Effects of Frozen Storage and Thawing Conditions on Physical Properties of Glutinous Rice: (Part 2) Rheological Measurement

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Summary

The effects of the freezing process and frozen storage periods on rheological parameters of freshly cooked and freeze-thawed glutinous rice have been investigated. Cooked glutinous rice (cv. Koganemochi) was frozen and stored in a household freezer at -20°C for 1, 5, 10, 30 and 60 days. After steaming and natural thawing processes, creep-recovery test (0.1N, small deformation), texture profile analysis (TPA) test (50% deformation) and the tensile testing (20N, 100% deformation) were performed on one-grain and block-shaped glutinous rice respectively by a rheometer, to measure the rheological parameters of the samples. One-grain samples showed more significant differences with freshly cooked rice in tensile testing, while block-shaped ones showed more in creep recovery tests. There were few significant differences in any fracture characteristics and viscoelastic properties between steaming and natural thawing methods for both block-shaped and one-grain samples in each storage period. The TPA results demonstrated that natural thawing method would produce softer as well as less sticky glutinous rice products during 60-day frozen storage.

Keywords: Freezing, Frozen storage, Glutinous Rice, Texture, Thawing

1. Introduction

In our previous study1), we investigated the effects of frozen storage and thawing conditions on physical properties of glutinous rice, focusing on ice crystal and color measurement. The objective of this work is to investigate the effects of frozen storage and thawing conditions on the rheological parameters of freeze-thawed glutinous rice.

According to the previous research3) on how the frozen storage period affected cohesiveness, hardness and adhesiveness of rice, the freeze-thawed one-grain rice showed larger hardness than freshly cooked rice, which turned out opposite to texture properties of block-shaped rice. Moreover, it exhibited larger adhesiveness in freeze-thawed block-shaped rice after frozen storage of 30 days than freshly cooked ones, while no significant difference was found in one-grain sample. Therefore, it should be needed to take the shape factor into account while doing research on rice samples.

In addition, studies on the influence of storage at room temperature to rice and glutinous rice flour indicated an immediate decrease in texture occurred after 1 day, which was likely due to starch retrogradation at 25°C3). When compared with soaked glutinous rice samples, cooked rice was markedly adhesive because of complete gelatinization of starch that occurred during cooking4). Breakdown of amylopectin to shorter fragments also resulted in progressive increase in adhesiveness of the processed samples5). However, since most researches focused on the influence of storage to glutinous rice flour, it suggested a crucial demand to investigate the texture property change specific to glutinous rice foods with complete grain.

Except in block-shaped rice measurements6), there were various choices in the form of materials found in previous studies such as the different number of granule used in the rheological tests, including single kernels7-8), two kernels9) as well as four kernels10-11) samples, which were considered as components of rice products. Yet, there were few studies on the effect of various shapes of samples, which may cause different results in rheological tests.

2. Materials and Methods

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2.1 Sample preparation

Glutinous rice (Koganemochi, produced in Niigata Prefecture, Japan; harvested in 2015) was kept in a thermostatic room controlled at 4 °C until the experiment. A 242 g of glutinous rice was soaked within 200 ml water of tap water under room temperature (25°C) for 1.0 h. Since glutinous rice has a high ability of water absorption, it was added another 100 ml water for cooling. Soaked glutinous rice was cooked in a household rice cooker (NP-NC 10, Zojirushi Co., Japan) for 70 min. The cooking mode of rice cooker was set as for cooking glutinous rice. After cooking, every 150 g cooked glutinous rice was removed from the rice cooker to a sheet of foil which was designed for cooling and absorbing extra moisture from cooked rice. The surface of rice piled on the sheet was flattened for cooling until it reached room temperature (25°C). The samples were made into discs of which the size was 150 mm in diameter and 10 mm in height and then be put into bags designed for freezing.

2.2 Freezing and thawing

A freezer (L595×W330×H575; RRS-102CNF, REMACOM Corp., Mishima, Japan) was used in this experiment as the one specialized for home and business use, equipped with quick freezing function, which could be as low as -27°C.

The samples were sealed in Freezer Preservation Bags (Ziploc, AsahiKASEI, Tokyo, Japan) and located at a level about half the total freezer height. Then the quick freeze function was enabled for 1 h and temperature data with time were recorded. The final temperature of the samples was -23°C. Afterwards the samples were stored at -20°C for 1, 5, 10, 30 and 60 days.

Two methods of thawing were used after each period of storage: heating by a household steamer (SERIE S02, T-fal Co., Tokyo, Japan) and natural thawing at room temperature (25°C). In the steaming method, the steamer was preheated up to 100°C in advance. After the temperature of samples came close to room temperature, they were then used in rheological measurements for the following steps.

2.3 Rheological measurements

In this study, tensile testing, creep-recovery test and texture profile analysis (TPA) were conducted on freshly cooked and the freeze-thawed samples of every storage period in triplicate at room temperature (25°C). 1 N force was applied to all samples in equilibration time of 1.5 min before measurement. Thawed samples were measured on 1-grain as well as block-shaped glutinous rice samples, which were put in a cylindrical container with a height of 15 mm and a diameter of 40 mm. The block-shaped samples were 20 g in weight, 19 mm in height and 16 mm in diameter. The samples were mainly tested by two probes to check the fitness: cylindrical one (No.3) with 16 mm in diameter and a wedge-shaped one (No.49) with 30 mm in length and 1 mm in width of the contact surfaces respectively.

2.3.1 Tensile testing

The tensile testing was performed by a rheometer (RE2-33005C, YAMADEN Corp., Tokyo, Japan). A dedicated software for the rheometer (Ver.2.3 (BAS-3305)) was employed to analyze the mechanical properties of glutinous rice samples. All operations were automatically controlled by the dedicated software (Ver.1.6 (CAS-3305)) for the rheometer.

During the tensile testing, the compression was performed up to 100% (distortion factor) in a speed of 1 mm/s, within a total time period of 19 s. Force-strain curves of glutinous rice were obtained with a load cell (200 N maximum) in 10 times the voltage magnification (20 N), where the rupture force was taken as the maximum force peak height (N) required to break the sample. Breaking strength (Pa) was calculated by dividing the rupture force by the cross-sectional area (thickness*width) of the portions. Four main stages were shown on the force-strain curve. The rupture force was calculated as the breaking strain reached 10%, which was adopted as the maximum load used in the following creep test. This is due to the initial (strain: 0~5%) and second stage (strain: 5~10%) on force-strain curve remained linear, which proved that the glutinous rice sample as a Newtonian material and could remain the linear viscoelasticity. Linear viscoelasticity is when the function is separable in both creep response and load, and is usually applicable only for small deformations. All linear viscoelastic models can be represented by a Volterra equation connecting stress and strain:

\[
\epsilon(t) = \frac{\sigma(t)}{E_{inst,creep}} + \int_0^t K(t-t')\sigma(t')dt'
\]

where t is time, σ(t) is stress, ε(t) is the strain, Einst, creep is instantaneous elastic moduli for creep and K(t) is the creep function.

The third stage (strain: 10~70%) show the structure deformation and the sample reached the breaking point. The fourth stage (strain: 70~95%) including the flexion point indicated a drastic change in compressing velocity and finally reached the maximum force point.

2.3.2 Creep-recovery test

Creep and creep recovery of cooked and freeze-thawed glutinous rice was analyzed by a 6-element model. This model was proved to be appropriate to test on glutinous rice, which contains two Kelvin-Voigt bodies (a spring in parallel with a dashpot) and a Maxwell element in series. The standalone spring describes the instantaneous elastic response, the
Kelvin-Voigt element allows for a delayed elasticity and the standalone dashpot is representative of the asymptotic behavior for \( t \to \infty \) since it models the permanent viscous strain\(^{15}\).

The model can be described as

\[
\epsilon(t) = \frac{P_0}{E_0} \left( 1 - e^{-\frac{t}{\tau K_1}} \right) + \frac{P_0}{E_2} \left( 1 - e^{-\frac{t}{\tau K_2}} \right) + \frac{P_0}{\eta N} t 
\]

(2)

\[
\tau K_1 = \frac{\eta_1}{E_1}, \quad \tau K_2 = \frac{\eta_2}{E_2} \tag{3}
\]

where \( \epsilon(t) \): the strain (%) of glutinous rice; \( t \): time after loading; \( P_0 \): constant stress; \( E_0 \): instantaneous elastic modulus (Pa); \( E_1, E_2 \): elastic modulus of the Kelvin–Voigt spring and dashpot; \( \tau K_1, \tau K_2 \): the retardation time; \( \eta_1, \eta_2 \): viscous coefficient of the Kelvin–Voigt spring and dashpot (Pa s). The retardation time \( (\tau) \) represents the time required to deform to \( (1 - 1/e) \) or 63.21% of the total deformation in the Kelvin body. The parameters above are obtained from fitting the experimental data to Eq (2) and Eq.(3).

Creep test measured strains caused by a specific load (0.1 N), which was decided by force-strain curve explained above. This definite force was applied in a certain range of time (120 sec). The strain values were collected as a function of time.

The one-grain analysis was performed after measuring the length (7.00±0.30 mm) and thickness (3.00±0.23 mm) of each granule of glutinous rice, which was by using a Vernier caliper (Shinwa Rules Co., Sanjo, Japan).

### 2.3.3 Texture profile analysis (TPA)

TPA was performed with a maximum force (20 N) to imitate the chewing action of the teeth as the material being subjected throughout the mastication process\(^{16}\). Hardness and adhesiveness were determined using the dedicated software (Ver.2.3 (TAS-3305)) for the rheometer.

Samples of cooked and freeze-thawed glutinous rice in block shape were placed at a mounted table and the experiment was conducted a two-bite process under a cylindrical probe (16 mm in diameter). The probe was 5 mm above the sample and then declining at a rate of 1 mm/s. Then the probe was back up to the original position after penetrating the sample at the distortion factor of 50%. The movement of the probe was repeated once on compression of the partly broken glutinous rice for completing the measurements.

During the test, 50% of deformation occurred in a total time period of 53 s and no time lapse existed between the two compression processes. Force-time deformation curves were obtained with a 200 N load cell in 10 times the voltage magnification. Hardness (H) was shown as the maximum force required to compress the sample; adhesiveness was shown as the area under the abscissa after the first compression.

### 3. Results and Discussion

#### 3.1 Freezing and thawing curves

Fig.1 shows the temperature-time plot of both glutinous rice sample and ambient temperature in the freezer. There was an initial rapid decrease in environmental temperature at the start of freezing because quick thawing function was enabled. It reached -27.5°C in 1 h as the lowest point of temperature during the freezing period. The total freezing time for the cooked glutinous rice sample was about 8 h.

Temperature change in central part of glutinous rice during natural thawing and steaming thawing was shown in Fig.2. In steaming thawing, frozen glutinous rice samples were heated by the steamer over 100°C within 5 min and the samples were taken out from the steamer to be cooled up to room temperature. The sample reached room temperature in approximately 70 min, which was a little faster than cooling of freshly cooked ones. The samples thawed by natural thawing reached room temperature within 3.5 h.

![Fig. 1 Freezing curve of glutinous rice and ambient air temperature profile in the household freezer](image1)

![Fig. 2 Temperature change during steaming and natural thawing of frozen glutinous rice](image2)
3.2 Tensile testing

The fracture properties could influence perception of texture attributes at later stages of chewing process. The freshly cooked glutinous rice, which could be considered as desirable quality, had a breaking strength of 6.08×10^4 Pa and a breaking strain of 72.75% in block-shape. These were smaller than one grain samples with the breaking strength of 9.69×10^4 Pa and a breaking strain of 84.71%. The larger breaking strength in one grain sample was due to the small contact area with the probe, which caused a larger distortion on it.

Both for the blocked-shaped and one grain samples, it showed no significant differences in any fracture characteristics between the steaming and natural thawing method during each storage period, which suggested to different external thawing conditions, frozen glutinous rice within 60-day thawing remained a stability to the imposed deformation.

Fig. 3 showed the fracture characteristics of block-shaped glutinous rice, which demonstrated that nearly all of the fracture characteristics of each period were higher than freshly cooked ones, except 5-day period samples. However, there were few significant differences found between freshly cooked glutinous rice and freeze-thawed ones in all storage periods, which meant when frozen glutinous rice was in an aggregation during a 60-day frozen storage, the seemingly larger effect of imposed deformation between the freshly cooked one could be neglected. Meanwhile, it could still be noticed that in each period, naturally thawed samples generally owned a larger breaking strain than steamed ones, which indicated an undetectable hardness that natural thawing brought about to block-shaped glutinous rice.

The fracture characteristics of one-grain glutinous rice were presented in Fig. 4. Unlike block-shaped samples, when compared to freshly cooked glutinous rice, one grain samples showed a significant (p < 0.05) decrease in breaking strength and energy from 5-day storage period, which might be due to unstable moisture content in the structure of one grain samples, and one grain samples were considered more vulnerable when subjected to deformation and lower resistance to swelling than aggregated ones. Then a significant (p < 0.05) increase of these two characteristics was found in 60-day period samples, which could be attributed to moisture loss that accelerate the deterioration occurred in texture, and led to a tough property as well as a decline in quality. In addition, breaking strain of one-grain samples showed higher values in steaming method, which was contrary to block-shaped ones. This suggested the breaking point of glutinous rice could be affected by thawing methods as well as the shape factor, even with a 100% distortion factor.

3.3 Creep-recovery test

In each storage period, there were few distinct differences between steaming and natural thawing methods in any viscoelastic property found in whether block-shaped or one-grain samples. This was consistent with the results in tensile testing, which suggested stable viscoelastic characteristics of glutinous rice to heat-moisture treatments in the frozen storage periods within 60 days.

The viscoelastic properties of block-shaped glutinous rice during frozen storage were presented in Fig.5. When compared to freshly cooked rice in block shape (E0: 3.05×10^4 Pa, E1: 5.17×10^4 Pa, E2: 7.16×10^4 Pa, η1: 4.89×10^5 Pa s, η2: 1.17×10^5 Pa s, ηN: 8.28×10^6 Pa s), almost all of the samples thawed by the two methods showed a decrease (p < 0.05) in elasticity and viscosity parameters, while steaming method tended to show a more significant difference than natural thawing method, which could be due to the collapse of waxy rice granules during heating.

Among them, the samples of 1-day period represented significant (p < 0.05) decrease in most of the viscoelastic properties. This indicated that freeze-thawed glutinous rice in block shape became softer and less chewy than freshly cooked ones, and the initial stage of freezing was affected in particular.

Fig. 6 showed a part of viscoelastic properties of one-grain glutinous rice during the frozen storage. Unlike block-shaped glutinous rice, when compared with freshly cooked glutinous rice (E0: 6.71×10^4 Pa, E1: 1.54×10^5 Pa, E2: 1.81×10^5 Pa, η1: 1.35×10^6 Pa s, η2: 1.96×10^5 Pa s, ηN: 1.19×10^6 Pa s), freeze-thawed one-grain samples showed larger (p < 0.05) values in most of viscoelastic properties by the natural thawing method, especially during 1~10-day storage period. This was in accordance with the previous study2), where one grain samples of rice presented more vulnerability than freshly cooked ones, which was opposite to block-shaped ones within 30-day frozen storage period. This could be attributed to the quick loss of moisture content in one-grain glutinous rice of initial frozen stages, for being in relatively external surface of the samples, and accelerated hardening.

3.4 Texture profile analysis (TPA)

Fig. 7 presented hardness and adhesiveness of the samples after two thawing methods during frozen storage period. First, in each period of storage, it was found that hardness of steamed samples showed significantly (p < 0.05) lower values than natural thawed ones during 1 and 5-day period. However, in 10, 30, and 60-day storage periods, no significant differences could be found between the two thawing methods in hardness and adhesiveness. Besides, when it comes to adhesiveness, there were no significant differences occurred between the two thawing methods during all 5 storage periods. It indicated that the texture change due to natural thawing might produce a larger hardening effect to short-time frozen preservation period (1 to 5 days) of glutinous rice. This could be interpreted that the crystalline structure
in starch became more readily disrupted in high moisture environment by steaming\textsuperscript{17), combined with the dehydration occurred during natural thawing. When compared with freshly cooked rice (hardness: 3.12×10\(^4\) Pa, adhesiveness: 354.99 J/m\(^3\)), it suggested significant loss in hardness and an increase in adhesiveness (p<0.05) of freeze-thawed rice during 1 and 5-day periods (especially by steaming method), which was consistent with the results in creep-recovery tests. This indicated that freeze-thawed glutinous rice became softer and less sticky than freshly cooked ones within 5-day storage, probably due to the influence of heat-moisture treatment.

4. Conclusion

It was found that one-grain samples showed more significant differences with freshly cooked rice in tensile testing while block-shaped ones showed more in creep recovery tests.
From tensile testing of block-shaped glutinous rice, it suggests that the growth of size rather than the number of ice crystals is the crucial factor in the increase of fracture characteristics of glutinous rice during the frozen storage. On the other hand, the decrease in fracture characteristics of one-grain samples during the storage, as well as the increase in 60-day period, is more affected by other factors like moisture content instead of microstructure changes or thawing conditions.

As to viscoelasticity changes in block-shaped glutinous rice, it indicates that a relatively short-time frozen storage period influences them most, makes glutinous rice softer and less chewy than freshly cooked one, especially when steamed. Furthermore, the changes during the storage period mostly attribute to the number of ice crystals and thawing conditions, where proper steaming speed could produce a chewy quality even after long time storage, while longer natural thawing time would make it softer.

Fig. 5 Viscoelastic properties of block-shaped glutinous rice
Fig. 6 Viscoelastic properties of one-grain glutinous rice

Fig. 7 Hardness and adhesiveness of glutinous rice
On the other hand, one-grain glutinous rice becomes harder than freshly cooked one after natural thawing due to moisture loss, especially in the initial frozen stage. Its viscoelasticity is influenced greatly by microstructure changes, thus will become less sticky with storage time, except for a short-time storage period.  

The TPA results demonstrated that natural thawing method will produce softer as well as less sticky glutinous rice products during 60-day frozen storage, while the steaming method might possess a recovering ability of the quality reduction, especially in short-time frozen preservation period.

References


