Effect of Amylose Content on the Rheological Property of Rice Starch[1]

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ABSTRACT

Cereal Chem. 73(4):415–420

The effects of starch variety and amylose on the rheological property of rice starch during heating were investigated with mechanical spectrometry. The results indicated that the storage modulus $(G)'$ of Kaoshiung Sen 7 (KSS7, indica) starch with sufficient concentration increased dramatically at gelatinization temperature ($T_{G'}$). $T_{G'}$ was close to $T\phi = 1$, the temperature for the starch granule to become close-packed. Nevertheless, $T_{G'}$ was slightly lower than $T\phi = 1$ in the low starch concentration system, and slightly higher in the high concentration system. The addition of amylose inhibited the swelling of the starch granule, and consequently, $T_{G'}$ raised slightly. With the addition of amylose, the G' of the starch gel decreased during heating, but may have reinforced the close-packed swollen granule matrix during the period of aging process at 5°C. The waxy rice starch (Taichung waxy 70, TCW70) with trace amylose content, showed much higher swelling power than that of indica rice starch (KSS7). Paste was formed even at high concentration. The rigidity of starch granular structure might be in proportion to its amylose content and in inverse proportion to the degree of granular swelling. Hence, the higher G' for KSS7 rice starch than that for TCW70 can be explained by its high inherent amylose content, which could enhance the rigidity of the starch granular structure. Also, the high G' value for the system with the high starch concentration could be attributed to the low swelling and the strong interaction among the granules. From these results, it was concluded that the major influencing factors on the rheological property of the starch during heating were the granular structure and component, followed by the amount of leached-out amylose in the process.

When the starch granule is heated up to the gelatinization temperature in excess water, heat transfer and moisture transfer phenomena occur. The granule swells to several times its initial size as result of the loss of the crystalline order and the absorption of water inside the granular structure (Whistler 1964). The swelling behavior of cereal starch is primarily the property of its amylopectin content, and amylose acts as both a diluent and an inhibitor of swelling, especially in the presence of lipid (Tester and Morrison 1990a). The maximal swelling might also be related to the molecular weight and the shape of the amylopectin (Tester and Morrison 1990b). Leloup et al (1991) indicated that the composite starch gel, prepared from the pure amylose and amylopectin, reduced the swelling index drastically as the amylose-amylopectin ratio was higher than 2.33.

When starch granules swell, the amylose inside the granules leaches out simultaneously. The leached-out amylose forms a three-dimensional network (Hennig et al 1976, Eliasson 1985, Tester and Morrison 1990a). The swollen granules are embedded in such a continuous matrix (Richardson et al 1981, Wong and Lelievre 1981, Ring and Stainsby 1982, Ring 1985). The paste is formed by gelatinizing the aqueous suspension of starch. When starch concentration is high enough, the paste can convert into gel during cooling. The paste and the gel may be considered as a composite material with the swollen starch granules filling the polymer solution, or polymer gel network (Ring and Stainsby 1982; Miles et al 1985a,b). The initial stages of gelation of starch is dominated by the gelation of the solubilized amylose (Miles et al 1985b). This may imply that the solubilized amylose plays the key role in the gelation of starch. However, Svegmark et al (1993) found that the inherent amylose of potato starch did not contribute to the gel formation, and suggested that the starch granules caused the rheological behavior of the hot paste. The swollen starch granules formed a close-packed gel structure which possessed high shear resistance (Svegmark and Hermansson 1990, 1991). Evans and Haisman (1979) indicated that the material outside the swollen granules (e.g., amylose) had little effect on the rheology of the starch suspensions.

Several effects were examined to acquire further understanding of the mechanisms of gelatinization and starch gel formation: starch variety, starch concentration, and the inherent and added amylose on the rheological properties of rice starch during heating, cooling, and aging.

MATERIALS AND METHODS

Rice Starches

The rice starches of indica (Kaoshiung Sen 7, KSS7) and waxy (Taichung waxy 70, TCW70) varieties were isolated using the alkaline steeping method (Yang et al 1984). The protein contents were <0.14%. The apparent amylose contents of the isolated starches were determined by a modified method introduced by Lii et al (1986) and were 25.60% for KSS7 and 0.99% for TCW70. The onset temperatures for gelatinization of KSS7 and TCW70 measured by differential scanning calorimetry (DSC) were 72.0 and 64.0°C, respectively.

Fractionation of Amylose

The fractionation of KSS7 rice amylose was performed using the method of Takeda et al (1986).

Swelling Power and Water Solubility Index

A 1 % starch suspension (w/w) was heated to 55, 65, 70, 75, 80, 85, 90, and 95°C, respectively, and was kept at that temperature for 60 min, followed by rapid cooling in a ice water bath to room temperature. The starch sample was then centrifuged at 5,000 × g for 20 min. Swelling power was measured with the precipitate (Leach et al 1959), and the water solubility index (WSI) was measured with the supernatant (Holm et al 1985). All tests were performed in triplicate.

Rheological Properties

Small amplitude oscillatory rheological measurements were performed by a Carri-Med-CSL 100 rheometer (TA Instruments Ltd., Surrey, England) equipped with a parallel plate system (20

[1]Presented in part at the AACC 80th Annual Meeting, San Antonio, TX, November, 1995.

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Publication no. C-1996-0530-01R.

mm, dia.). For these measurements, the gap size was set at 1.0 mm. Strain and frequency were set at 0.5% and 1 Hz, respectively.

Effect of starch concentration. Starch suspensions of different concentrations were loaded on the ram of a Carri-Med-CSL 100 rheometer and were covered with a thin layer of mineral oil. The rheological properties were scanned from 45 to 95°C at rate of 1°C/min (Lii et al 1995).

Effect of starch variety. Mixed starch suspensions (20%, w/w) of KSS7 and TCW70 with different ratios (20:0, 15:5, 10:10, 5:15, and 0:20, by weight) were prepared for the investigation of the effect of starch variety on the rheological property. The rheological properties of the samples were scanned from 45 to 95°C at rate of 1°C/min during heating, and from 95 to 5°C at 5°C/min during cooling, and at 5 min intervals up to 1 hr during aging at 5°C. All tests were performed in triplicate.

Effect of amylose content. Purified KSS7 amylose was added to the samples for the measurement of the influence of the amylose on the rheological properties. An amylose solution (2%, w/w) was prepared by dissolving amylose in 8% 1-butanol solution, followed by removal of 1-butanol by heating in a boiling water bath (Miles et al 1985a,b). For the preparations of different concentrations of the amylose-starch suspensions, 2% amylose solution was used as the solvent instead of deionized water. The concentration and the condition used to measure the rheological property of amylose-starch sample were identical to that for the effect of starch variety.

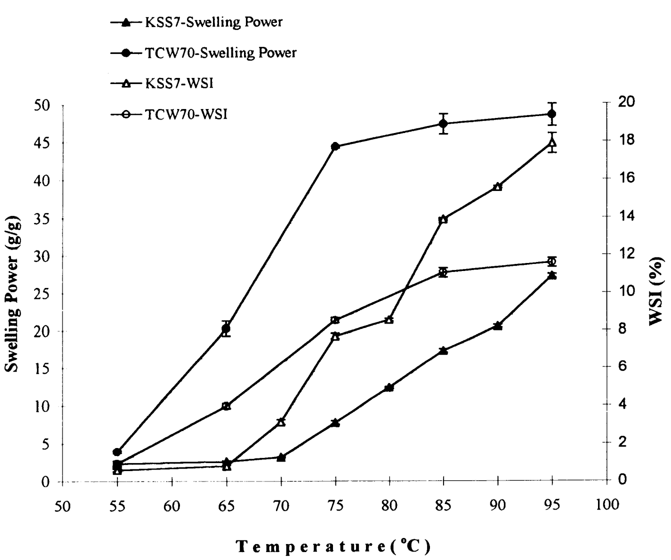

Fig. 1. Swelling powers and water solubility indices of KSS7 and TCW70 at different temperatures.

RESULTS AND DISCUSSION

Swelling Power and Water Solubility Index

The swelling powers and the water solubility index (WSI) of KSS7 and TCW70 rice starches during heating are shown in Figure 1. Both the swelling power and the WSI increased with the increase of the temperature. The swelling power of waxy starch TCW70 was, as expected, higher than that of nonwaxy KSS7.

Rheological Properties

Effect of starch concentration. As shown in Figure 2, the storage modulus (G') of KSS7 starch with the sufficient concentration increased dramatically at a certain temperature, which is hereinafter designated as $T_{G'}$ (Table I) during the heating period. By continuous heating, G' rose to a maximum and then dropped down. Tan δ is the ratio of G'' (loss modulus) to G'. Tan δ was <0.15 when heating temperature was >80°C. KSS7 starch with high G' (>5,000 dyne/cm^2) and low tan δ (<0.2) showed gelling behavior, as suggested by Lii et al (1995), during heating at concentrations of 10–30%, but formed paste at concentrations <10%. The values of G' for TCW70 fluctuated during heating and were very small (<1000 dyne/cm^2), even at concentration up to 30% (Lii et al 1995). On subsequent cooling, the G' of both starch varieties increased. The G' of KSS7 was much higher than that of TCW70 at the same temperature and concentration.

The $T_{G'}$ of KSS7 are listed in Table I. The larger the concentration, the lower the $T_{G'}$. Eliasson (1986) reported that the initial increase in G' was due to the starch granules swelling progressively and becoming closely packed in the system. It has been proposed by Bagley and Christianson (1982) and Doublier et al (1987) that the volume fraction of swollen starch granules in a

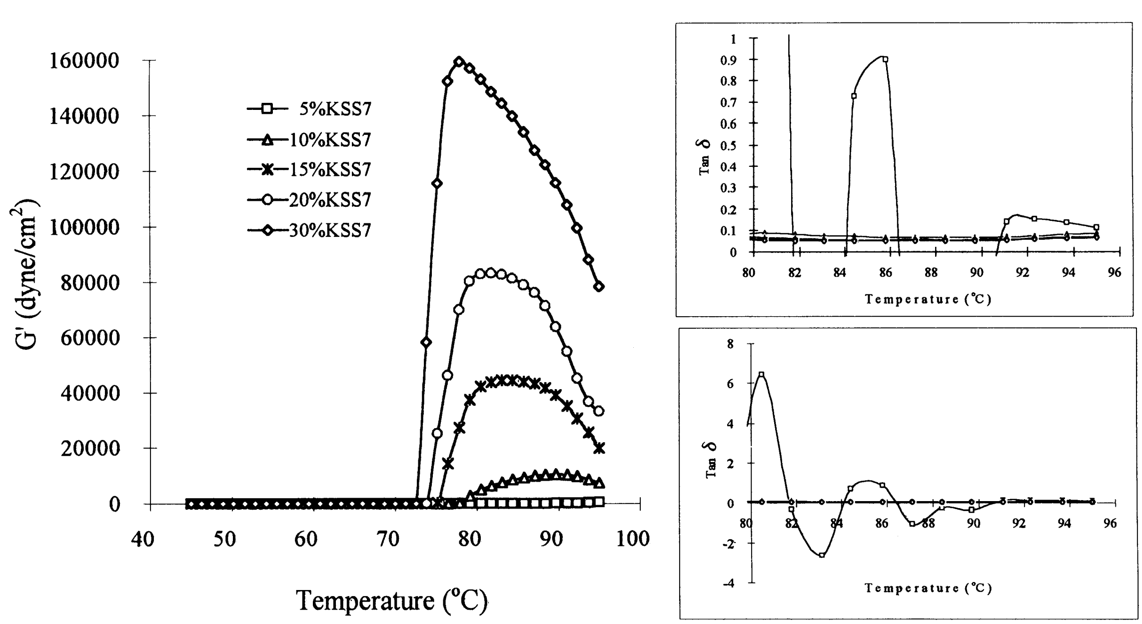

Fig. 2. Storage modulus (G') and loss tangent (tan δ) measurements of KSS7 at different concentrations during heating. (Statistical reliabilities are shown in Tables I and II.)

suspension could be used as an index to determine whether the starch suspension system was closely packed. The volume fraction of the swollen starch granules (ϕ) was expressed in the equation:

$$\phi = C \times Q \times (1 - S) \quad (1)$$

where C was the starch concentration, Q was the swelling power, and S was the WSI. Because of the weight basis of Equation 1, it might be more appropriate to define the volume fraction as the mass fraction (ϕ). When $\phi < 1.0$, the swollen granules are dispersed in excess water. For $\phi = 1.0$, the starch system is just full of closely packed swollen granules, and for $\phi > 1.0$ the starch system is full of deformed particles (Bagley and Christianson 1982). Sandhyarani and Bhattacharya (1989) suggested that low-amylose starch granules were less firm and tended to disintegrate easily while swollen and extensively overcrowded. A high-amylose starch granule was more rigid and not easily ruptured. It could be assumed that the rigidity of starch granule was in inverse proportion to the swelling power and was dependent on the amylose content. In the high-concentration starch system, the swelling power at $\phi = 1$ was not too high; so the rigidity of the swollen starch granule was large.

For predicting the close-packing temperature in starch suspension systems, mass fraction (ϕ) at different concentrations (C) and temperatures could be calculated with Equation 1 by applying the values of swelling power (Q) and WSI (S) shown in Figure 1, which were determined at 1% concentration. The temperature for the mass fraction of 1.0 was designated as $T_{\phi = 1}$, and could be obtained by interpolation method.

The result indicated that the $T_{\phi = 1}$ (Table I) was close to the $T_{G'}$, implying a correlation between $T_{G'}$ and the close-pack temperature of the swollen starch granules. Nevertheless, the $T_{\phi = 1}$ were higher than the $T_{G'}$ when the starch concentration was <10%, and the $T_{\phi = 1}$ became lower when the concentration was >10%. Such differences may be attributed to the fact that starch granules swelled without restriction in the low concentration system (<10%). The swelling behavior was similar to that of the 1% system, in which this concentration was applied for the determination of swelling power and WSI. For the formation of the close-packed structure, some leached-out components might be required. Therefore, the $T_{G'}$ was lower than the $T_{\phi = 1}$. On the other hand, the water amount was not so ample in the high concentration system (>10%), which caused the swelling power of the starch granule to be smaller than in the 1% system. More heat and mass transfer were needed to enhance the swelling of the granule. Consequently the $T_{G'}$ was higher than $T_{\phi = 1}$.

As shown in Figure 2, continuing heating beyond $T_{G'}$ would promote the granule interactions with each other. The water bound to the outside of the swollen granule would lessen, and the surface of the granule would be exposed. This can induce the interaction among the granules to form the gel structure. G' raised rapidly to a plateau or a peak value; 30% KSS7 at 78.1°C showed the maximal G' of 159,300 dyne/cm^2. After G' reached the maximum, continuous heating provided the energy to the inside of granule to break down the residual crystalline structure of the granule. This caused G' to drop back down.

It was concluded from these phenomena that the rheological property of starch depended mainly on the interaction among the close-packed granules and their rigidity during the heating process. Different concentrations displayed different degrees of swelling power, resulting in different granular rigidity, size, and interactions. Evans and Haisman (1979) and Wang and Chiew (1994) indicated that the storage modulus and yield stress of the starch paste were concentration-dependent, and the threshold concentration corresponded closely to the point at which the volume of flocculated and swollen starch granules just filled the total volume of the system. The rheological behavior of gelatinized starch suspension was primarily due to intergranular interaction, such as entangle-

TABLE I
Effects of Starch Concentrations of KSS7 Rice at Different Temperatures (°C)[a]

Concentration, %	$T_{G'}$	$T_{G'}$ (2% amylose added)	$T_{\phi = 1}$
5	nd[b]	nd	94.8
6	88.6	...	91.8
8	81.8	...	84.5
10	79.3	82.7	79.4
12	79.0	...	77.5
15	77.3	80.9	76.4
18	75.5	...	74.2
20	75.2	78.3	73.9
30	72.0	...	69.9
40	70.2	...	65.1

[a] Temperature at which G' increases dramatically with dynamic rheometer ($T_{G'}$). Temperature at which starch becomes close packed ($T_{\phi = 1}$). Standard deviation was less than ±1%. $n = 3$.
[b] Not detectable.

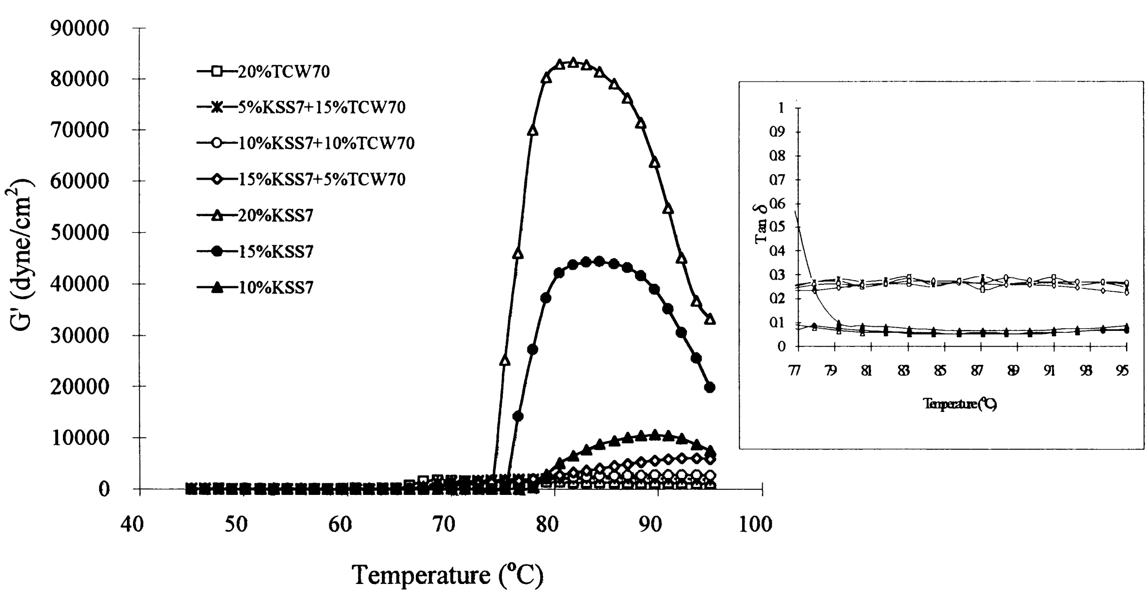

Fig. 3. Storage modulus (G') and loss tangent (tan δ) measurements of mixed rice starch systems with different ratios of KSS7 and TCW70 during heating. (Statistical reliabilities are shown in the text.)

was primarily due to intergranular interaction, such as entanglement between surface molecules of adjacent granules and the properties of the granules themselves.

Effect of starch variety. The rheological properties of the mixed starch samples with different ratios of KSS7 and TCW70 during heating and cooling are shown in Figures 3 and 4. The average standard deviation was ±5%. The result indicated that the 20% KSS7 starch sample gave the highest G', and the 20% TCW70 gave the lowest. With the increasing ratio of KSS7, both G' and $T_{G'}$ increased; tan δ decreased in favor of gelling behavior. The swelling power of TCW70 was higher than that of KSS7 from 55 to 95°C (Fig. 1). The starch mixture easily became close-packed as the concentration of TCW70 increased. However, the rigidity of the swollen granules of TCW70 was much lower than that of KSS7. Consequently, the rigidity of the system decreased. The result coincided with the fact that starch granules with low amylose content were less rigid and tended to disintegrate easily when swollen intensely and overcrowded (Sandharani and Bhattacharya 1989). The structure of the granule with high amylose content was highly strengthened and more rigid.

A peculiar phenomenon is depicted in Figures 3 and 4. Although the total starch concentration of the mixture of 15% KSS7 and 5% TCW70 sample was higher than that of 15% KSS7 alone, the G' of KSS7 (5,854 ± 289 and 10,460 ± 446 dyne/cm^2 for 95 and 5°C, respectively) was smaller than that for TCW70 (19,700 ± 300 and 40,130 ± 1,978 dyne/cm^2 for 95 and 5°C, respectively) during both heating and cooling cycles. Perhaps the TCW70 starch swelled before KSS7 and acted like added amylopectin. Consequently, the added amylopectin restricted the starch granules from swelling (Svegmark and Hermansson 1993). However, it was more likely that there was a small amount of large swollen and less rigid TCW70 starch granules in a matrix of rigid granules, and leached out KSS7 amylose may weaken the whole structure of the mixed system. It implied that the rheological behavior of rice starch was largely affected by the characteristics and the property of the starch granule.

Effect of amylose content. The result of the investigation on the effect of the added amylose on the KSS7 starch during heating indicated that G' of the sample was lower (Table II), and $T_{G'}$ was higher with the addition of 2% amylose (Fig. 5). The only exceptions were the G' of 2% amylose and 20% KSS7 at 95°C. The addition of amylose competed with the starch granule for water. This also elevated the $T_{G'}$. The gelation of amylose was regarded as a partial crystallization process (Collison 1968). Ring (1985) found that the swollen starch granule was entrapped in the gel matrix of the amylose and strengthened the gel structure. The granule with limited swelling would increase the rigidity of the amylose gel dramatically. Liu and Lelievre (1992) and Ojima and Ozawa (1985) reported that the native starch granule would also reinforce the structure of gel matrix, and such reinforcement increased with the starch gelatinization. Strong interaction or adhesion between the gel matrix and the rigid filler with a sufficient volume fraction was required for the solid gel structure. However, Green et al (1990) proposed that the filler weakened the structure. The addition of potato amylose did not contribute notably to the gel strength of potato starch (Svegmark et al 1993). Evans and Haisman (1979)

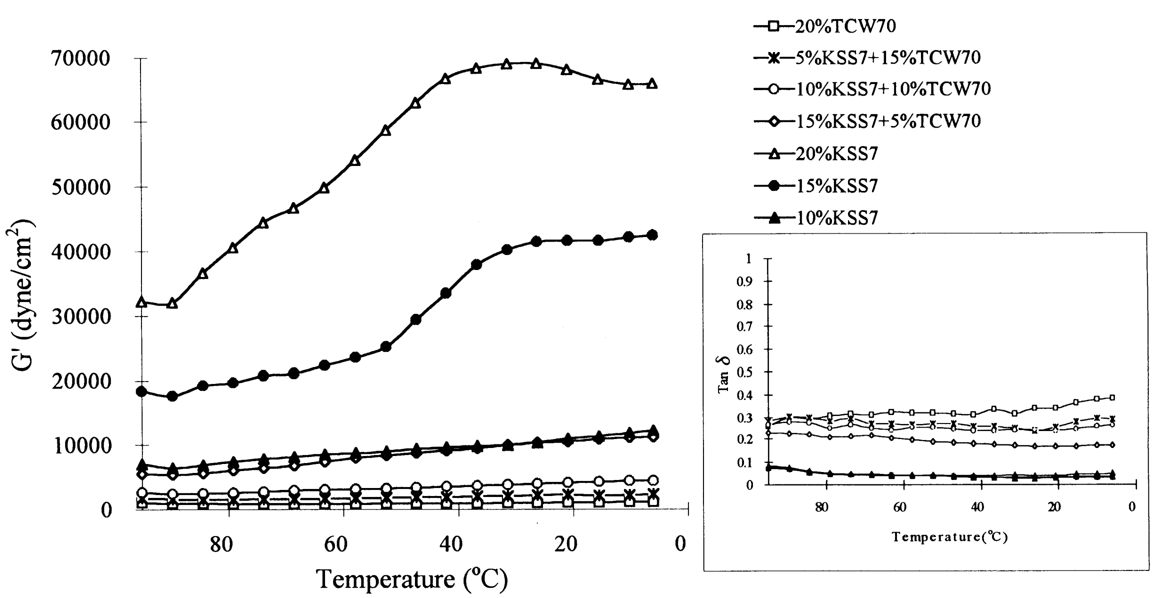

Fig. 4. Storage modulus (G') and loss tangent (tan δ) measurements of mixed rice starch systems with different ratios of KSS7 and TCW70 during cooling. (Statistical reliabilities are shown in the text.)

TABLE II
Effect of Amylose on Rheological Properties of KSS7 Starch with Different Treatments[a,b]

Concentration, %	Heated to 95°C		Cooled to 5°C		Aged at 5°C for 1 hr	
	G'	tan δ	G'	tan δ	G'	tan δ
5	507 ± 8	0.10 ± 0.00	563 ± 12	0.06 ± 0.00	578 ± 24	0.11 ± 0.01
5 + 2% AM[c]	188 ± 5	0.18 ± 0.01	557 ± 10	0.14 ± 0.02	1,710 ± 283	0.06 ± 0.00
10	6,995 ± 190	0.08 ± 0.00	12,730 ± 156	0.14 ± 0.02	17,120 ± 230	0.03 ± 0.00
10 + 2% AM	4,072 ± 269	0.10 ± 0.02	12,730 ± 2,140	0.05 ± 0.00	19,050 ± 1,715	0.04 ± 0.00
20	32,210 ± 645	0.07 ± 0.00	65,960 ± 255	0.04 ± 0.00	66,290 ± 270	0.03 ± 0.00
20 + 2% AM	33,450 ± 2,311	0.07 ± 0.00	60,530 ± 2,185	0.04 ± 0.00	61,250 ± 2,267	0.04 ± 0.00

[a] G' (dyne/cm^2) storage modulus.
[b] Mean ± standard deviation; n = 3.
[c] 2% KSS7 amylose added.

also suggested that the substance outside the granule (e.g., amylose) had little effect on the rheology of the starch suspensions. The data shown in Figure 5 and Table II might imply that the swollen granule influenced the rheological property of the starch much more than did amylose added during heating.

The effect of added amylose on rheological properties of KSS7 during cooling is shown in Figure 6. The result indicated that the G' of the 5 and 10% starch samples increased slightly more with the addition of amylose than without. Similar results were observed during the aging at 5°C up to 60 min (Fig. 7). These phenomena were due to the addition of amylose to starch (Svegmark and Hermansson 1993). They showed that 1–10% potato starch swollen in the amylose solution exhibited a lower complex modulus (G^*) than the starch swollen in water during heating and a sharp increase in G^* due to amylose gel formation by cooling the system. However, G' increment for the sample of 20% concentration was higher than that with added amylose. It hinted that a very strong interaction occurrence at the cooling period probably existed between the adjacent (or aggregated) granules; also, the added amylose did not contribute noticeably to the gel rigidity at a high concentration.

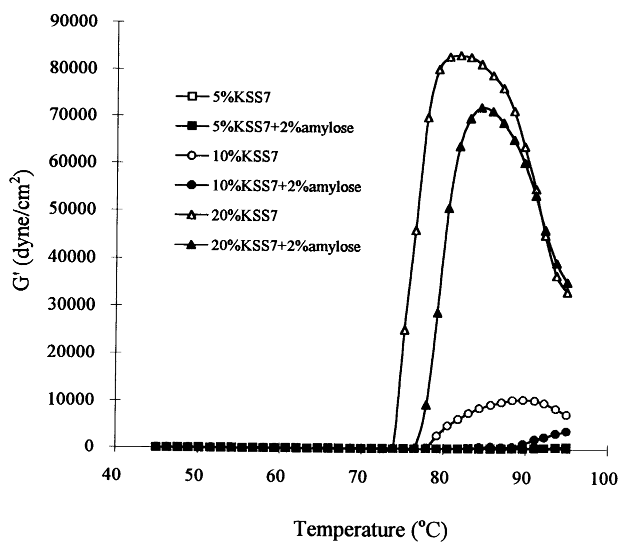

Fig. 5. Effect of amylose on storage modulus (G') of KSS7 during heating. (Deviation from the mean is shown in Tables I and II.)

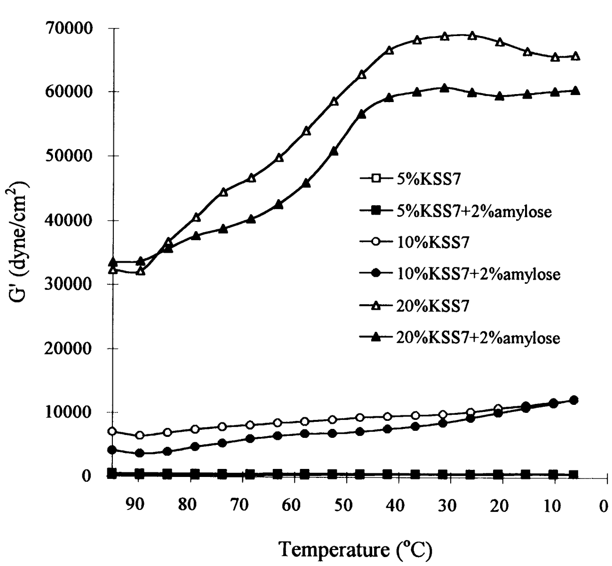

Fig. 6. Effect of amylose on storage modulus (G') of KSS7 during cooling. (Deviation from the mean is shown in Table II.)

Figure 8 showed the rheological properties of TCW70 starch sample at various concentrations, with and without the addition of 2% purified KSS7 amylose, during their aging cycles. After aging for 1 hr, the G' of the systems with and without added amylose were 1,268 ± 69 and 282 ± 23 dyne/cm^2 for 5% TCW70, 1,098 ± 33 and 608 ± 22 dyne/cm^2 for 10% TCW70, and 1,483 ± 144 and 1,163 ± 20 dyne/cm^2 for 20% TCW70. The average standard deviation was ±5%. The addition of amylose increased the G', especially at 10% concentration or lower, which was attributed to the less rigid swollen granule of starch. Furthermore, the added amylose might entangle with granules to enhance the structure of the matrix in the low starch concentration system. Consequently, G' increased drastically. However, the addition of amylose did not show a significant effect on the high concentration of starch with inherent amylose, as previously indicated. These results further proved that the rheological behavior of starch was mainly dependent on the characteristics and properties of the granule and less on the amount of amylose. It was concluded that the rigidity of

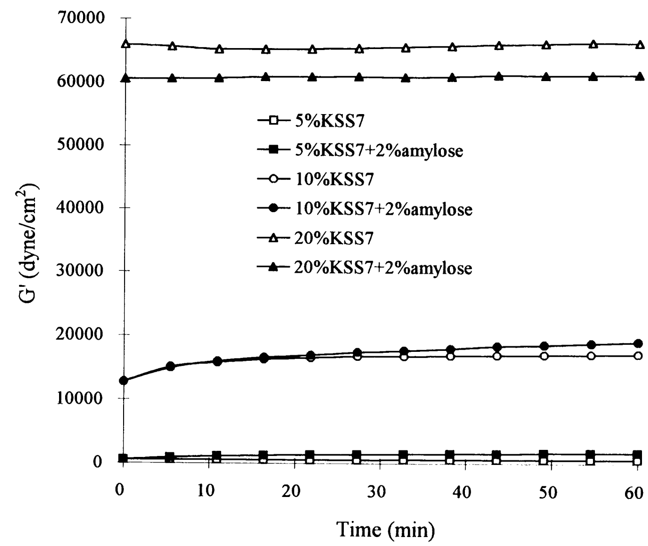

Fig. 7. Effect of amylose on storage modulus (G') of KSS7 during aging at 5°C. (Deviation from the mean is shown in Table II.)

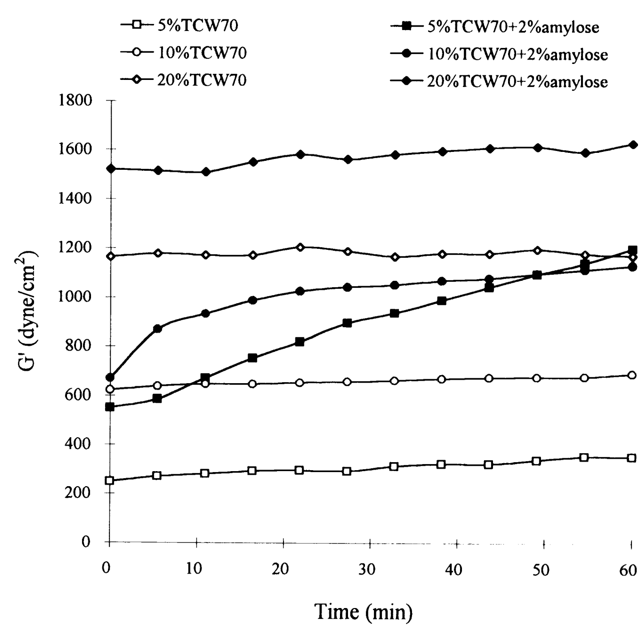

Fig. 8. Effect of amylose on storage modulus (G') of TCW70 during aging at 5°C. (Deviation from the mean is shown in the text.)

swollen granule played a major role on the rheological behavior by building a basic structure of the matrix, and the leached-out or added amylose strengthened the matrix during cooling and aging.

CONCLUSION

In mass and energy transfers, heating raises the moisture diffusivity and provides energy to break down the crystalline structure inside the granule. For waxy rice starch with a very low amylose content, the crystalline structure was easily destroyed. The starch granule also absorbed much water, exhibiting high swelling power. It was assumed that the rigidity of starch granule was in inverse proportion to the swelling power and was dependent on the amylose content. Starch granules were close-packed at low degrees of swelling with more rigid structure at the high starch concentration. Starch granules were not close-packed when the swollen granule was less rigid in the low concentration system. The addition of the purified amylose to the starch system increased $T_{G'}$ slightly. Added amylose did not increase the G' during heating, but increased during aging at low temperature for waxy rice or low starch concentration. This suggested that the amylose leached out from the starch granule on heating did not contribute much to reinforcing the structure of the matrix of swollen starch granules. Nevertheless, the recrystallization of amylose and the interaction between amylose and the swollen granule strengthened the cross-linking of the starch gels during aging. Therefore, it was concluded that the starch granular properties and characteristics were the major factors for the starch rheological behavior, followed by amylose. However, for the complete elucidation the rheological properties of starch under different conditions, further investigation would be necessary.

ACKNOWLEDGMENT

We wish to thank the National Science Council, Taiwan, for partial financial support (NSC 84-2113-M-001-002).

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[Received November 11, 1995. Accepted March 11, 1996.]