increase in the osmotic solution concentration. The highest solid gain of 9.3 % was determined in the sucrose solution of 60 °C and 65 °Bx. The lowest solid gain of 5.5 % was determined in the sucrose solution of 40 °C and 50 °Bx. Some authors have reported that higher concentrations do not increase solid gain. Giraldo et al. (2003) found that water transfer increased during the osmotic drying of mangos at the solution concentration of 45 °Bx. The water transfer decelerated between 55 °Bx and 65 °Bx, probably due to the high viscosity of the sucrose solution. The measurement was conducted at the solution temperature of 30 °C.
Equation 10 is used for calculating the volume change of the quince sample during osmotic drying:

\[
SV = \frac{m_i \omega_i - m_o \omega_o}{\rho_s} \cdot \frac{m_o - m_i}{\rho_s} \cdot 100
\]  

(10)

where \( m_i \) is the initial sample mass, \( m_s \) is the sample mass at a certain time of drying, \( \omega_i \) is the initial sample moisture, \( \omega_o \) is the sample moisture at a certain time of drying, \( m_o \) is the dry matter mass of the fresh sample, \( m_s \) is the dry matter mass at a certain time of osmotic drying, \( \rho_s \) is the water density, \( \rho_t \) is the sucrose density and \( \rho_o \) is the quince tissue density.

Equation 10 multiplied by the water density \( \rho_w \), equals:

\[
SV = \frac{m_i \omega_i - m_o \omega_o}{\rho_t} \cdot \frac{m_o - m_i}{\rho_w} \cdot 100
\]  

(11)

The quince tissue density was measured by immersing the sample in a liquid of known density. The quince tissue density is 0.96457 kg/dm³ and the sucrose crystal density is 1.587 kg/dm³ (Asadi, 2006). The water density is 1 kg/dm³. It is necessary to correct Equation 11 by multiplying the denominator by 1.1 due to the air in the tissue (Koc, 2008). Introducing these numerical values in Equation 11 creates the final equation for calculating the cube sample volume change:

\[
SV = \frac{m_i \omega_i - m_o \omega_o}{\rho_t} \cdot \frac{m_o - m_i}{1.587 \cdot 1.1}
\]  

(12)

Figure 4 a,b,c and d display the measured and calculated values of the volume change by using Equation 12.

The values of R and MPE are shown in Table 1. Based on these values and the graphic display of the experimental and calculated values of the volume shrinkage, it can be concluded that Equation 12 successfully predicts the quince volume change during osmotic drying.

**Table 1. Values of the correlation coefficient and the mean percentage error**

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Correlation coefficient R</th>
<th>Mean percentage error MPE (%)</th>
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<tbody>
<tr>
<td>40 °C, 50 °Bx</td>
<td>0.9832</td>
<td>3.24</td>
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<tr>
<td>60 °C, 50 °Bx</td>
<td>0.9962</td>
<td>0.93</td>
</tr>
<tr>
<td>40 °C, 65 °Bx</td>
<td>0.9850</td>
<td>0.22</td>
</tr>
<tr>
<td>60 °C, 65 °Bx</td>
<td>0.9934</td>
<td>2.12</td>
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</tbody>
</table>

The analysis of osmotically dried quince samples (which were previously in a deepfreeze) was conducted in order to confirm the obtained results. The samples indicated an increase in the dry matter of 43% in comparison with the initial sample mass. Due to a large amount of sucrose diffusing into the tissue, a decrease in the volume of merely 12% was recorded after osmotic drying (Radojčin et al., 2014). Equation 12 can be successfully applied to the results obtained during the osmotic drying of previously frozen quince samples. This confirms the fact that the change in material volume during osmotic drying depends on the matter exchange ratio.
Figure 5 displays the quince samples after 180 minutes in the osmotic solution with the temperature of 60 °C and the concentration of 65 °Bx, with and without previous freezing. A compound “methyl blue” is added to the solution as an indicator of sucrose penetration into the tissue during osmotic drying. The samples demonstrate different penetration depths of sucrose, which accounts for the significant volume preservation of previously frozen samples. The cell membrane was damaged during the freezing of quince fruits.

The damaged cell membrane is no longer a barrier for the entrance of sucrose molecules into the cell.

Figure 6 shows a microscopic image of the cross section of the samples shown in Figure 5. The tissue cells which were previously frozen kept the somewhat circular shape, whereas the tissue cells which were not frozen suffered more significant deformations, i.e. volume changes. The frozen tissue indicates more intensive colour properties, i.e. greater depths of sucrose penetration. According to the cross section of quince samples, the cell volume reduction is uneven. The volume changes at the microstructural level are the most significant close to the tissue surface due to greater moisture diffusion.
The volume change in relation to the moisture content during osmotic drying

The volume change during osmotic drying is in accordance with the volume change dependence on the amount of matter exchanged. The most significant change in volume during osmotic drying amounting to 52.66 % was recorded in the osmotic solution treatment with the temperature of 60 °C and the concentration of 65 °Bx. The most significant change in the sample moisture was recorded in the treatment. The slightest volume change of 36.23 % was measured in the first treatment as well as the slightest change in the quince moisture content. An increase in the temperature and concentration of osmotic solution intensifies a decrease in volume. Based on the measured results of the volume change, it can be concluded that the solution concentration is a more influential factor than the solution temperature. The most intensive volume changes were measured in the first 20 minutes of osmotic drying. As volume depends on the material moisture content, the volume reduction in neither experiment was final due to the fact that the moisture equilibrium was not achieved during 180 minutes of osmotic drying. The results are in accordance with the research results on quince volume change as stated by Babić et al. (2008).

The volume change in relation to the quince sample moisture content is shown in Figure 7 and 8. The volume changes at the same sample moisture contents are different. The differences between the measured values are slight, which can be a consequence of sucrose diffusing into the quince tissue. The differences are the most significant at the beginning of osmotic drying and become slighter during the process.

The quince volume change after convective drying

The results of the quince volume change after convective drying are displayed in Table 2. The mean value of the volume change is $SV = 68.97\pm 1.19$ % at similar sample moisture contents $\omega = 23.68\pm 0.7$ %.

If the measured values of the volume change after osmotic and convective drying are compared, it can be concluded that after convective drying the volume change is affected only by the amount of moisture migrating from the samples. After 180 minutes of osmotic drying, the effect of the exchanged matter relation between moisture and sucrose is noticeable. At lower moisture contents of the quince samples, the volume change ceases to depend on the relation of moisture/sucrose matter exchange. The amount of sucrose in the tissue does not affect the sample volume due to the sufficient space created by a significant decrease in moisture.

The significantly higher volume change of 82.63 % was measured in the control samples which were not osmotically dried. If the values of the sample volume change after convective drying are compared, with and without osmotic drying, the effect of sucrose on the volume is evident, i.e. the sample volume shrinkage is slighter, which is definitely a favourable effect of osmotic drying.
Table 2. Values of the quince shrinkage $SV$ at different moisture contents of the dried quince samples with and without osmotic drying

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</thead>
<tbody>
<tr>
<td>$t=40^\circ C$, $c=50^\circ Bx$</td>
<td>$SV$ (%)</td>
<td>70.95</td>
<td>69.39</td>
<td>69.33</td>
<td>70.62</td>
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<tr>
<td>$t=60^\circ C$, $c=50^\circ Bx$</td>
<td>$SV$ (%)</td>
<td>67.21</td>
<td>70.12</td>
<td>70.52</td>
<td>70.81</td>
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<tr>
<td>$t=40^\circ C$, $c=65^\circ Bx$</td>
<td>$SV$ (%)</td>
<td>65.02</td>
<td>68.80</td>
<td>70.70</td>
<td>69.44</td>
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<tr>
<td>$t=60^\circ C$, $c=65^\circ Bx$</td>
<td>$SV$ (%)</td>
<td>69.85</td>
<td>70.24</td>
<td>72.03</td>
<td>71.49</td>
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<tr>
<td>Without osmotic drying</td>
<td>$SV$ (%)</td>
<td>78.63</td>
<td>79.30</td>
<td>81.91</td>
<td>82.63</td>
<td></td>
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<tr>
<td>$t=40^\circ C$, $c=50^\circ Bx$</td>
<td>$\omega$ (%)</td>
<td>28.31</td>
<td>23.57</td>
<td>21.26</td>
<td>20.66</td>
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<tr>
<td>$t=60^\circ C$, $c=50^\circ Bx$</td>
<td>$\omega$ (%)</td>
<td>24.51</td>
<td>19.31</td>
<td>16.62</td>
<td>16.33</td>
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<tr>
<td>$t=40^\circ C$, $c=65^\circ Bx$</td>
<td>$\omega$ (%)</td>
<td>31.76</td>
<td>27.25</td>
<td>25.08</td>
<td>22.81</td>
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<tr>
<td>$t=60^\circ C$, $c=65^\circ Bx$</td>
<td>$\omega$ (%)</td>
<td>23.84</td>
<td>20.55</td>
<td>19.70</td>
<td>18.56</td>
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<tr>
<td>Without osmotic drying</td>
<td>$\omega$ (%)</td>
<td>34.95</td>
<td>28.69</td>
<td>26.08</td>
<td>23.01</td>
<td></td>
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</table>

The highest values of the correlation coefficient and the lowest values of the mean percentage error were recorded by applying Equation 6 (Ratti, 1994). However, by applying Equation 7 (Mayor and Sereno, 2004) similar values of the correlation coefficient and the mean percentage error were obtained but with one less coefficient, which makes the equation simpler for application.

CONCLUSION

Based on the obtained results, it can be concluded that an increase in the osmotic solution temperature ranging from 40°C to 60°C and the osmotic solution concentration ranging from 50° “Bx to 65° “Bx intensifies the process of matter exchange during 180 minutes of quince drying. The most significant changes in the moisture content and the dry matter mass increase were recorded within the first 20 minutes of osmotic drying. The volume change of quince cubes during 180 minutes of osmotic drying depends on the amount of exchanged matter. The effect of the amount of exchanged matter on the quince sample volume was confirmed by Equation 12. At lower moisture contents of the quince samples, recorded during convective drying, the water-sucrose exchange matter relation ceases to impact the volume change. Due to the moisture diffusion, the sucrose in the tissue has enough space not to affect the sample volume. The comparison of the SV values after convective drying, with and without osmotic solution, confirmed significant differences, which indicates the positive effects of osmotic drying.

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