EVALUATION OF STRUCTURAL PROPERTIES OF FREEZE-DRIED FOOD PRODUCTS

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Abstract: The effect of process conditions on the structural properties of freeze-dried food products was investigated. Rice, boiled for different time periods, and potatoes, mushrooms and strawberries, were freeze-dried under various vacuum conditions. The porosity of freeze-dried materials decreased with the increment of the applied pressure during freeze-drying. In addition, the porosity of freeze-dried rice increased with the increment of boiling time. Simple mathematical models were developed in order to correlate the structural properties with process conditions. The above results were also strengthened by mercury porosimetry, Scanning Electron Microscopy images, and image analysis. Sorption isotherms were also examined for all the dried products.

Keywords: Food products; Freeze drying; Mercury porosimetry; SEM; Structure

INTRODUCTION

The microstructure and morphology of foods and consequently, their quality, is significantly changed during drying and as a result, is related to the drying method and selected conditions applied [Koc et al., 2007]. Freeze-drying is considered one of the most advanced methods for drying high-value products [Krokida et al., 1998]. Depending on the process conditions, the sublimation of ice crystals creates pores or gaps with different characteristics.

Density, porosity and pore size distribution, are very useful properties for food process design, since they characterize the texture and quality of dehydrated products [Koc et al., 2007]. Krokida et al. (1998) and Rahman et al. (2003) examined the effect of drying conditions on structural properties of freeze-dried food products and abalone, respectively. Regier et al. (2007) determined the pore size distribution of bread and extruded snacks using image analysis. In addition, sorption isotherms of foods are important in food processing and help to the determination of the shelf-life of the products.

The objective of the present research is the determination of the effect of process conditions on the structural properties of freeze-dried food products, as well as the examination of their sorption isotherms. Simple mathematical models were developed, in order to predict the values of porosity.
The true volume of the samples was estimated using a helium stereopycnometer (Quantachrome multipycnometer MVP-1). The true density is given by the equation:

$$\rho_{ts} = \frac{m_t}{V_s}. \quad (2)$$

The porosity is estimated using the equation:

$$\varepsilon = 1 - \frac{\rho_{ts}}{\rho_o}. \quad (3)$$

Porosity and pore size distribution of representative samples were also measured using a mercury porosimeter (Porosimeter 2000, Fisons Instruments).

**Mathematical modeling**

Simple mathematical power models were developed for the prediction of porosity of food products from process conditions. The models are expressed with Eq. (4) for rice and Eq. (5) for agricultural products:

$$\varepsilon = k_o \cdot \left(\frac{P}{P_o}\right)^{m_o} \cdot \exp\left(\frac{t}{t_o}\right)^{n_o}. \quad (4)$$

$$\varepsilon = k_1 \cdot \left(\frac{P}{P_o}\right)^{m_1}. \quad (5)$$

The GAB model was applied to the sorption isotherms of freeze-dried products:

$$X = X_m \cdot C \cdot K \cdot \alpha_w \cdot \left(1 - K \cdot \alpha_w + C \cdot K \cdot \alpha_w\right). \quad (6)$$

**Scanning Electron Microscopy (SEM)**

Freeze-dried materials were coated with gold and photographed using a Scanning Electron Microscope (Quanta 200 FEI (2004)) operated at 20 kV for rice kernels and 25kV for agricultural products.

**Data Analysis**

The obtained data from SEM images were processed with image analysis in order to estimate the pore size distribution. Regression analysis was used to estimate the models’ parameters.

**RESULTS & DISCUSSION**

**Bulk Density, True Density and Porosity**

Bulk density of freeze-dried materials decreased significantly \((p<0.001)\) with the decrement of the applied pressure. In addition, bulk density of rice decreased significantly \((p<0.001)\) with the increment of the boiling time. True density of freeze-dried food products was estimated using Eq. 2 and considered constant, equal to the density of the solid material. The values of true density are 1504±33 kg/m\(^3\) for rice, 1543±12 kg/m\(^3\) for potatoes, 1591±39 kg/m\(^3\) for strawberries and 1602±56 kg/m\(^3\) for mushrooms.

Figs. 1a and 1b, illustrate that porosity decreased as the pressure increased. Mushrooms presented more porous structure, whereas potatoes showed the lowest porosity. In addition, porosity of freeze-dried rice increased significantly \((p<0.001)\) while elongating the boiling procedure. The results of parameter estimation of the mathematical model for porosity of freeze-dried foods, are summarized in Table 1.

![Fig. 1. Porosity of freeze-dried samples versus process conditions a) rice, b) agricultural products.](image)

![Table 1. Parameter estimation for porosity of freeze-dried food products](image)

<table>
<thead>
<tr>
<th>Material</th>
<th>(k_o)</th>
<th>(m_o)</th>
<th>(n_o)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rice</td>
<td>0.208</td>
<td>±0.001</td>
<td>-0.017</td>
</tr>
<tr>
<td>Potato</td>
<td>0.876±0.001</td>
<td>0.002</td>
<td>-0.014±0.001</td>
</tr>
<tr>
<td>Strawberry</td>
<td>0.929±0.001</td>
<td>0.002</td>
<td>-0.014±0.001</td>
</tr>
<tr>
<td>Mushroom</td>
<td>0.247±0.001</td>
<td>0.002</td>
<td>-0.010±0.001</td>
</tr>
</tbody>
</table>

Volume of mercury intruded of representative freeze-dried samples, is shown in Figs. 2a and 2b, for rice and potatoes, respectively. The same results were observed for strawberries and mushrooms.

![Fig. 2. Volume of mercury intruded for freeze-dried a) rice \((t=12\ \text{min})\), b) potatoes, for various pressures.](image)
Moisture sorption isotherms

Figs. 3a and 3b, present the sorption isotherms of freeze-dried rice and potatoes, respectively, for various applied pressures. The equilibrium moisture content increased with the increment of water activity. The same results were observed for mushrooms and strawberries. The estimated constants of the GAB model for a representative pressure (rice: \(P=0.04\) mbar, agricultural products: \(P=0.06\) mbar) are presented in Table 2.

![Fig. 3. Sorption isotherms of freeze dried a) rice (\(t=4\) min), b) potatoes.](image)

### Table 2. Estimated values of constants (\(X_m, K, C\)) of GAB model for freeze-dried products

<table>
<thead>
<tr>
<th>Material</th>
<th>(X_m)</th>
<th>(K)</th>
<th>(C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rice</td>
<td>9.628</td>
<td>0.598</td>
<td>10.219</td>
</tr>
<tr>
<td>Potato</td>
<td>0.053</td>
<td>0.896</td>
<td>14.787</td>
</tr>
<tr>
<td>Strawberry</td>
<td>0.193</td>
<td>0.856</td>
<td>0.813</td>
</tr>
<tr>
<td>Mushroom</td>
<td>0.047</td>
<td>0.991</td>
<td>3.369</td>
</tr>
</tbody>
</table>

Scanning Electron Microscopy

The above mentioned results are visualized with SEM images presented in Fig. 4.

![Fig. 4. Microstructure of freeze-dried products as a function of process conditions.](image)

Fig. 5 presents the pore size distribution of freeze-dried rice and potatoes, estimated using image analysis. The same results were obtained for all agricultural products.

CONCLUSIONS

Process conditions affected significantly the structural properties of dried food products. The porosity increased when decreasing the applied pressure during freeze-drying. Rice boiled for lower time period presented the lowest value of porosity. Mushrooms presented the highest porosity, whereas potatoes showed the lowest one. The above results were strengthened using mercury porosimetry and were visualized with SEM and image analysis. Sorption isotherms revealed that moisture content increased with the increment of water activity.

NOMENCLATURE

- \(m\): mass \(g\)
- \(P\): pressure \(mbar\)
- \(P_o\): reference pressure \(mbar\)
- \(V\): volume \(cm^3\)
- \(t\): time \(min\)
- \(t_o\): reference time \(min\)
- \(k_{eo}, m_o, n_o, k_1, m_1\): parameters
- \(X\): moisture content (d.b.) \(kg\) water/kg dry solids
- \(C, K\): constants

Greek letters

- \(\varepsilon\): porosity
- \(\rho\): density \(kg/m^3\)
- \(\alpha\): activity

Subscripts

- \(t\): true
- \(b\): bulk
- \(t^*\): total
- \(s\): dry solids
- \(m\): absorbed monolayer
- \(w\): water

REFERENCES


