ABSTRACT: To determine the effects of high-pressure-freezing, changes in temperature, texture, and structure of konnyaku (a gel with high water content) were measured during freezing for 60 min at 0.1 to 700 MPa and -20°C. During freezing at 0.1, 100, 500, 600, and 700 MPa, exothermic peaks were detected (konnyaku froze). However, at 200 to 400 MPa, exothermic peak was not detected and temperature rose when pressure was released at -20°C; the supercooled konnyaku froze by pressure-shift-freezing. The coarse gel network observed in unfrozen konnyaku was compressed by freezing due to formation of ice crystals. The rupture stress increased and strain decreased in all frozen konnyaku. High-pressure-freezing was ineffective in improving the texture of frozen-then-thawed konnyaku.

Key Words: konnyaku, high pressure, freezing, thawing, texture, electron microscope

Introduction

Konnyaku (Konjac Glucomannan Gel) is a food gel that is produced from a plant cultivar of Amorphophallus Konjac K. koch. It has been a common dish for more than 600 years in Japan. It was brought from China to Japan as a medicine along with Buddhism in the 6th century. However, Europeans called it the Devil’s Tongue because of the somewhat ugly appearance of the tubers (Ozu and others 1992).

The konjac glucomannan is a polysaccharide, made up of D-glucose units alternating with D-mannose units in the approximate 2:3 or 1:1.6 molar ratios, which are linked by β-(1,4) bonds. The glucomannan also has branches attached to a main chain at the C-3 position through β-(1,3) bonds with length of branches as short as 2 to 3 units. The branching degree may range from 1 to 19 units. The molecular weight of glucomannan is about 100 × 10^4 ~ 120 × 10^4 (Ozu and others 1992). The glucomannan gel (konnyaku) is formed by a glucomannan (konjac mannan) sol heated with alkaline compounds (for example, sodium carbonate or calcium hydroxide). The molecules of konjac mannan, which lose their acidic moieties with the aid of alkalis, aggregate in part with one another through a linkage (for example, the hydrogen bond) and form a network structure; thus a gel is formed (Maekaji 1974).

Konnyaku is elastic and insoluble in water. Probably, the most important factor of konnyaku is its texture. However, it is very difficult to produce frozen konnyaku with good textural quality, because konnyaku is a food with high water content (approximately 97% water). In other words, its unique texture changes after freezing and thawing, and damage of the structure is extensive.

When water is frozen at atmospheric pressure (0.1 MPa), volume increases (ice I). This causes tissue damage during freezing at 0.1 MPa. However, under high pressure, several kinds of ices (ice II to IX) with different chemical structures and physical properties are formed. The densities of high pressure ices (ice II to IX), except ice I, are higher than that of water, so high pressure ices do not expand in volume during phase transition from water to ices (Fletcher 1970; Franks 1989; Hobbs 1974; Maeno 1981). In previous studies, we indicated that even in foods with a high water content, such as tofu (soybean curd) (Fuchigami and Teramoto 1996, 1997), carrot (Fuchigami and others 1996, 1997a, 1997b), Chinese cabbage (Fuchigami and others 1998a), and agar gel (Fuchigami and Teramoto 1998), the damage of texture and structure was reduced by high-pressure-freezing at 200 to 400 MPa. Furthermore, we indicated that when high-pressure-frozen tofu was thawed at the same pressure (high-pressure-thawing), tofu kept the quality (texture and structure) the same as unfrozen tofu (Fuchigami and others 1998b; Teramoto and others 1999). However, the effect of high-pressure-freezing depended on the type of food. Therefore, we investigated whether high-pressure-freezing and high-pressure-thawing were effective for improvement in quality of frozen konnyaku (a gel different from tofu). Moreover, it was assumed that foods are not frozen but supercooled during pressurization at 200 to 400 MPa and −20°C. Thus, we measured the temperature of konnyaku during high-pressure-freezing in order to examine freezing under these pressures. Although konnyaku was not frozen during pressurization at 200 MPa at −20°C (liquid phase), in this report the freezing method of 100 to 700 MPa at −20°C is referred to as high-pressure-freezing.

Analysis and evaluation of food texture can be classified into tests outside of, or within a linear viscoelastic range. Rheological tests outside the linear range, that is, “large deformation” measurement, are related to the sensory texture evaluations (Bourne 1982). However, testing within a linear range can provide important data relating to structure (Chanyongvorakul and others 1995). In this report, both large deformation and creep compliance tests were performed. Microstructures, determined by cryo-scanning electron microscope, were also compared.

Results and Discussion

Changes in temperature of konnyaku during high-pressure-freezing

Changes in pressure and temperatures of konnyaku and pressure medium during high-pressure-freezing were compared (Fig. 1). After the defined pressure was reached within 1 min, pressure was maintained for 60 min, then reduced to atmospheric pressure for approximately 20 s. The temperature of a pre-chilled thermocouple was approximately −20°C, but the temperature rose when the thermocouple
was inserted into the sample. The initial temperatures of samples were about 15 to 20 °C. However, when konnyaku was frozen in a pressure vessel at atmospheric pressure, temperature of a sample decreased to 1 °C quickly and took about 10 min to decrease from 1 °C to −5 °C. When pressurized at 100 MPa, konnyaku was supercooled to −8 °C, and it took approximately 22 min from −8 °C to −10 °C due to the release of latent heat. The onset temperature of freezing at 100 MPa was −8 °C, while final temperature of konnyaku was approximately −20 °C. When pressure was released, the temperature decreased rapidly due to heat absorbed by decompression.

When konnyaku was frozen at 200, 340, and 400 MPa then supercooled to −20 °C, an exothermic peak was not detected during pressurization. However, when depressurized, the temperature rose quickly then decreased to −20 °C. Thus, the konnyaku supercooled at −20 °C froze for 8 min when pressure was removed. In other words, the konnyaku froze through pressure-shift-freezing (Kanda and others 1992, 1993).

When konnyaku was frozen at 500, 600, and 700 MPa, konnyaku was supercooled to about −15, −6, and −3 °C, respectively, and the temperature rose quickly then decreased gradually. An exothermic peak was detected in these samples during high-pressure-freezing. When pressure was released at −20 °C, temperature decreased quickly, and the endothermic peak was detected. This indicated that the konnyaku froze during pressurization at 500 to 700 MPa.

Thus, the phase transition of water from liquid to ice during high-pressure-freezing was detectable in the konnyaku samples as changes in temperature. At 0.1, 100, 500, 600, and 700 MPa, phase transition occurred during pressurization. On the other hand, at 200 to 400 MPa, the konnyaku samples kept supercooling at −20 °C, and only when pressure was removed did the phase transition occur.

The temperature of the pressure medium rose slightly at the beginning of pressurization due to the heat that evolved from pressurization and freezing, but it decreased quickly and maintained at approximately −20 °C. No great change of temperature in the pressure medium located in the lower part of the pressure vessel was detected during pressurization.

Change in temperature of konnyaku during freezing-thawing under high pressure

Changes in temperature of konnyaku during freezing-thawing at high pressures (HPF + HPT) were compared (Fig. 2). The defined pressure was maintained below 0 °C, but pressure increased as the temperature rose from 0 to 20 °C. At 0.1, 100, 500, and 600 MPa, an exothermic phenomenon was detected during freezing, while an endothermic reaction occurred during thawing. The onset temperatures for melting the konnyaku were lower than the solid liquid equilibrium line of water experimentally determined (Fletcher 1970; Franks 1989; Hobbs 1974; Maeno 1981).

On the other hand, at 200 to 400 MPa, neither exothermic nor endothermic peaks were detected (not shown). Therefore, the phase transitions from liquid to ice and from ice to liquid did not occur during pressurization. This indicated that the konnyaku was supercooling during pressurization at 200 to 400 MPa and −20 °C.

Texture of high-pressure-frozen konnyaku by large deformation tests

Typical stress-strain curves of high-pressure-frozen konnyaku with various thawing methods were compared (Fig. 3). The averages of rupture stress and strain were also compared (Table 1). When konnyaku was frozen at 0.1 to 700 MPa and thawed at atmospheric pressure (HPF + CT, HPF + S + CT), the slope of stress-strain curves and the rupture stress of konnyaku increased significantly (p < 0.001), while the rupture strain decreased significantly (p < 0.001) compared to unfrozen konnyaku. In other words, the konnyaku lost elastic properties and became hard. Thus, the texture of frozen konnyaku differed from that of unfrozen konnyaku greatly. There were no great differences in either rupture stress or strain of konnyaku frozen at 0.1 to 400 and 700 MPa. However, when frozen at 500 MPa, the initial angle of curve was smaller, and the rupture stress was greater than other frozen konnyaku, but the decrease in rupture strain was inhibited. In comparing HPF + CT to HPF + S + CT, the rupture stress and strain of konnyaku did not change greatly during storage for 1 d in
the freezer.

As a result, when high-pressure-frozen konnyaku was thawed at atmospheric pressure, great textural changes occurred. Therefore, even a high-pressure-freezing method had no recognizable effect. This result differed from the results of tofu (Fuchigami and Teramoto 1996, 1997; Fuchigami and others 1998b; Teramoto and others 1999) and agar gel (Fuchigami and Teramoto 1998). According to the kinds of gels, the effect of high-pressure-freezing on improving the texture varied greatly.

On the other hand, when high-pressure-frozen konnyaku was thawed at the same high pressure (HPF + HPT), changes in rupture stress reduced only slightly compared to conventionally thawed konnyaku. Due to unfreezing (supercooling), the rupture stress and strain of konnyaku frozen then thawed at 200 to 400 MPa were similar to unfrozen konnyaku.

**Texture of high-pressure-frozen konnyaku by creep compliance tests**

Creep and recovery creep compliance curves for frozen konnyaku were compared (Fig. 4). When a 10 g weight was placed on konnyaku, there was an immediate deformation called “instantaneous elastic deformation.” This was followed by deformation that continuously increased over time. When the weight was removed, there was an instantaneous elastic recovery followed by further recovery. Finally, irreversible deformation was detected due to viscoelasticity.

The instantaneous creep compliance of the unfrozen control was larger than that of the frozen-thawed konnyaku (HPF + S + CT): control > 500 MPa > 700 MPa > 0.1 to 400 MPa, respectively. This implied that gels frozen at 0.1 to 400 MPa were stiffer. Furthermore, the difference in creep-compliance curves was noted in the retarded deformations. When konnyaku was frozen below 400 MPa, the compliance first increased rapidly followed by a slower increase at the same rate as unfrozen konnyaku, but creep and recovery creep compliances decreased. On the other hand, when konnyaku was frozen above 500

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**Table 1—Effect of high-pressure-freezing on rupture stress and strain of konnyaku**

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>HPF+CT</th>
<th>HPF+S+CT</th>
<th>HPF+HPT</th>
<th>HPF+CT</th>
<th>HPF+S+CT</th>
<th>HPF+HPT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>31.0 ± 3.7</td>
<td>33.4 ± 4.6</td>
<td>32.7 ± 4.5</td>
<td>82.4 ± 2.1</td>
<td>81.7 ± 2.4</td>
<td>83.4 ± 1.9</td>
</tr>
<tr>
<td>0.1</td>
<td>44.6 ± 7.1</td>
<td>48.6 ± 3.6</td>
<td>41.9 ± 3.8</td>
<td>55.6 ± 4.2</td>
<td>58.6 ± 3.2</td>
<td>55.2 ± 3.2</td>
</tr>
<tr>
<td>100</td>
<td>46.4 ± 2.3</td>
<td>49.0 ± 7.9</td>
<td>46.3 ± 5.5</td>
<td>50.7 ± 3.2</td>
<td>52.2 ± 4.7</td>
<td>56.4 ± 1.5</td>
</tr>
<tr>
<td>200</td>
<td>42.1 ± 5.3</td>
<td>46.0 ± 3.2</td>
<td>21.7 ± 4.8</td>
<td>59.5 ± 2.7</td>
<td>55.6 ± 3.7</td>
<td>80.4 ± 2.6</td>
</tr>
<tr>
<td>340</td>
<td>41.4 ± 4.1</td>
<td>58.1 ± 4.3</td>
<td>24.3 ± 5.1</td>
<td>62.5 ± 3.3</td>
<td>53.8 ± 2.0</td>
<td>82.0 ± 3.0*</td>
</tr>
<tr>
<td>400</td>
<td>41.2 ± 4.8</td>
<td>63.2 ± 8.3</td>
<td>37.7 ± 7.2</td>
<td>64.6 ± 2.2</td>
<td>56.7 ± 3.0</td>
<td>83.9 ± 2.5*</td>
</tr>
<tr>
<td>500</td>
<td>73.9 ± 11.8</td>
<td>64.3 ± 9.4</td>
<td>39.1 ± 6.6</td>
<td>78.2 ± 3.7</td>
<td>73.2 ± 5.3</td>
<td>58.7 ± 4.4</td>
</tr>
<tr>
<td>600</td>
<td>67.1 ± 3.9</td>
<td>58.5 ± 6.2</td>
<td>47.7 ± 4.4</td>
<td>68.1 ± 1.8</td>
<td>65.4 ± 4.4</td>
<td>62.1 ± 2.0</td>
</tr>
<tr>
<td>700</td>
<td>50.1 ± 4.3</td>
<td>49.1 ± 5.0</td>
<td>47.7 ± 4.4</td>
<td>57.0 ± 3.5</td>
<td>62.2 ± 5.5</td>
<td>62.1 ± 2.0</td>
</tr>
</tbody>
</table>

*HPF + CT: High-pressure-frozen for 60 min, then immediately thawed at atmospheric pressure;*

*HPF + S + CT: High-pressure-frozen for 60 min, stored for 1 d at ~30 °C, then thawed at atmospheric pressure;*

*HPF + HPT: High pressure-frozen for 60 min, then immediately thawed at high pressure for approximately 70 min.*

*No significant difference compared to control.*
High-pressure-frozen Konnyaku...

Table 2—Viscoelastic parameters of high-pressure-frozen konnyaku

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>$E_0 (\times 10^5 \text{N/m}^2)$</th>
<th>$\eta_N (\times 10^3 \text{Pa} \cdot \text{s})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>1.4 ± 0.3</td>
<td>1.3 ± 0.2</td>
</tr>
<tr>
<td>0.1</td>
<td>4.0 ± 0.2</td>
<td>3.9 ± 0.3</td>
</tr>
<tr>
<td>100</td>
<td>4.3 ± 0.5</td>
<td>3.8 ± 0.3</td>
</tr>
<tr>
<td>200</td>
<td>3.3 ± 0.7</td>
<td>3.2 ± 0.4</td>
</tr>
<tr>
<td>340</td>
<td>4.0 ± 0.3</td>
<td>3.5 ± 0.3</td>
</tr>
<tr>
<td>400</td>
<td>4.0 ± 0.1</td>
<td>3.2 ± 0.4</td>
</tr>
<tr>
<td>500</td>
<td>3.1 ± 0.2</td>
<td>2.0 ± 0.5</td>
</tr>
<tr>
<td>600</td>
<td>3.2 ± 0.2</td>
<td>2.7 ± 0.8</td>
</tr>
<tr>
<td>700</td>
<td>3.5 ± 0.3</td>
<td>3.1 ± 0.3</td>
</tr>
</tbody>
</table>

$E_0$: instantaneous elastic modulus, $\eta_N$/final Newtonian viscosity.

MPa, the 2 phases (rapid and slow increase) were not clearly distinguishable, and compliance increased almost at an equal speed from beginning to end. Therefore, compliance of konnyaku frozen at 500 MPa was lower in the beginning, but after 60 s it was higher than unfrozen konnyaku.

When konnyaku was frozen at 200 to 400 MPa then thawed at the same high pressure (HPT), the creep and recovery creep compliances were the same as unfrozen konnyaku, while the creep compliance of konnyaku frozen-thawed at 0.1, 100, 500, and 600 MPa decreased. This implied that they became stiffer. However, the pattern of curves was similar to that of unfrozen konnyaku; the compliance increased at the same rate as unfrozen konnyaku, even above 500 MPa. The changes in the recovery creep compliance of high-pressure-thawed konnyaku (HPT) were smaller compared to those conventionally thawed (CT).

The rheological behavior of viscoelastic materials can be described by several mechanical models consisting of springs (elastic property: $E$) and dashpots (viscous property: $\eta$). A 6-element model is expressed as follows: The top is a spring + Voigt model + Voigt model + a dashpot at the bottom. The creep parameters ($E_0$, $E_1$, $E_2$, $\eta_1$, $\eta_2$, $\tau_1$, and $\tau_2$) according to a 6-element model were calculated. The instantaneous elastic modulus ($E_0$) and Newtonian viscosity ($\eta_N$) of frozen-thawed konnyaku were compared (Table 2). The other parameters ($E_1$, $E_2$, $\eta_1$, $\eta_2$, $\tau_1$, and $\tau_2$) according to a 6-element model were calculated. The instantaneous elastic modulus shows springiness. The springiness of konnyaku became stronger by freezing-thawing. However, $E_0$ of konnyaku frozen-thawed at 200 to 400 MPa (HPT) was the same as the $E_0$ of the unfrozen control, while $\eta_N$ was greater than that of the unfrozen control. This indicated that supercooled (not frozen) konnyaku was difficult to deform by flow. The $\eta_N$ of konnyaku frozen above 500 MPa decreased. This suggested that deformation by flow became greater.

Structure of high-pressure-frozen konnyaku

Cryo-scanning electron micrographs of konnyaku high-pressure-frozen then thawed under various conditions were compared in Fig. 5. When konnyaku was frozen at 0.1 to 700 MPa then thawed at atmospheric pressure (HPT), ice crystal traces (pores) were observed (low magnification). The size of ice crystals varied with a location of specimen (1 mm$^2$) for cryo-SEM. There was no notable size difference of ice crystals in konnyaku frozen at 0.1 to 700 MPa. It is difficult to produce homogeneous konnyaku gel even for a maker. It appears that not only freezing-thawing methods but also the presence of air in konnyaku and coarse or fine structures of gel network affected the size of ice crystals. However, the size of ice crystals in the frozen konnyaku was considerably smaller than that of frozen tofu (Fuchigami and Teramoto 1996, 1997;

![Fig. 3—Stress-strain curves of high-pressure-frozen konnyaku. HPT + CT: High-pressure-frozen for 60 min, then immediately thawed at atmospheric pressure; HPT + S + CT: High-pressure-frozen for 60 min, stored for 1 d at -30 °C, then thawed at atmospheric pressure; HPT + HPT: High-pressure-frozen for 60 min, then immediately thawed at high pressure for approximately 70 min. Control: unfrozen.](image-url)
Fig. 5—Cryo-scanning electron micrographs of high-pressure-frozen konnyaku. HPF + CT: High-pressure-frozen for 60 min, then immediately thawed at atmospheric pressure; HPF + S + CT: High-pressure-frozen for 60 min, stored for 1 d at -30 °C, then thawed at atmospheric pressure; HPF + HPT: High-pressure-frozen for 60 min, then immediately thawed at high pressure for approximately 70 min. Control: unfrozen.
Fuchigami and others 1998b; Teramoto and others 1999) and frozen agar gel (Fuchigami and Teramoto 1998).

A gel network was observed in unfrozen konnyaku (high magnification). With pressurization at 20 °C, there was no notable change in the minute structure of the gel network; therefore, micrographs are not shown. However, a gel network could not be observed in the any of frozen konnyaku (HPF + CT, HPF + S + CT). This occurred because the gel network was compressed due to the formation of ice crystals. The concentration of solute containing a coagulating agent (calcium hydroxide) from the formation of ice crystals might have affected the dense gel network. Perhaps this is one cause pertaining to the firm condition of frozen konnyaku. Consequently, high-pressure-freezing was ineffective when konnyaku was thawn at atmospheric pressure. This result differed from that of high-pressure-frozen tofu, which maintained a comparatively coarse network.

The structure of konnyaku frozen-then-thawed at 0.1, 100, 500, or 600 MPa (HPF + HPT) changed greatly; ice pores and dense gel network were observed. There was no notable difference between HPT and CT. Conversely, when konnyaku was frozen-then-thawed at 200 to 400 MPa (HPF + HPT), no ice crystals were observed. This konnyaku had the same gel network as the unfrozen konnyaku because they were not frozen during pressurization.

Conclusions

The texture and structure of konnyaku frozen at 0.1 to 700 MPa then thawed at atmospheric pressure changed greatly. High-pressure-freezing was ineffective in improving the texture of frozen konnyaku.

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**Materials and Methods**

**Sample preparation and method of high-pressure-freezing**

Sashimi konnyaku that was produced from konjac flour and calcium hydroxide (Soryo Konnyaku Co. Ltd., Hiroshima-ken, Japan) was used. Konjac flour was added to water, stirred slowly for 5 min, and allowed to stand 60 to 90 min at room temperature. Calcium hydroxide solution was added, and the mixture (Table 3) was stirred vigorously for 1 min then poured into forms. The forms were heated for 30 min in 80 °C water. Konnyaku was decorticated with a borer of 18 mm in dia to produce cylinders and cut into disks 10 mm thick using an ultrasonicator. The dia of disks shortened approximately 15 mm due to shrinkage like rubber. The 8 pieces of konnyaku were vacuum-packed in heat-sealed polyethylene bags. High hydrostatic pressure treatments were carried out using a high pressure food processor Dr. Chef (Kobe Steel Ltd., Kobe, Japan) as described by Kato and others (1997), Fuchigami and others (1997a), and Fuchigami and Teramoto (1997). Propylene glycol (pressure medium) in a pressure vessel (6 cm inside dia and 20 cm high, surrounded with a jack), previously kept at approximately −20 °C by circulation-type cooler (−35 °C to 10 °C), was removed. The samples were placed into a pressure vessel, a thermocouple was inserted in the middle part of the sample, then the pressure medium (−20 °C) was poured into a pressure vessel. Samples were immediately pressurized at 100 to 700 MPa. The operation was fully automated, and both pressure and temperatures of the sample (the upper part of the pressure vessel) and pressure medium (the lower part) were recorded.

**Methods of thawing**

After high-pressure-freezing for 60 min, samples were thawed as follows (Fuchigami and Teramoto 1998): Pressure was released, and konnyaku was thawed immediately at 20 °C for 45 min in a low temperature incubator (HPF + CT: high-pressure-freezing + conventional thawing); pressure was released, and konnyaku was immediately stored for 1 d in a freezer of −30 °C (designated as S) and then thawed at 20 °C (HPF + S + CT); pressure vessel was heated by circulation-type heater (60 °C) for about 70 min at the same pressure as high-pressure-freezing (HPF + HPT). When the temperature of samples reached 20 °C, pressure was released (designated as high-pressure-thawing). These gels were compared with unfrozen original konnyaku (control) and konnyaku frozen in a pressure medium at −20 °C for 60 min at atmospheric pressure (0.1 MPa) then thawed at 20 °C.

**Large deformation tests**

Large deformation tests were performed using a creepmeter (Rheoner, RE-33005, Yamaden Co. Ltd., Tokyo, Japan) by the method reported in a previous paper (Fuchigami and Teramoto 1997). Thickness of samples was measured using a sample-height counter (HC-3305, Yamaden, Tokyo, Japan) then punctured by using a plunger (cylindrical shape: 5 mm dia, 22 mm long) at 0.5 mm/s stopping at 99% the thickness using a load cell of 2 kg. Because the surface area of the specimen was changed from freezing-thawing, a plunger about 1/3 the dia of the specimen was used for a puncture test. Several different speeds were used (0.5 mm/s, 1 mm/s, 5 mm/s), but best results could be obtained at 0.5

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**Table 3—Konnyaku mixture**

<table>
<thead>
<tr>
<th>Konnyaku mixture</th>
<th>HPF+HPT</th>
</tr>
</thead>
<tbody>
<tr>
<td>270 g konjac flour</td>
<td>Control</td>
</tr>
<tr>
<td>10 L water</td>
<td>0.1 MPa</td>
</tr>
<tr>
<td>1 L 1.8% calcium hydroxide solution</td>
<td>200 MPa</td>
</tr>
<tr>
<td></td>
<td>500 MPa</td>
</tr>
<tr>
<td></td>
<td>600 MPa</td>
</tr>
<tr>
<td></td>
<td>700 MPa</td>
</tr>
</tbody>
</table>

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**Fig. 4—Creep-compliance curves of high-pressure-frozen konnyaku. HPF + S + CT: High-pressure-frozen for 60 min, stored for 1 d at -30 °C, then thawed at atmospheric pressure; HPF + HPT: High-pressure-frozen for 60 min, then immediately thawed at high pressure for approximately 70 min. Control: unfrozen.
mm/s. Both rupture stress and strain were indicated.

**Creep compliance tests**

Creep behavior was analyzed using a creepmeter (Rheoner RE-33005, Yamaden Co. Ltd., Tokyo, Japan). Konnyaku was compressed by a plunger (5 mm dia) at a cross-head speed of 1.0 mm/s, and a constant force of 10 g was applied to the konnyaku. Creep and recovery were measured for 1-min intervals. Results were analyzed using computer software for creep analysis (CAS-3305-16, ver. 2, Yamaden Co. Ltd., Tokyo, Japan). Creep and recovery curves are shown as compliance (strain \( \times \) stress).

**Structure measurement**

The structure of the central parts of high-pressure-frozen konnyaku was observed with a cryo-scanning electron microscope (S-4500, Hitachi Co. Ltd., Tokyo, Japan). The magnifications used to observe ice crystals and gel networks were \( \times 1000 \) and \( \times 30,000 \), respectively.

**References**


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