

DMA thermal analysis of yacon tuberous roots**

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Abstract. Specimens prepared from yacon roots in first two weeks after harvest were tested by dynamic mechanical analysis thermal analysis at temperatures between 30 and 90°C. No differences between different parts of roots were proved. There were indicated some differences in the test parameters that were caused by short time storage of the roots. One source of the differences was loss of water during the roots storage. The measured modulus increased during short time storage. Detailed study of changes of the modulus during the specimen dynamic mechanical analysis test provided information about different development of the storage and loss moduli during the specimen heating. The observed results can be caused by changes in cellular membranes observed earlier during vegetable heating, and by composition changes due to less stable components of yacon like inulin.

Keywords: yacon, storage, inulin, stability, dynamic mechanical analysis

INTRODUCTION

The yacon (*Smallanthus sonchifolius*, Syn.: *Polymnia edulis*, *P. sonchifolia*) is a plant that was originally cultivated in the area of the Andes (in Peru, Bolivia, Ecuador) as a source of sweet roots. The roots contain storage sweet polysaccharide inulin, similarly to a few other plants like chicory (*Cichorium intybus* L.), dahlia, Jerusalem artichoke (*Helianthus tuberosus* L.) and others. Inulin is a linear fructan consisting of β -(2 \rightarrow 1)-linked D-fructofuranose units and one terminal α -(1 \rightarrow 2)-linked D-glucopyranose unit. The polydispersity of the inulin molecular chain length depends not only on the plant species but also on plants life cycle (De Leenheer and Hoebregs, 1994).

Food industry has shown an increasing interest in inulin because its sweetness and impossibility of its direct human digestion (Nilsson *et al.*, 1988). It is an interesting soluble fibre that selectively stimulates the growth of Bifidobacteria

in the large intestine (Roberfroid *et al.*, 1993), which results in a more healthy intestinal microflora. Moreover, the high molecular weight fraction of inulin makes it possible to replace fat in dietary food products.

The industrial use of inulin has to be connected with its isolation from natural sources. Another and more natural way of using inulin in human nutrition is consumption of inulin as a part of a natural product. In many cases it is possible, like in the case of yacon which is a component of many traditional dishes in the area of the Andes (Fernández *et al.*, 2005, Lebeda *et al.*, 2011). Yacon storage is difficult due to inulin losses by transformation to simple saccharides (Blecker *et al.*, 2002, 2003). Storage temperature, as well as air humidity, strongly influence the level of the inulin losses in stored yacon (Doo *et al.*, 2000, Narai-Kanayama *et al.*, 2007). In some cases heating is involved in the preparation of dishes from this product (Miyaguchi *et al.*, 2012). This is why the details on the physical properties and on the thermal-dependent yacon properties are valuable for its storage and processing.

Isolated and dried inulin is relatively stable up to temperatures close to 150°C. DSC (differential scanning calorimetry – Laye, 2002) thermal analysis above room temperature gave two endothermic peaks, the first above ~70°C, connected with drying of the specimen, and the second at temperature above ~150°C, connected with melting of inulin (Dan *et al.*, 2009; Panchev *et al.*, 2011; Roukart *et al.*, 2009). Glibowski and Bukowska (2011) conclude that inulin is stable also in solutions at temperatures lower than ~100°C in non-acid conditions.

In this study we used dynamic mechanical analysis (DMA) thermal analysis for the determination of the mechanical properties of yacon tissue at temperatures between 30 and 90°C to detect changes caused by short-time storage.

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MATERIAL AND METHODS

Twenty roots with about the same dimensions (length approx. 15 cm, max. diameter approx. 8 cm) were cleaned and then put into refrigerator and stored there till the experiment at 6°C. The roots were in a special box to prevent water evaporation. The experimental material was divided into two groups: the first group was denoted as I and it was tested during the first four days after harvest. The second group (II) of ten roots was tested in 8-12 days after harvest. The test began by determination of moisture content (wet basis) of one part of root and determination of its mean density (Table 1). Moisture content was determined gravimetrically (4 h of drying at 104°C was used), the density was determined by two weightings (in air and in water) using the Archimedes law.

Rectangular specimens of 8 (width) x 3 (thickness) x 22 mm (length) with the long axis parallel to the root axis were cut from different parts of roots (C – central, I – internal: below surface but not in its central part, and S – surface that contained the outer part of the tested tuber) using special cutting jigs. From one root, three specimens were prepared. The process was repeated on 10 different roots for every experimental groups (I and II).

The DMA experiment was performed with a special DMA instrument, constructed by RMI company (Pardubice, Czech Republic), model DX04TC. The specimen was mechanically fixed in two points so that the longitudinal axis was perpendicular to the fixing jaws. The free length of the specimen between the jaws was 4.4 mm. The height of the fixed specimen was approximately 3 mm. One of the jaws was fixed, while the other moved up and down with a constant amplitude of 1 mm and frequency of 1 Hz. The force connected with the oscillation was recorded, being the basis for the complex moduli determination (storage (*SM*) and loss (*LM*)). The modulus values (originally in Pa) sensitively depend on precision form of the tested specimen. To prevent this source of variation we calculated the resulting *SM* and *LM* values as a ratio of the value obtained for *SM* at 30°C. This method is suitable for determination of peak positions and the slope analysis. Every experiment started at a temperature of 30°C and 90% air humidity in the test chamber. The humidity was kept constant during the whole experiment, while the temperature was increasing up to 90°C with a heating rate of 1°C min⁻¹.

The obtained values were analyzed using the standard laboratory software Origin[®], OriginPro Ver. 7 (Origin Lab, Northampton, MA, USA) in corporation with Excel scripts. The analysis was focused on the temperature plots of *SM* and *LM* as basis for the calculation of:

- the loss tangent $LT = LM/SM$ and
- the temperature slope of both modulus components $SSM = \frac{dSM}{dt}$ and $SLM = \frac{dLM}{dt}$.

The outliers were identified by Tukey outlier filter (Hoaglin *et al.*, 1983) and filtered from data sets. $Y < (Q1 - 1.5IQR)$ and $Y > (Q3 + 1.5IQR)$ where $Q1$, $Q3$ are the first and third quartiles, Y represents outliers. The interquartile range is calculated as follows $IQR = (Q3 - Q1)$. Data were analyzed using the software Origin[®] with initial data smoothing by averaging every 5 neighbouring points, followed by differentiating the smoothed data.

The data obtained from the analysis of the slopes of the individual plots were then unified and classified into classes of 1°C wide. The basic statistical analysis of the individual classes was then done using a special Excel script made for this purpose.

RESULTS AND DISCUSSION

Mean density value (ρ) of the tested specimens was 993 kg m⁻³ with coefficient of variation 3%. No difference either among different parts of roots or the time of testing was proved for the specimen density. For moisture content of specimens tested in different weeks after the harvest, differences were proved; in the 1st week the moisture content w.b. was 91.8±4.0%, whereas the same quantity in the 2nd week was 82.8±7.0%. It means that some portion of the root water was evaporated during the storage of the roots. The observed yacon root density was lower than the density of water, and it means that yacon pores were filled at least partially with air. We will try to estimate the level of the aeration. Supposing that the volumes of the components (water, dry matter and air) and their masses are additive, and mass of air can be omitted, then the product aeration (ratio of air and total volumes) A should be estimated as:

$$A = \left[MC \left(1 - \frac{\rho}{\rho_w} \right) + (1 - MC) \left(1 - \frac{\rho}{\rho_{DM}} \right) \right],$$

where: ρ_w is water density, ρ_{DM} dry matter density and MC root moisture content. Using $\rho_w \approx 1000 \text{ kg m}^{-3}$ and $\rho_{DM} \approx 1500 \text{ kg m}^{-3}$ (Gibson, 2012), we obtained results presented

Table 1. Basic characteristics of the measured moduli. The differences in data obtained in different weeks (I, II) were proved on 95% confidence level (*SM* initial is excluded)

Week	<i>SM</i> initial (%)	<i>SM</i> final (%)	<i>LM</i> initial (%)	<i>LM</i> final (%)
I	100	6.2	20.3	1.94
II	100	3.7	18.1	1.65

in Fig. 1. The root aeration is nearly doubled after one week storing: it increased from the value of about 3.4% at $MC = 91.8\%$ in week I to 6.8% at $MC = 82.8\%$ in week II.

Experimental data on storage modulus (SM) and loss modulus (LM) in week I represents two decreasing functions without any changes that were observed in potato tissues containing starch (Blahovec and Lahodová, 2012a) (Fig. 2). This fact could be explained by different kinetics of inulin swelling that needs about 1 hour for the whole process (Vervoort *et al.*, 1998). The basic properties of the modulus plots versus temperature at the initial values (at 30°C) and final values (approximately at 90°C) are given in Table 1. The values obtained in week II are lower than those from the first week.

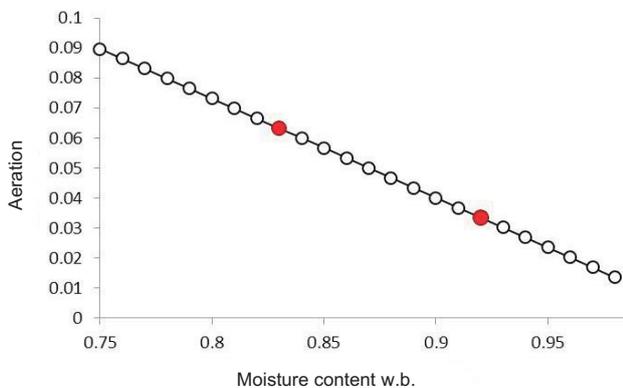


Fig. 1. Plot of aeration (A) calculated from equation versus moisture content (MC) for yacon roots. $\rho_w = 1\,000\text{ kg m}^{-3}$, $\rho = 993\text{ kg m}^{-3}$ and $\rho_{DM} = 1\,500\text{ kg m}^{-3}$ (Gibson, 2012) were used. Results corresponding to mean values of moisture content in weeks I and II are denoted by dark points.

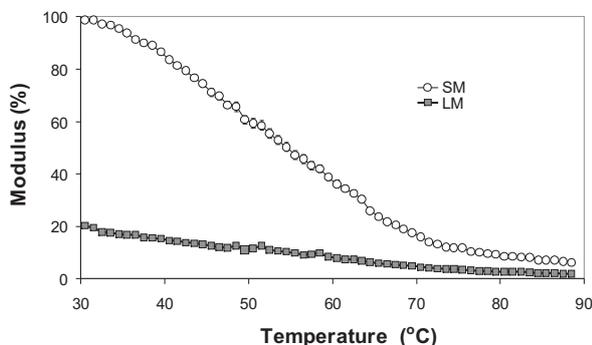


Fig. 2. Plot of modulus components (storage modulus – SM and loss modulus – LM) versus temperature during the test of all specimens during the first week (I). The modulus between the initial temperature of 30°C and the final temperature of 90°C are expressed as a percentage of the initial SM value.

Further details are expressed by the first derivatives of the temperature–modulus plots. The results are given in Fig. 3. The plots can be divided into three parts: the first one with temperatures up to about 40°C , the second one with temperatures between 40 and 60°C , and the third one with temperatures higher than 60°C . In the first part, the derivatives determined in week II are higher than the derivatives determined in week I. The derivatives decreased in the first part of the plot and the decrease was bigger in week II than in week I. A slow decrease was observed especially at LM in week I. The differences were so big that crossing of the temperature plots was observed close to 40°C . In the second part, the derivatives in week II are lower than in week I. The end of the second part was connected with new crossing of the plots so that in the third part the derivatives in week II were higher than in week I. Figure 3 also shows that standard errors expressed by bars were higher in the first and the second parts.

The basic characteristics of the plots in Fig. 3 were determined and tested. The results are given in Table 2. With exclusion of SM final, the differences between the corresponding characteristics obtained in the first and the second weeks after harvest were proved. The data showed that yacon tissue tested in week II softened mainly at temperatures between 40 and 60°C . In week I, the softening was observed mainly in its first part and then in the third part. It is known that during storage the concentration of inulin in yacon decreases (Blecker *et al.*, 2002) in dependence on storage temperature. Our experiments indicated higher values of SM and LM in the second week than in the first week after harvest (the initial value of SM at 30°C was 21.6 and 41.6 kPa in the first and in the second week after harvest, respectively). This result is in agreement with lower moisture content in specimens tested in week II. The final values of SM at 90°C were comparable in the first and the second week (1.62 and 1.54 kPa). The LM values in the second week were also higher than in the first week: in the first week the initial value of 5.31 kPa and final value of 0.51 kPa , and in the second week initial value of 7.53 kPa and final value of 0.69 kPa were observed. These results are in agreement with lower moisture content in roots stored longer prior to their test.

The changes in vibration mechanisms can be simply detected by the loss tangent (LT) that is expressed as a ratio of LM and SM (Pain, 2007). Loss tangent is plotted for both tested weeks in Fig. 4. The initial parts of the loss tangent temperature plots are very similar as in other plant parenchyma tissue (Blahovec and Lahodová, 2012b): the values decreased slightly from the initial ones between 0.15 and 0.2 , but in week II they were systematically lower than in week I. The lower initial value of LT in week II indicated lesser participation of inelasticity in the deformation process. The curves in Fig. 4 are similar to each other, with some differences between 30 and 40 , 50 and 60°C and mainly at temperatures higher than 70°C . The temperature range between 50 and 60°C corresponds to the parts of the derivative plots (Fig. 3) just above their minima. All above mentioned

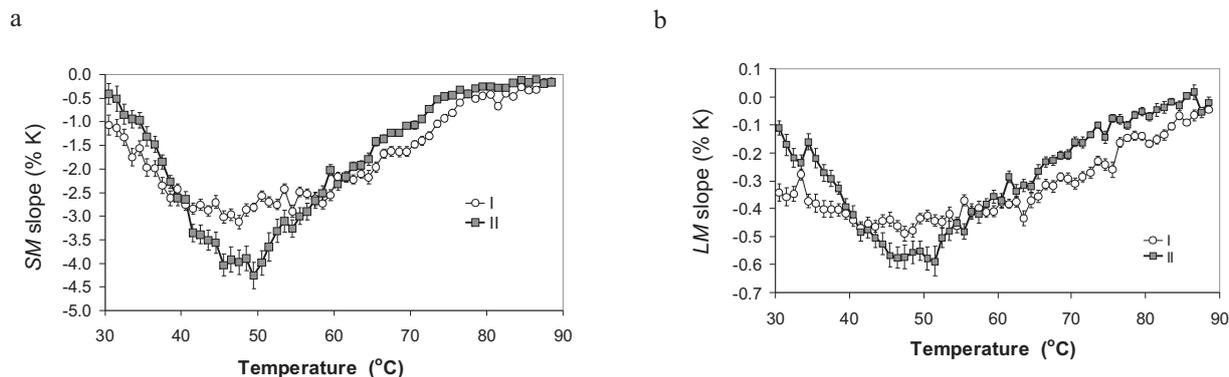


Fig. 3. Temperature derivatives of the modulus components. I and II denote the first and second week after harvest: a – data for storage modulus, b – data for loss modulus. Points denote mean values, bars are the corresponding standard errors.

Table 2. Basic characteristics of the modulus-temperature derivatives

Week	<i>SSM</i> initial	<i>SSM</i> minimal	<i>SSM</i> final	<i>SLM</i> initial	<i>SLM</i> minimal	<i>SLM</i> final
I	-1.07	-3.13	-0.15	-0.34	-0.49	-0.045
II	-0.41	-4.27	-0.17	-0.11	-0.59	-0.020

The differences in data obtained in different weeks (I and II) were proved on 95% confidence level (*SM* final is excluded).

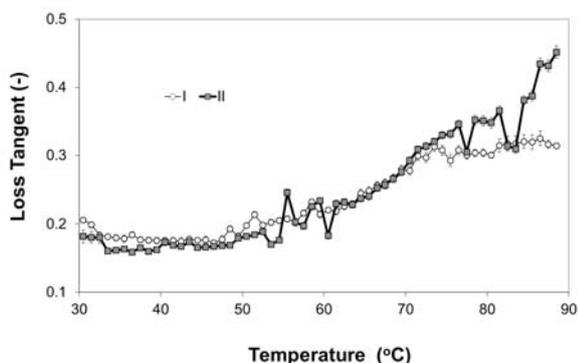


Fig. 4. Loss tangent (ratio of *LM* and *SM*) of the vibration process. I and II denote the first and second week after harvest.

parts of the *LT*-temperature plot in week II were less smooth, indicating presence of time-dependent internal processes. The details of such processes should be studied in future experiments with different rates of temperature changes. The biggest differences between the plots were observed at temperatures higher than 70°C. Nearly constant *LT* in this area for week I expressed proportionality of the changes in *SM* and *LM*, whereas increase of *LT* in week II indicated an increase of *LM* compared to *SM* *ie* the increasing role of flowing compared to elastic deformation.

Our observations detected changes in elastic module during short time storing of yacon that is more pronounced at *LM* than at *SM*. Some differences were also observed during the process of measurement where the potential changes in chemical composition can be combined with the changes in the cell membrane function (Blahovec and Lahodová, 2012b). Further experiments with chemical composition under control are needed for final explanation of the thermal processes and their changes during yacon storage.

CONCLUSIONS

1. Decrease of moisture content and increase of aeration are characteristic for short time storage of yacon roots.
2. The changes of yacon roots characteristics are indicated by many parameters of DMA test (storage modulus, loss modulus, and temperature derivatives of these data), including of loss tangent. Time dependent changes during the test at temperatures of 60-70°C and at temperatures above 70°C were indicated.
4. The storage-dependent and test-dependent changes in the yacon tissue were not fully explained and they need further experiments where the tissue composition will be under control.

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