Gelation Mechanism and Rheological Properties of Rice Starch

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Cereal Chem. 72(4):393–400

ABSTRACT

Three rice starches were isolated from indica (KSS7), japonica (TNu67) and waxy (TCW70) varieties, respectively. Amylose contents of isolated starches were 25.6% for KSS7; 14.80% for TNu67; and 0.99% for TCW70. When heated from 55 to 95°C at 10°C intervals, starch with higher amylopectin content had higher swelling power. Both swelling power and water solubility index increased with increased temperature. The blue values of all samples were <0.08. As examined by scanning electron microscope and x-ray diffractometer, the typical A-type diffraction pattern of the peak became slightly flattened at the temperature below the gelatinization temperature (GT), and no peak was detected at the temperature higher than the GT. The change of starch during heating or cooling was also analyzed by mechanical spectrometry with an oscillatory rheometer. G' and G'' were dependent on amylose content and starch concentration.

During gelatinization, starch granules swell irreversibly and can be ruptured, releasing amylose (Nikuni 1978, Imberty et al 1991). The leached-out amylose forms a three-dimensional network with the swollen granule embedded in the matrix (Hennig et al 1976, Richardson et al 1981, Wong and Lelievre 1981, Ring and Stainsby 1982, Eliasson 1985, Ring 1985, Tester and Morrison 1990). Consequently, the gel is developed when it cools. The paste form will appear when no network is developed. The formation of a gel or a paste is one of the principal factors that control the texture and quality of starch-containing food. The structure of gel or paste is determined by the starch concentration and the structure of the swollen starch granule; the amounts and the types of amylose and amylopectin leached out from the granule; the interaction among amylose, amylopectin, and the granule; and heating conditions such as temperature, heating period, and rate, etc. Previous studies have reported the relationship of starch gelatinization and gelation with gel aging using Brabender amylography, x-ray diffractometry, β-amylase-pullulanase (BAP) methodology, and differential scanning calorimetry (Yang et al 1987, Chang and Chang 1988, Chang and Liu 1991). To further understand the transition mechanisms of gelatinization and gelation of rice starch, transitions of rice starches with various amylose contents in different concentrations and different heat treatments were measured. A rheometer with small amplitude was applied for more complete information about the viscoelastic property of the sample. The mechanisms of gelatinization and gelation could be illustrated from the changes of rheological properties combined with other physical and chemical analyses.

MATERIALS AND METHODS

Rice Starches

Three kinds of rice starches, indica (Kaoshiung Sen 7, KSS7), japonica (Tainung 67, TNu67), and waxy (Taichung waxy 70, TCW70) varieties were isolated by the modified alkaline steeping method (Yang et al 1984). The amylose content of the isolated starch was determined by the modified method as described by Lii et al (1986).

Calorimetry

The gelatinization of starch was examined by differential scanning calorimetry (DSC) (1090B DSC, Du Pont, Wilmington, DE). The starch and water slurry (1:4) was applied as the sample. Slurry samples at 8–10 mg were hermetically sealed in a coated aluminum pan (Du Pont) at room temperature for 1 hr. An empty pan was used as the reference. The sample was heated from 25 to 120°C at a rate of 10°C/min. The temperatures of the characteristic transitions, onset (T_o), peak (T_p) and completion (T_c) were recorded. The heat enthalpy (ΔH) of the transition was expressed as cal/g of dry starch.

Swelling Power, Water Solubility Index, λmax, and Blue Value

Starch suspension (1%, w/v) was heated to 55, 65, 75, 85, and 95°C, respectively, and was kept at that temperature for 30 min, followed by cooling down to the room temperature rapidly in an ice water bath. The cooled sample was centrifuged at 5,000 × *g* for 20 min. The swelling power was measured from the precipitate (Leach et al 1959). The water solubility index (WSI) was measured from the supernatant (Holm et al 1985). The blue value and the maximum absorbance wavelength (λmax) of the soluble fraction (supernatant) were examined with the method of Gilbert and Spragg (1964).

X-ray Diffraction Pattern and Scanning Electron Microscopy

The precipitate was collected with the method described for the measurement of swelling power, followed by freeze-drying and grinding to a powder as the sample for the x-ray diffractometry and scanning electron microscopy (SEM). An x-ray diffractometer (D/MAX-IIIA, Rigaku Denki Co. Ltd., Japan) with the method of Zobel (1964) and a scanning electron microscope (JSM-5400, Jeol Co., Japan) were applied in the investigation.

Rheological Properties

The small amplitude oscillatory rheological measurement was made with a rheometer (Carri-Med-CSL 50, TA Instruments Ltd., Surrey, England) equipped with parallel plate geometry (20-mm dia.). The gap size was 1,000 μm. The strain and frequency were set at 1.5% and 1 Hz, respectively, for all determinations, except in the frequency sweep test (0.1–20 Hz). The effect of starch concentration on dynamic rheological properties of freshly prepared gel was investigated by measurements of the storage modulus (G'), loss modulus (G''), and tanδ. The fresh gel was prepared by loading each of the 5–30% KSS7, TNu67, and TCW70 starch suspensions on the ram, and covering with a thin layer of mineral oil. The starch sample was heated from 45–95°C, then cooled from 95 to 25°C at 1°C/min during the determination.

During the investigation on the effect of heating-cooling rate, 20% KSS7 starch suspensions were heated from 45 to 95°C and cooled from 95 to 25°C at 1, 2, and 5°C/min, respectively. For the examination of the effects of the different heat treatments, 20% of KSS7 starch suspension was first heated to 70–95°C at 5°C intervals, then cooled to 25°C. The rate of heating was set at 1°C/min; the rate of cooling was set at 5°C/min. After being cooled, the samples with the 80–95°C treatments were held at 25°C for a frequency sweep test with a range of 0.1–20 Hz.

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RESULTS AND DISCUSSION

Amylose Content and DSC Characteristics

The apparent amylose contents of KSS7, TNu67, and TCW70 were 25.60, 14.80, and 0.99%, respectively (Table I). The results were similar to those of previous studies (Chang and Lee 1985, Lii and Lee 1993). DSC characteristics showed that KSS7 had higher T_o of gelatinization temperature (GT) than did TNu67 and TCW70.

TABLE I
Amylose Contents and Differential Scanning Calorimetry (DSC) Characteristics of Rice Starches

		DSC Characteristics[a]			
Starch	Amylose (%)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (cal/g)
KSS7	25.60	72.0	76.6	89.2	3.35
TNu67	14.80	64.4	71.0	82.3	2.94
TCW70	0.99	64.0	71.9	85.0	3.28

[a] T_o = onset of gelatinization; T_p = peak temperature; T_c = completion temperature; ΔH = enthalpy.

TABLE II
Some Properties of KSS7, TNu67, and TCW70 Rice Starches at Different Temperatures

Starch	Temperature (°C)	Swelling Power	WSI[a] (%)	Blue Value	λmax[b]
KSS7	55	2.31	0.70	0.0010	599
	65	2.59	0.81	0.0011	598
	75	7.26	3.27	0.0281	610
	85	16.15	13.09	0.0400	600
	95	29.44	16.87	0.0594	600
TNu67	55	2.95	0.46	0.0014	599
	65	9.18	2.14	0.0189	610
	75	10.18	2.80	0.0260	607
	85	11.94	4.12	0.0312	600
	95	27.26	18.13	0.0725	599
TCW70	55	3.87	0.93	0.0025	550
	65	20.32	3.97	0.0003	550
	75	44.37	8.55	0.0006	538
	85	47.32	11.07	0.0008	538
	95	48.49	11.61	0.0040	538

[a] Water solubility index.
[b] Maximum absorbance wavelength.

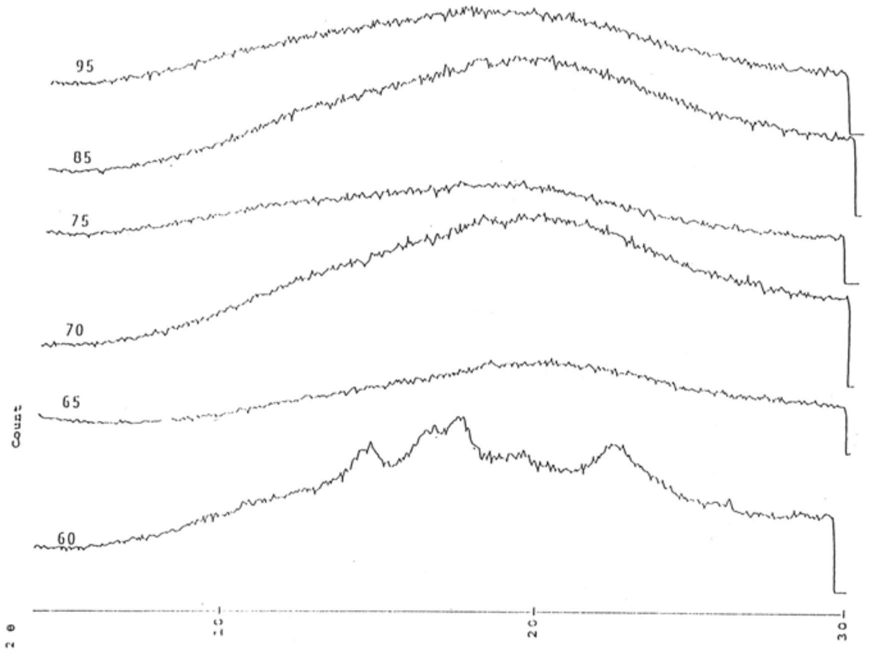

Fig. 1. X-ray diffractograms of TNu67 at different temperatures.

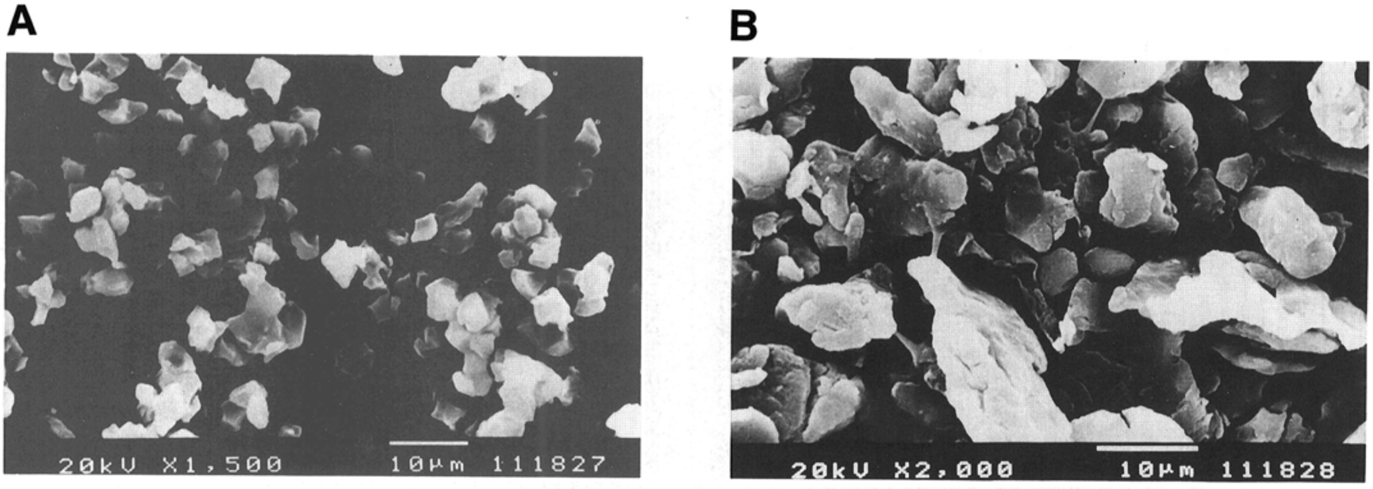

Fig. 2. Scanning electron microscope of TNu67 rice starch heated to 60 **(a)** and 65°C **(b)**.

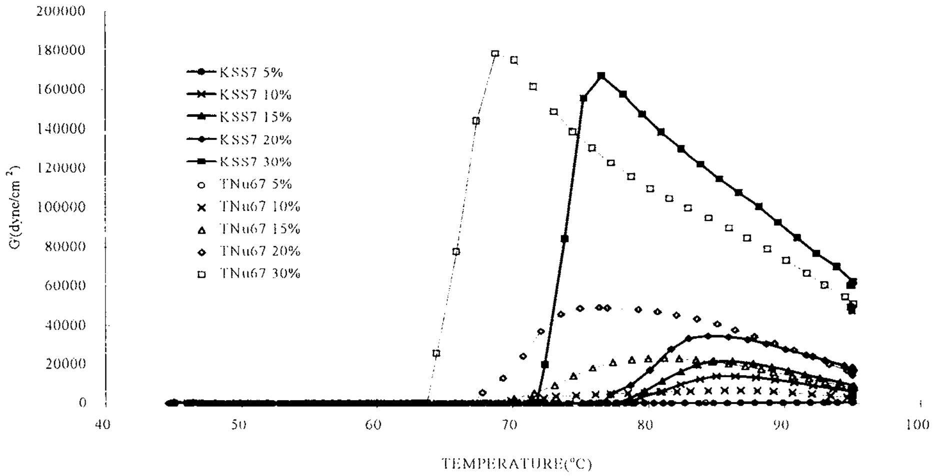

Fig. 3. Storage modulus (*G'*) measurements of KSS7 and TNu67 at different concentrations during heating.

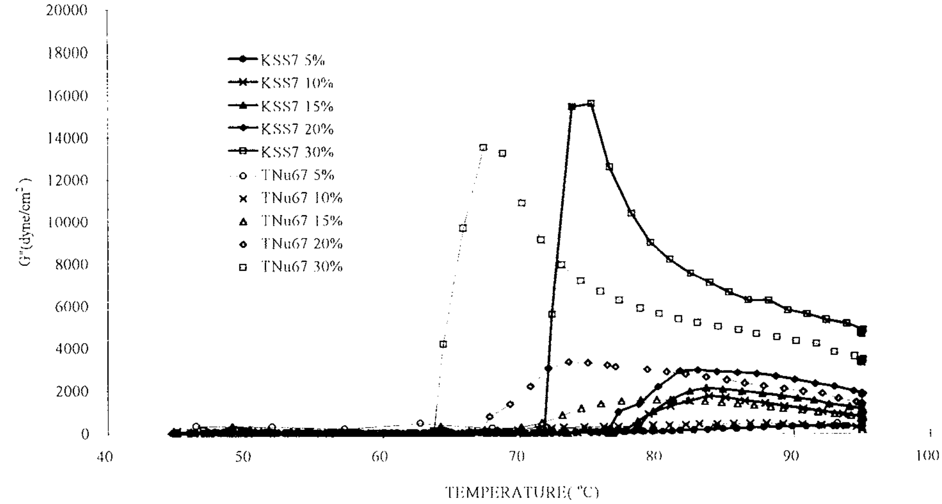

Fig. 4. Loss modulus (*G''*) measurements of KSS7 and TNu67 at different concentrations during heating.

TABLE III
Effect of Starch Concentration on Rheological Properties of Rice Starch Gel

		95°C		25°C	
Starch	**Concentration (%)**	**G'^a (dyne/cm²)**	**tanδ**	**G' (dyne/cm²)**	**tanδ**
KSS7	5	329	0.28	298	0.11
	10	5,097	0.13	3,288	0.07
	15	9,220	0.12	19,060	0.04
	20	17,320	0.11	45,010	0.03
	30	62,420	0.08	68,200	0.04
TNu67	5	479	−0.06[b]	486	−0.04[b]
	10	2,609	0.10	3,438	0.06
	15	7,573	0.09	12,380	0.05
	20	15,510	0.09	24,810	0.05
	30	48,500	0.07	45,720	0.06
TCW70	5	30	−1.87[b]	15	−0.07[b]
	10	55	0.13[b]	202	0.38
	15	46	0.50	442	0.39
	20	391	0.40	426	0.48
	30	950	0.39	1,107	0.54

[a] Storage modulus.
[b] Unstable systems.

Swelling Power, WSI, Blue Value, and λmax

The values for swelling power, WSI, blue value, and λmax of the soluble fraction for the starch sample heated from 55 to 95°C are listed in Table II. The swelling power started to rise drastically at 75°C for KSS7 and at 65°C for TNu67 and TCW70. These temperature were higher than the corresponding T_0 of GT. When the crystal region in the starch granule began to melt, it enhanced the swelling power. TCW70, with the smallest amylose content, had the highest swelling power during heating. The data were similar to those reported by Yang et al (1988). Tester and Morrison (1990) also indicated that waxy barley starch had higher swelling power than did nonwaxy barley starch, and that the fraction of amylopectin was responsible for the swelling power. Both the swelling power and the WSI of starch increased as the temperature increased.

The blue values of the soluble fractions from KSS7 and TNu67 were higher at temperatures over the GT than below the GT (Table II). The low blue value before gelatinization could be the result of insufficient leached-out material from the granule at this stage. TCW70 had the lowest blue value among the samples examined because of its trace amount of amylose content.

The λmax of the soluble fractions of KSS7 and TNu67 ranged from 598 to 610 nm; for TCW70, the range was 538–550 nm (Table II). Chang (1993) reported that complexes of the pure amylose from KSS7 and TNu67 and iodine were 650 and 653 nm, and the complexes of amylopectin from KSS7, TNu67, and waxy rice were 579, 537, and 534 nm, respectively. Such results could indicate that the short-chain amylose or intermediate fraction of the starch molecule would leach out from the granule easier than the long-chain amylose would. The λmax of TCW70 decreased from 550 to 538 nm with increased temperature. This could be a result of the leached-out intermediate fraction or short-chain amylose at low temperature, followed by amylopectin at high temperature.

X-ray Diffractogram and SEM

The change of starch granular structure during heating was examined using both x-ray diffractography and scanning electron micrography. The typical A-pattern of x-ray diffractogram of TNu67 disappeared when the temperature reached to 65°C (Fig. 1). The SEM of TNu67 displayed that the granular structure of starch would start to rupture at that temperature (Fig. 2).

Effect of Concentration on Rheological Properties

Figures 3 and 4 displayed a sudden increase in *G′* and *G″* of KSS7 and TNu67 at 5–30% concentrations that only occurred above the gelatinization temperature (T_0 = 72.0 and 64.4°C for KSS7 and TNu67, respectively) (Table I). The results were in accordance with the observations of Eliasson and Bohlin (1982) and Liu and Lelievre (1992). Below the gelatinization temperature, the starch suspension was not stable. Therefore, both *G′* and *G″* fluctuated in a relatively small range of values. Gelatinization could facilitate starch granule swelling, and more material, including amylose molecules, could leach out into water. Examination of the swelling power in the 1% starch system showed that the swollen granule (KSS7) contained more water and that its weight increased 7–29 times as the temperature increased from 75 to 95°C. The increased volume of swollen granules made a close packed system and had a positive effect on the increase of *G′* and *G″*, although in the system with the higher starch concentration, the amount of water might not be sufficient to reach maximum swelling. The higher starch concentration would assist the earlier rise of *G′* because of the shorter time to get a close packed system. Eliasson (1986) also reported that the initial increase in *G′* and *G″* was caused by the starch granules swelling progressively and finally becoming close packed.

Continuing heating could not only further swell granules and make more amylose to leach out, but could also increase the mobility and collision of swollen granules and amylose molecules. Leached-out amylose and swollen granules arranged themselves in a special three-dimensional conformation by entanglement of the molecule chains, formation of junction zones, and embedment of swollen granules. The swollen granules as a filler (Eliasson and Bohlin 1982) would also strengthen the networklike structure. These all increased the ability for the formation of networklike structure to display in the growth of *G′*. The *G″* would reach maxima at 76–86°C for 10–30% KSS7 (Fig. 3). It presented the strongest network or gel structure formed earlier at the greater starch concentration. Further heating reduced the *G′* to half or one-third of the maximal *G′* value, as observed by Eliasson and Bohlin (1982) and Eliasson (1986). More heat and greater mobility or entropy might loosen the previous structure by breaking some hydrogen bonds or other possible interactions and rupturing the structure of swollen starch granules. The strength of networklike structure weakened and *G′* dropped.

The food industry generally recognizes that a viscoelastic gel is formed by cooling the gelatinized starch dispersions of sufficient concentration (Ring 1985). Nevertheless, examination of the systems heated to 95°C shows that, in some cases, the gel could have already been developed during heating. Our studies revealed that the gel was formed at 10–30% KSS7 and the paste was formed

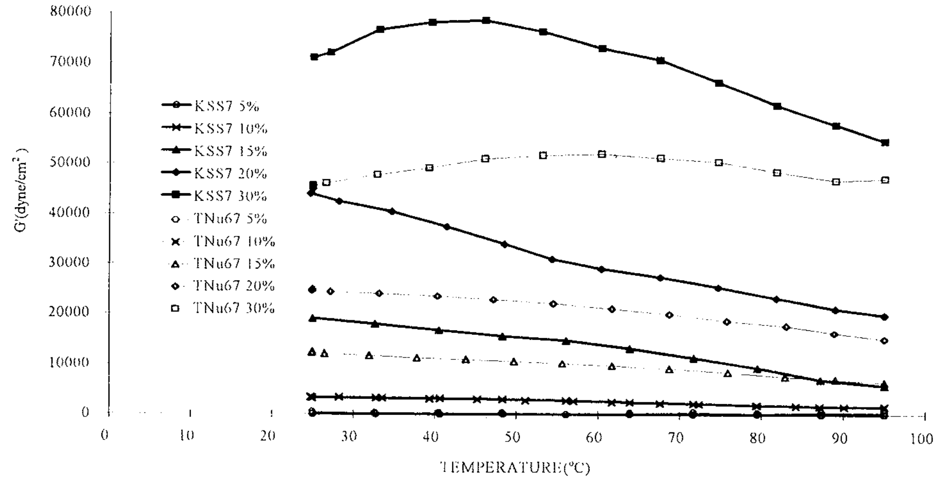

Fig. 5. Storage modulus (*G′*) measurements of KSS7 and TNu67 at different concentrations during cooling.

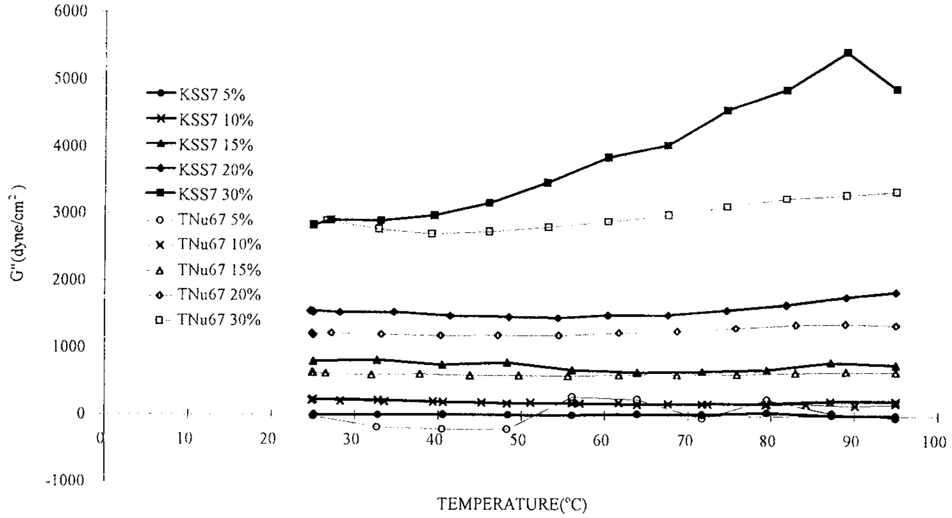

Fig. 6. Loss modulus (*G″*) measurements of KSS7 and TNu67 at different concentrations during cooling.

at 5% KSS7. The starch concentration effect was very important for producing a continuous matrix with embedded swollen granules. In the lower starch concentration (5% KSS7), the concentrations of the leached-out amylose and swollen granules were not high enough to interact themselves to form a matrix. The higher concentration (10–30% KSS7) gave sufficient interaction to enhance gel formation and display higher G' values at 95° C. Thus, both G' and $\tan\delta$ are necessary to describe the rheological properties of a gel. We, therefore, suggest that a gel has a $G' > 5{,}000$ dyne/cm^2 and a $\tan\delta < 0.1$ (Table III).

In 5% TNu67, 5% TCW70, and 10% TCW70 during heating, the starch was gelatinized but the rheological properties of the systems were still unstable with fluctuating G' and G'' (Table III). The pastes were developed in the 10% TNu67, and 10–30% TCW70 systems. Because amylose was required to form a continuous matrix to embed swollen granules in gel forming (Ring and Stainsby 1982, Ring 1985), TCW70 in this study, with only a trace amount of amylose, could not develop a gel matrix.

TNu67 with high concentrations (15–30%) and KSS7 (10–30%) could form a networklike structure. As discussed earlier, the swelling power, WSI, blue values, and λmax of TNu67 and KSS7 were similar at 95° C (Table I). The difference between KSS7 and TNu67 in amylose content inside the swollen granules might lead to the different gel-paste behavior (a gel in 10% KSS7 and a paste in 10% TNu67), as well as a higher G' in the KSS7 gel than in the TNu67 gel at the same concentration levels (15–30%). Because KSS7 was reported to have longer average chain length of amylopectin ($\overline{\mathrm{CL}} = 22$) than TNu67 ($\overline{\mathrm{CL}} = 17$) (Chen 1994), and higher blue value than TNu67 (Chang 1993), it is possible that more amylopectin with longer chain length might also somehow enhance the gel-forming process.

The G'' is an indicator of viscosity of the system (Hamann et al 1990, Kokini 1994). It had changes similar to those of G' during heating (Fig. 4). The concentrations of both amylose and swollen granules would increase the G'' value, but their greater mobility would decrease it.

During cooling, the G' steadily grew (Fig. 5, Table III). KSS7 ($\geq$15%) had a higher rate of increase in G' than did TNu67 ($\geq$15%) at the same levels of starch concentration during cooling. It was not clear why the G' in the 30% TNu67 and 30% KSS7 increased as the temperature cooled from 95° C to ~50° C, then decreased during further cooling. Further studies on higher starch concentrations (>30%) will be conducted in the future. In Figure 6, the G'' showed no obvious change for 5–20% KSS7 and 10–20% TNu67, but it declined for 30% KSS7 and TNu67, and fluctuated at 5% TNu67 during cooling. Higher G' during cooling might be explained by the interactions including hydrogen-bond formation favored at lower temperatures (exothermic). Cooling was a process of starch retrogradation, mainly due to amylose retrogradation during the short period (Biliaderis and Zawistowski 1990). The cooled gel was more rigid than the hot gel (Biliaderis 1992).

Effect of Concentration on G'

The dependence of the paste-gel system G' at 25° C on starch concentration (5–30%) followed the power law relationships: G' varies as $C^{3.2}$ ($r^2 = 0.97$) for KSS7, $C^{2.6}$ ($r^2 = 0.99$) for TNu67 and $C^{2.3}$ ($r^2 = 0.92$). The results were in accordance with the previous studies in rice and garbanzo bean starches with $C^{2.1-2.9}$ (Biliaderis and Tonogai 1991) and in rice starch with $C^{2.2-2.9}$

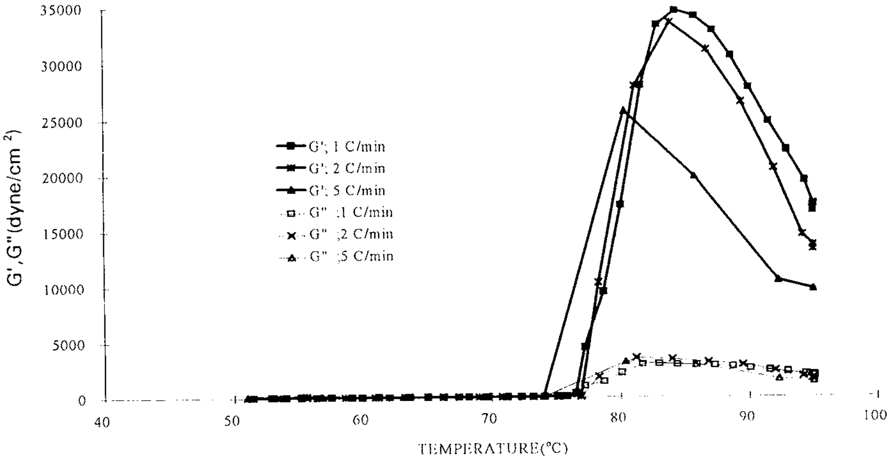

Fig. 7. Storage modulus (G') and loss modulus (G'') measurements of 20% KSS7 during heating at different heating rate.

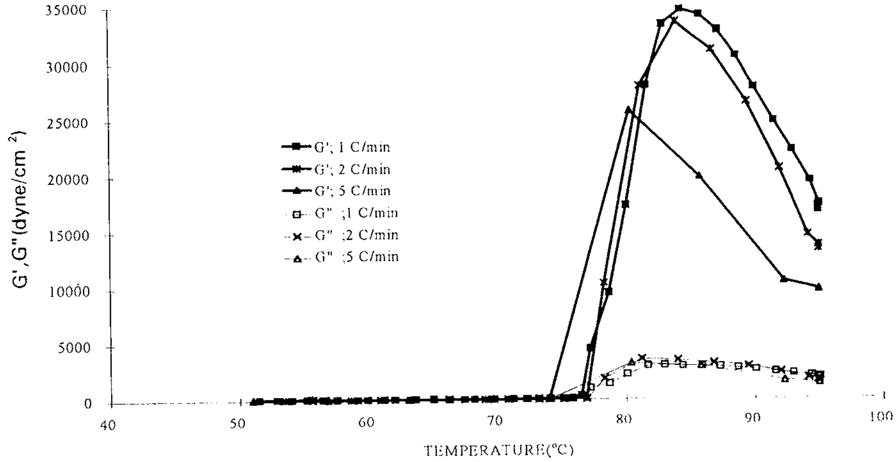

Fig. 8. Storage modulus (G') and loss modulus (G'') measurements of 20% KSS7 during cooling at different heating rate.

(Biliaderis and Juliano 1993). The linear relationships between G' and concentration have been reported for 4–8% maize starch (Evans and Haisman 1979) and for 6–30% potato, wheat, and maize starches (Ring 1985). Our rice systems (5–30% starch) could also show the linear relationships with $r^2 > 0.94$–0.98.

Effect of Heating-Cooling Rate on Rheological Properties

Figures 7 and 8 for the 20% KSS7 starch suspensions heated from 45 to 95°C and cooled from 95 to 45°C at 1, 2, and 5°C/min, respectively, demonstrated similar rheological patterns in G' and G''. At the highest heating rate (5°C/min), the rise of G' seemed to occur earlier and the maximum G' seemed to occur later than the other two. This was the result of less time to get more data to characterize the curve. However, the heating rate had some influence on G' at 95°C (Table IV). The lowest heating rate would give the system enough time to enhance the interaction among amylose and swollen starch granules, therefore, it made the most rigid or viscoelastic gel with the highest G' at 95°C. The increase rates of G' at different cooling rates were very similar (Fig. 8). Cooled to 25°C, all the G' values were ~40,000 dyne/cm^2, and the $\tan\delta = 0.03$ (Table IV). These indicated that the historical heating-cooling rate had no significant effect on the rheological properties of the cooled gels.

Effect of Heating Treatment on Rheological Properties

The effect of heating treatment on starch gel formation was investigated by heating 20% KSS7 to 70, 75, 80, 85, 90, and 95°C, and then cooling to 25°C. Table V shows that the system at 70°C remained unchanged as a suspension with fluctuating G'. The concentrations of the leached-out amylose and large swollen granules were low, so there were few effective collisions and the interaction among particles was weak. For the system at 75°C, the blue value and swelling power raised to high levels (Table II); however, both G' and G'' were still low (G'' were not shown in Table V). Holding the system at 75°C for 10 min resulted in G' increasing 74–51,710 dyne/cm^2, indicating that both time and temperature were required for particle collision and interaction in forming a network. During heating, the systems at 80 and 85°C had the strongest gel structure with highest G' (≥40,000 dyne/cm^2) and lowest $\tan\delta$ (~0.08). At this time, the amylose chain would entangle, the microcrystalline would build the junction zone (Miles et al 1985a,b), and the swollen granules would embed in a continuous matrix. These factors combined to form the strongest gel structure. By continuing heating to 95°C, some interaction or bonding in a gel dissociated or loosened through greater mobility and energy, so that G' declined to a relatively low value (17,300 dyne/cm^2). The G' of the starch system held at 95°C for 1 hr declined to 13,336 dyne/cm^2, which also proved that the energy destroyed some interactions in the system (Table V). Nonetheless, it still retained the gel properties.

During cooling, the G' steadily grew in the gel systems (80–95°C), was unchanged at the 75°C treatment, and oscillated at the 70°C treatment (Fig. 9). One explanation is that starch granules should be gelatinized at least partially to achieve a stable starch system (a paste at 75°C or gels at 80–95°C). Table V showed that G' and $\tan\delta$ for the cooled gel (25°C) in the systems at 80–95°C were in the same levels, respectively. For gel preparation, it was sufficient to heat a 20% KSS7 starch suspension to 80°C.

Frequency Sweep Test

The G' and G'' of KSS7 gels from different heating treatment (80–95°C) were essentially independent of frequency from 0.1 to 20 Hz at 25°C (Figs. 10 and 11). This implied that testing the starch gels at 1.5% strain were within the linear viscoelastic region for the systems (Biliaderis and Zawistowski 1990). G' always predominated over G'' at all frequencies tested. This was the typical pattern of the true gel (Biliaderis and Tonogai 1991, Biliaderis and Juliano 1993, Kokini 1994). Kokini (1994) had clarified that intramolecular stretching and distorting were primarily conforma-

TABLE IV
Effect of Heating-Cooling Rate on Rheological Properties of Gel at 95 and 25°C

	95°C		25°C	
Heating-Cooling Rate (°C/min)	**G'^a (dyne/cm^2)**	**tanδ**	**G' (dyne/cm^2)**	**tanδ**
1	17,230	0.11	45,010	0.03
2	13,660	0.12	45,640	0.03
5	9,697	0.14	38,350	0.03

[a] Storage modulus.

TABLE V
Effect of Heating Treatment on Rheological Properties of 20% KSS7 Starch Systems

	Heating		Cooling to 25°C	
Heating Temperature (°C)	**G'^a (dyne/cm^2)**	**tanδ**	**G' (dyne/cm^2)**	**tanδ**
70	...[b]	...[b]	...[b]	...[b]
75	74	0.48	1,186	0.39
80	40,710	0.08	65,890	0.03
85	41,660	0.08	71,880	0.03
90	31,770	0.09	63,630	0.03
95	17,320	0.11	45,010	0.03
95[c]	13,336	0.10		

[a] Storage modulus.

[b] Data fluctuated.

[c] Data was obtained as gel held at 95°C for 1 hr.

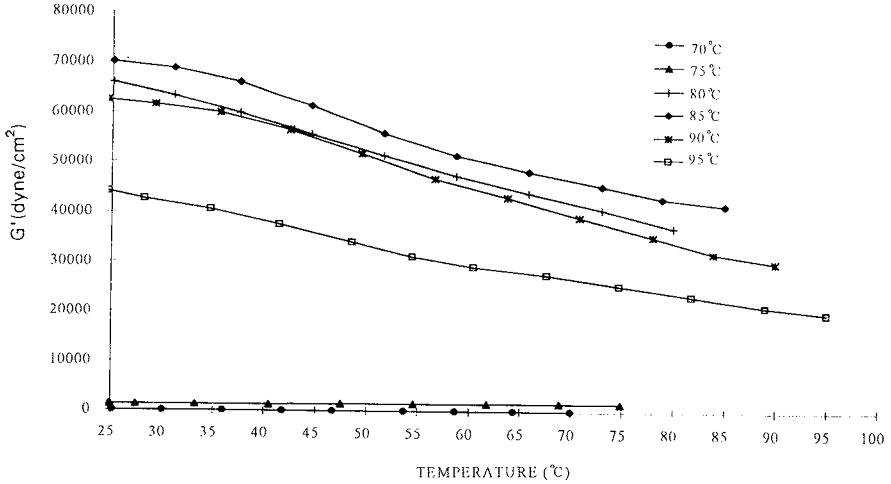

Fig. 9. Storage modulus (G') measurements of 20% KSS7 at different heat treatments during cooling.

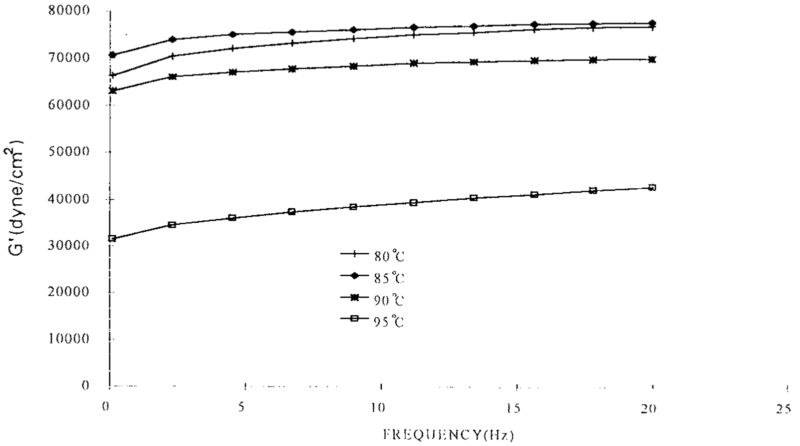

Fig. 10. Storage modulus (G') measurements of 20% KSS7 gels from different heat treatments (frequency sweep).

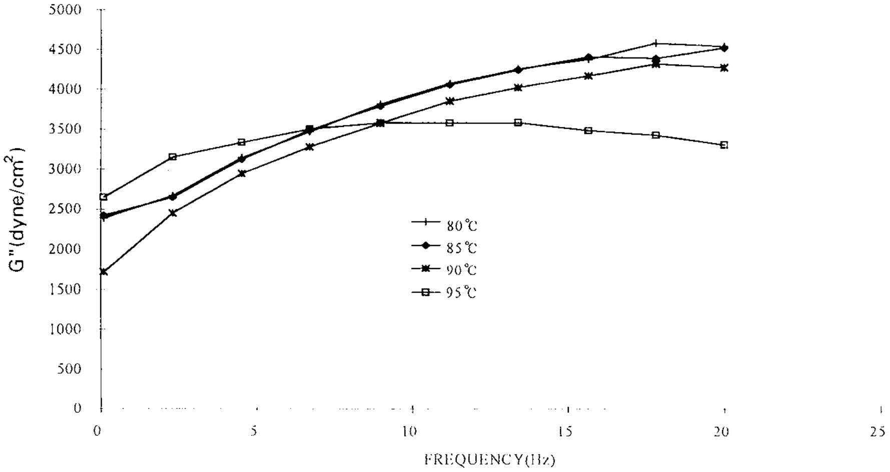

Fig. 11. Loss modulus (G'') measurements of 20% KSS7 gels from different heat treatments (frequency sweep).

tional movements as stress and frequency applied to the hydrocolloid gel. Because its forces for forming a network could hinder actual translational movement, the gel acted as an elastic solid in the tested frequency range.

CONCLUSION

Before gelatinization, the starch granule swelled two to four times its weight with <1% solubility. The blue value of starch suspension was very low. The results indicated that relatively small amounts of molecules were leached out from the granule. Mechanical properties of G' and G'' fluctuated in the heterogeneous starch suspension. However, the swelling power and WSI increased dramatically (swelling power to 27-48 times and WSI to 12–18%) when temperature increased from T_0 to 95°C. For KSS7 and TNu67, the blue value increased to 0.06 and 0.07, respectively, indicating that some amylose molecules leached out after starch gelatinization. At the same time, G' and G'' increased quickly. As the leached-out amylose and swollen granule formed a gel matrix, G' and G'' remained unchanged within the frequency range of 1–20 Hz at 25°C. For TCW70, the blue value did not change after gelatinization. Because it contained only trace amounts of amylose, the increase of G' and G'' were limited, and only the paste could develop at 15–30%. In a starch gel, the leached-out amylose could form a continuous matrix with the swollen granules embedded inside. During gelation, the leached-out amylose concentration was influenced by the amylose content in the starch granule, the starch concentration, and the heating temperature. Sufficient time was required for leached-out amylose and swollen granules to interact to form a special three-dimensional formation. The cooling process could enhance the G' of a starch gel.

ACKNOWLEDGMENTS

We wish to thank the National Science Council, Taiwan, ROC, for financial support. We also appreciate technical assistance of K. M. Lee.

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[Received November 28, 1994. Accepted April 28, 1995]