Analysis of Changes in Electrical and Rheological Properties of Agricultural Products in Heating Process

Keiko NAKAMURA¹ and Osato MIYAWAKI²,†

¹Faculty of Human Development and Culture, Fukushima University, Kanayagawa 1, Fukushima 960–1296, Japan
²Department of Food Science, Ishikawa Prefectural University, Suematsu 1–308, Nonoichi, Ishikawa 921–8836, Japan

Changes in electrical and rheological properties of agricultural products were investigated to clarify the softening mechanism of the organization in heating process. Potato, Japanese radish and carrot were used as the sample. These were heated in normal saline solution. Impedance was measured by LCR meter, and dynamic viscoelasticity was measured by the vibrating reed method. As a result, the impedance of potato tuber decreased gradually with an increase in temperature, then decreased rapidly at 65°C. Optical absorbance at 263 nm of the heating saline solution containing the test sample also rose drastically at this temperature, which suggested the release of intracellular nucleic acid related materials. Moreover, dynamic viscoelasticity decreased in the vicinity of this temperature, which suggested that the turgor pressure of the tissue cells was lost because of the cell membrane disruption. Similar phenomena were observed for Japanese radish and carrot although the temperature and the extent of the changes in physical properties were different depending on individuals. These results strongly suggest that the major softening mechanism of agricultural products in heating process exists in the thermal injury of cell membrane which caused the loss in turgor pressure of tissue.

Key words: impedance, viscoelasticity, cell membrane disruption, turgor pressure, thermal softening

1. Introduction

Agricultural products are heated for the starch gelatinization, softening of the organization, blanching, and sterilization, etc. The hardness of agricultural products changes in two stages during heating [1]. At the initial stage, hardness decreases rapidly by the loss of the turgor pressure of the cell [2,3]. Then the hardness decreases gradually in the second stage because of softening of the organization with β-elimination reaction of pectin.

On the other hand, the electrical measurement of agricultural products is used for quality evaluations, for example, evaluating the ripening process and confirming the mechanical injury [4]. Zhang and Willison [5] showed the electrical model of the plant cell, and clarified that the destruction of the cell membrane in thermal or freezing process were measured by the changes in the electrical resistivity [6–8].

Ohnishi et al.[9–11] measured the change in electrical and the physical properties of agricultural products in freezing–thawing process and in osmotic dehydro–freezing [12]. They reported that physical properties of agricultural products changed because the intracellular turgor pressure was lost by the cell membrane injury.

In the present paper, the electrical properties of agricultural products were measured in heating process, which was compared with rheological properties to clarify the mechanism of softening of agricultural products in heating process.

2. Materials and Methods

2.1 Test sample preparation

Potato, Japanese radish and carrot were obtained from a local market and kept in a refrigerator until use. Cylindrical samples (6.9 mm in diameter, 50 mm in length) were prepared along the long axis of agricultural products with a cork borer.

2.2 Measurement of electrical properties

Electrical properties of agricultural products were measured by a LCR meter (HP4285A, Hewlett-Packard Japan). Two platinum electrodes (0.3 mm in diameter) were fixed to the holder and inserted into the center of
cylindrical sample (5 mm deep and 7 mm in distance) as shown in Fig. 1. Cylindrical sample and the holder were fixed with a vinyl tape. One sample with the holder was immersed in a beaker containing 300 ml of normal saline (0.9% (w/w) NaCl) solution at about 20°C. The beaker was heated with heating rate at about 1°C/min. Resistance (R) and reactance (X) were measured over a frequency range from 1 kHz to 1 MHz when the normal saline solution reached the desired temperature. Impedance (Z) was calculated by the following equation.

\[ Z = \sqrt{R^2 + X^2} \]  

(1)

### 2.3 Measurement of rheological properties

Dynamic viscoelasticity of the sample was measured by the vibrating reed method [13]. Eight to ten cylindrical samples were heated in normal saline solution under the same condition as described above. When the sample heated over 80°C to 95°C, electric heater was used instead of water bath, although the heating condition of normal saline solution was arranged as the same. Sample was taken out at the specified temperature in the heating process, and immediately put in normal saline solution at 20°C to avoid overheating. Then the sample was soaked in liquid paraffin to avoid drying before the measurement. The top end of the cylindrical sample was held by a sample holder, which was vibrated at the frequency varied from 0 to 100 Hz by a vibrator (NVO2, Nack Electronics) as shown in Fig. 2. The resonance frequency and amplitude of the bottom edge of the sample as a free end were measured. Dynamic elasticity \( E' \) [N/m²] and dynamic viscosity \( \eta' \) [N·s/m²] were calculated from the following equations [13].

\[ E' = \rho \frac{4\pi l^4}{a_0 k_T f_0^2} \left[ \frac{3}{8} \left( \frac{\Delta f}{f_0} \right)^2 \right] \]  

(2)

\[ \eta' = \rho \frac{2\pi l^4}{a_0 k_T f_0^2} \left[ \frac{3}{8} \left( \frac{\Delta f}{f_0} \right)^2 \right] \]  

(3)

where, \( \rho \) [g/cm³] is the density of the sample, \( l \) [cm] is the length of the cylindrical sample excluding the part held by the sample holder, \( f \) [Hz] is the resonance frequency, \( \Delta f \) [Hz] is the width of the resonance peak at 1/(2)°.5 amplitude, \( a_0 = 1.875 \), and \( k_T = D/16 \) (D [cm] is the diameter of the sample). The measurements were repeated three or four times for different samples and the mean values were obtained as the dynamic viscoelasticity.

### 2.4 Measurement of optical absorbance

About 40g of the sample was cut into 1mm thickness and immersed in a beaker containing 1 liter of normal saline (0.9% (w/w) NaCl) solution. The beaker was heated in a water bath under the same condition as described above. Three milliliter of the saline solution containing test sample was taken out at the specified temperature in the heating process and poured into the test tube. The test tube was immediately cooled in ice water to avoid overheating. The optical absorbance was measured with a spectrophotometer (DU–65, Beckman). The wavelength employed for the measurement was 263, 260, and 255 nm for potato, Japanese radish, and carrot, respectively. These wavelengths showed the biggest absorption in each sample.

---

**Fig. 1** Experimental apparatus for measurement of impedance.

**Fig. 2** Experimental apparatus for measurement of rheological properties.
3. Results and Discussion

3.1 Change in impedance of potato in heating process

Figure 3 shows the frequency dependence of the impedance of potato tuber samples in heating process. The impedance in a low-frequency range was large at 25°C, and it decreased with an increase in frequency showing a relaxation phenomenon. This phenomenon was also observed at 40, 50 and 60°C, although the impedance in the low-frequency range decreased with the rise in temperature. At 70 and 80°C, the impedance became very small even in the low-frequency range and the relaxation with frequency was lost.

Ohnishi et al. [9–11] reported similar changes in impedance after freezing–thawing treatment for various samples of agricultural products. They reported that the formation of intracellular ice crystal in freezing process leaded a critical damage on cell membrane, which caused a drastic reduction in electrical resistance. Zhang et al. [8] also reported that cells in the tissue acted as capacitors, and the fall of the capacitance and extracellular resistance by heating must result from cellular lysis.

To confirm the state of cells in a tissue, resistance and reactance of the sample were measured at each temperature to obtain Cole–Cole plots. Figure 4 shows this plot for potato tuber at various temperatures. The characteristic Cole–Cole arcs were clearly observed at 25, 40, 50 and 60°C although the arcs became smaller with an increase in temperature. As the characteristic Cole–Cole arc corresponds to the existence of closed cell structure with an intact membrane, it indicated that the substantial heat injury didn’t occur until the sample temperature reached 60°C. On the contrary, the characteristic arc completely disappeared at 70 and 80°C. This drastic change in Cole–Cole plot suggests that the heat injury to destroy the cell structure occurred between 60 and 70°C.

The impedance change of potato tuber samples by heating is plotted against temperature to specify the temperature range of heat injury (Fig. 5). The impedance at 10 kHz is shown as a relative value to that at 25°C. The impedance decreased linearly with the temperature rise from 25 to 65°C mainly because of the increase in electric conductivity of the aqueous electrolyte solutions [14], then it decreased drastically between 65 and 70°C and became small to be almost constant above 70°C.

Zhang et al. [8] reported that the heat injury of potato tuber occurred at 43°C when the sample was heated in

![Fig. 4](image-url)  
**Fig. 4** Change in Cole–Cole plot of potato tuber in heating process. 
- •, 25°C; ○, 40°C; ▲, 50°C; △, 60°C; ■, 70°C; □, 80°C.

![Fig. 5](image-url)  
**Fig. 5** Change in relative impedance and optical absorbance of potato tuber in heating process. 
Impedance was measured at 10 kHz. 
- •, relative impedance; ○, optical absorbance of saline solution containing the sample (measured at 263nm).
water and at 55°C when the sample was wrapped and not directly contact in water. Dejmek and Miyawaki [15] also reported that a drastic change in impedance of potato tuber occurred between 60 and 65°C where sample was heated in liquid paraffin.

Figure 5 also shows the change in optical absorbance of saline solution containing potato tuber sample. The wavelength (263 nm) employed mainly corresponds to the absorption of nucleic acids. Optical absorption increased gradually from 25 to about 60°C, and increased exponentially over 64°C. This increase shows the release of intracellular nucleic acid materials into the heating solution. The drastic increase was observed at 64°C, which agrees well with the temperature of heat injury measured by impedance as shown before.

Zhang et al. [8] measured electrical impedance spectra of potato tuber under heat stress and reported that two stages might be involved in the heat injury to membranes: the functional injury leading to electrolyte leakage to extracellular space, and the structural damage leading to membrane disintegration. Belie et al. [1] measured turgor changes in red cabbage leaves and also reported that the turgor loss at the higher temperature could be due to the increased permeability of membranes, resulting in passive efflux of solutes and irreversible reduction of cell turgor.

### 3.2 Change in rheological properties and appearance of potato in heating process

Figure 6 shows the changes in dynamic elasticity ($E'$) and dynamic viscosity ($\eta'$) of potato tuber during heating. Dynamic elasticity ($E'_{25}$) was 231±49×10⁵ N/m², and dynamic viscosity ($\eta'_{25}$) was 18.0±2.7×10⁶ N·s/m² at 25°C. These values were almost equal to those of Ohnishi et al. [9] and Dejmek and Miyawaki [15] reported in their papers. Dynamic elasticity decreased a little with temperature rise from 25 to 60°C. It decreased drastically between 60 and 65°C. As discussed above, heat injury occurred at about 65°C for potato tuber sample so that the turgor pressure of tuber cells must be lost around this temperature, which caused the drastic change in elasticity. Dynamic viscosity decreased gradually with an increase in temperature up to 90°C. In this case, the gelatinization of potato starch might have counteracted the decrease in $\eta'$ by the heat cellular injury.

Ramana and Taylor [16] and Ramana et al. [17] heated various vegetable tissues and measured the change in shear modulus. They reported that the apparent shear modulus remained constant or rose slightly from 20 to 50°C, then decreased around 50 to 60°C. Dejmek and Miyawaki [15] measured the changes in storage modulus of potato tuber by heating and reported that there was a considerable decrease in the storage modulus between 60 and 65°C.

The appearance of potato tuber also changed after the heat injury occurred (Fig. 7). The heated samples were taken out at each temperature and immersed in the saline at room temperature to cool immediately. The photographs were taken about 3 hours later. The samples taken out at 25 to 60°C showed no change in appearance, but the sample at 65°C showed substantial browning. This suggests that the cell membrane was injured between 60 and 65°C, and the enzymatic browning occurred. The samples at 70 and 80°C showed an appearance being cooked, showing the inactivation of enzymes responsible for browning reaction above 70°C. Figure 7 shows the importance of blanching before freezing of agricultural products.

![Fig. 6](image_url) Changes in relative dynamic elasticity and relative dynamic viscosity of potato tuber in heating process.

- ■, relative dynamic elasticity; ○, relative dynamic viscosity.

![Fig. 7](image_url) Change in appearance of potato tuber samples left about 3 hours after heating at different temperatures.

Samples were heated up to each temperature and soon cooled to the room temperature (20°C).
3.3 Changes in electrical and rheological properties of other agricultural products in heating process

Similar measurements were carried out for other agricultural products. Figure 8 shows the changes in electrical and rheological properties of Japanese radish samples in heating process. Impedance decreased linearly with an increase in temperature and then decreased drastically at 60°C, which was a little lower as compared with the case of potato. Optical absorbance of the heating saline solution containing the sample increased gradually with an increase in temperature, then increased exponentially over 60°C, which agreed well with the change in impedance. Dynamic elasticity ($E'_{\alpha}$) of Japanese radish was $226\pm28\times10^{6}\,\text{N/m}^2$, and dynamic viscosity ($\eta'_{\alpha}$) was $13.8\pm3.3\times10^{2}\,\text{N}\cdot\text{s/m}^2$ at 25°C. These values were almost equal to those of raw potato tuber shown before in Fig. 6. Dynamic elasticity decreased gradually with an increase in temperature and then decreased drastically also at about 60°C. In this case, $\eta'$ also decreased greatly as well as $E'$ at the same temperature. As the carbohydrate content in Japanese radish is as low as 4.1% [18] so that the gelatinization of carbohydrate did not counteract the decrease in $\eta'$ in heating process in this case.

Figure 9 shows the results for the similar measurements for carrot sample. Impedance decreased linearly with an increase in temperature then decreased greatly at about 55°C. A similar drastic change in impedance above 50°C in heating process for Japanese radish was also reported in the literature [19]. Optical absorbance of the heating saline solution increased at around the same temperature. Dynamic elasticity ($E'_{\alpha}$) of carrot was $549\pm128\times10^{6}\,\text{N/m}^2$, and dynamic viscosity ($\eta'_{\alpha}$) was $22.1\pm5.6\times10^{2}\,\text{N}\cdot\text{s/m}^2$ at 25°C, both of which greatly decreased between 50 and 60°C.

Ramana and Taylor [16] and Ramana et al. [17] measured the changes in shear modulus ($G'$) during heating for four kinds of agricultural products. They reported that $G'$ of every sample decreased drastically around 60°C although the decline temperatures were different among agricultural products.

4. Conclusion

The electrical and rheological properties of agricultural products were measured in heating process along with the change in optical absorbance of heating solution containing test sample for potato, Japanese radish, and carrot. For all the samples tested, a drastic change in both electrical and rheological properties and optical absorbance of heating solution were observed at the same temperature showing that the major softening mechanism of agricultural products in heating process exists in the thermal injury of cell membrane which caused the loss in turgor pressure of tissue. For potato sample, an enzymatic browning was observed because of the difference in temperature for cell membrane disruption and that for inactivation of enzymes responsible for browning.
References


和文要約

加熱過程における農産物の電気的・レオロジー的特性の変化

中村恵子1, 宮脇長人2,†

1福島大学 人間発達文化学類, 2石川県立大学 生物資源環境学部

農産物の加熱による軟化機構を解明するために、加熱過程における電気物性および力学物性の変化を解析した。試料としてパライショ、ダイコン、およびニンジンを用い、これらを生理的食塩水中で加熱し、LCRメータを用いてインピーダンス測定を、リード共振法により動的粘弾性測定を行った。その結果、パライショ凍結において、インピーダンスは加熱温度上昇とともに漸減し、65℃において急激に減少し、同じ温度領域で、試料を浸漬した食塩水の263nmにおける吸光度が急激に增大した。このことは、この温度領域において、細胞内の核酸関連物質などが溶出したことを示唆する。さらに、同じ温度領域で、動的粘弾性も急激に減少し、細胞原形質膜破壊による膨圧の減少が推定された。これら物性の変化の程度や温度変化領域は異なるものの、同様の現象はダイコンおよびニンジンにおいても観測することができた。以上の結果は、農産物の加熱による軟化機構の主要な原因が細胞膜構造の破壊とそれに伴う細胞膨圧の喪失によるものであることを示している。