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Rheological properties of rice amylose gels and their relationships to the structures of amylose and its subfractions

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Abstract

Rice amyloses, extracted from three rice starches (KSS7, TCS10 and TNu67), were used in this study. The rheological properties of amylose gels (3%, w/w) and the structures of amylose and its subfractions were investigated, the relationships between rheological properties and structures of amylose were then considered. During aging for 5 h, TCS10 amylose gel showed the greatest G'_{initial} and G'_{final} values and the lowest $\tan\delta_{\text{initial}}$ and $\tan\delta_{\text{final}}$ values among the three amylose gels studied. On the other hand, TNu67 amylose gel had the lowest G'_{initial} and G'_{final} values. The DP_n (number-average degree of polymerization) of rice amyloses studied ranged from 1004 to 1289. The GPC profiles of the amyloses, on a TSK HW-65F column, showed bimodal distributions and the amyloses could be separated into subfractions with larger ($F1$) and smaller ($F2$) molecular weights. Among the samples studied, TCS10 had the greatest DP_n and longest CL (average chain length) for amylose and the $F1$ and $F2$ subfractions. KSS7 and TNu67 had similar DP_n for the amylose and subfractions, while the CL of the $F1$ and $F2$ amylose subfractions of KSS7 were longer than those of TNu67. The subfractions of TNu67 amylose also showed obviously greater NC (number of chains per molecule) and lower β-amylolysis limit than did the other amyloses studied. Results indicated that the rheological properties of rice amylose gel were highly related to the structures of amylose and its subfractions. Amylose with greater DP_n, longer CL, and smaller NC led to an amylose gel with higher gel strength or elasticity.

Keywords: Rice; Amylose; Subfraction; Rheological property

1. Introduction

Starch consists of two polysaccharides, amylose and amylopectin. Amylose is essentially a linear molecule containing glucose unit linked by α-1,4 linkages with a few branches (Hizukuri, Takeda, Yasuda, & Suzuki, 1981). In general, amylose plays an important role in determining the characteristics of starch from various botanical sources (Thitipraphunkul, Uttapap, Piyachomkwan, & Takeda, 2003; Doublier & Choplin, 1989). In addition, variations in the structures of both amylose and amylopectin, including molecular size, average chain number, and average chain length, result in differences in physicochemical properties of starch granules, such as gelatinization, pasting, and retrogradation (Swinkels, 1985; Mua & Jackson, 1997; Thitipraphunkul et al., 2003).

The chain length and molecular weight of amylose have been proposed to be related to the retrogradation tendency (Suzuki, Hizukuri, & Takeda, 1981; Takeda, Takeda, & Hizukuri, 1983). The aggregation of amylose in aqueous solution has been observed by a number of workers principally using light-scattering methods (Kodama, Noda, & Kamata, 1978). A major finding of such studies is that the rate of aggregation of amylose is strongly dependent on the chain length. Pfannemüller, Mayerhöfer, and Schultz (1971) indicated that the aggregation of amylose

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was found to be most rapid for chain lengths with degrees of polymerization (DP) of around 80 anhydrous glucose units. Amylose with a chain length of DP < 110 was found to precipitate from aqueous solution (Gidley & Bulpin, 1989). Both precipitation and gelation occurred for chain lengths (DP) ranging from 250 to 660. For longer chains (DP > 1100), gelation was found to predominate over precipitation. The relatively increasing rate of storage modulus (G') for amylose gel was found to be dependent on its chain length (Clark, Gidley, Richardson, & Ross-Murphy, 1989). Studies indicate that, in addition to the concentration of system, the molecular properties, such as chain length and molecular size, are the most important variables in the gelation of amylose (Ellis & Ring, 1985; Gidley & Bulpin, 1989). However, the molecular weight distribution of amylose is very wide (Hizukuri, 1984; Hizukuri et al., 1988; Takeda, Maruta, & Hizukuri, 1992), and the distribution patterns and the structures of amylose are uniquely related to their botanical sources (Hizukuri, 1984; Takeda & Hizukuri, 1987). Hizukuri et al. (1988) indicated that amylose from the water chestnut contained three components with various sizes and different structures. The same was true for maize and rice starches (Takeda et al., 1992). Mua and Jackson (1997) further indicated that both weight-average molecular weights and branching ratios of amylose fractions influenced the pasting, gel textural and retrogradation functions of starch.

It was suggested that gelation of the amylose component was important in starch gelation, at least during the initial stage (Miles, Morris, Orford, & Ring, 1985). Studies of the gelation of amylose are also relevant to the understanding of retrogradation of starch. Although the mechanism involving in amylose gelation has been addressed (Miles, Morris, & Ring, 1985), the relationships between gelation (rheological properties) of amylose and the structures of amylose and its subfractions have rarely been studied. In this study, amyloses were extracted from rice starches, the subfractions of amylose were fractionated by gel permeation chromatography (GPC). The rheological properties of amylose gels during aging for 5 h were monitored, and the molecular sizes and structures of amylose and its subfractions were examined. The discrepancies on the structures and gelation properties of amyloses from different rice varieties were compared, and the relationships between the rheological properties of amylose gel and the structures of amylose and its subfractions are considered.

2. Materials and methods

2.1. Rice

Polished rice kernels of two indica (Kaohsiung Sen 7, KSS7 and Taichung Sen 10, TCS10) and one japonica (Tainung 67, TNu67) varieties were obtained from the Taichung District Agricultural Research and Extension Station (Changhua, Taiwan).

2.2. Isolation of rice starch and amylose

Rice starch was isolated from the polished rice by a modified alkaline steeping method (Yang, Lai, & Lii, 1984). The apparent amylose contents of KSS7, TCS10 and TNu67 starches were 24.2%, 15.1% and 15.7%, respectively.

The rice starch was thoroughly defatted for 48 h with 85% methanol by Soxhlet extraction (Lu, Chen, Lin, & Chang, 2005), and amylose was fractionated and purified from the defatted rice starch, using the method of Takeda, Hizukuri, and Juliano (1986), except that the amylose was collected by centrifuging at 39,000*g*. The molecular size distribution and the λ_{max} profile of the extracted amylose were examined by GPC on a Fractogel TSK HW-75F column (Merck, Germany) following the procedures described in Takeda, Shirasaka, and Hizukuri (1984) Biliaderis, Grant, and Vose (1979).

2.3. Fractionation of amylose

A GPC method was used to fractionate the amyloses according to the procedures proposed by Takeda et al. (1992). Amylose solution (15 ml, 10 mg/ml) was applied to a column (2.6 × 100 cm) packed with Fractogel TSK HW-65F (Merck). The column was eluted with distilled water at a flow rate of 30 ml/h under 45 °C, and the eluent was collected at 15 ml per tube. Total carbohydrate content was measured by the phenol–sulfuric acid method (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956). The amylose fractions were lyophilized for further investigations.

2.4. Structural analysis

Blue value, λ_{max}, iodine affinity and β-amylolysis were determined according to the methods proposed by Gilbert and Spragg (1964), Biliaderis et al. (1979), Schoch (1964), and Hizukuri et al. (1981), respectively. The number-average degree of polymerization (DP_n) of amylose and its subfractions were measured by the modified Park–Johnson method described in Hizukuri, Kaneko, and Takeda (1983). The modified rapid Smith degradation method (Hizukuri & Osaki, 1978; Hizukuri et al., 1981) was used to determine the average chain length (CL) of molecules. The average number of chains (NC) per molecule was then calculated by DP_n/CL.

2.5. Rheological measurement

Amylose solution (3%, w/w) was prepared by mixed 30 mg amylose with 88 μl of 8% *n*-butanol (w/v) and 7.8 μl distilled water in a 1.5 ml vial. The vial was heated in a boiling water bath under streaming nitrogen to remove *n*-butanol. The final weight of the sample was adjusted to 1 g with boiling water. Small amplitude oscillatory rheological measurements of 3% amylose solution were performed on a rheometer (Carri-Med CSL-100, TA Instruments, Surrey, England) equipped with a parallel plate system (20 mm diameter). A layer of silicone fluid was applied to the exposed edge of sample to minimize the water evaporation. The gap size was set at 1 mm. The strain and frequency used were 1.5% and 1 Hz, respectively. The sample was cooled down to 4 °C in 5 min. The rheological properties of amylose gel aging at 4 °C for 5 h were monitored.

3. Results and discussion

3.1. Iodine affinity, blue value and λ_{max} of amyloses

Table 1 summarizes the iodine affinity, blue value and λ_{max} of amyloses from the three rice starches studied. The iodine affinity of amyloses ranged from 17.6% to 19.0%. KSS7 amylose had the highest iodine affinity, while TCS10 had the lowest. The blue value of rice amyloses ranged from 1.06 to 1.10. The λ_{max} of KSS7, TCS10 and TNu67 amyloses were 630, 633 and 638 nm, respectively.

3.2. Rheological properties of amylose gels during aging

Fig. 1 shows the G' profiles of amylose gels aged at 4 °C for 5 h. The change of G' of amylose gels could be separated into two stages, the first (initial) and the second stages. The initial stage occurred within the first 20 min after the gel was cooled to 4 °C, during which time there was a rapid increase in G' values. During the second stage (20 min to 5 h), G' values increased more slowly. G' values of the gels of TCS10 and TNu67 amyloses increased less than that of the KSS7 amylose gel during the second stage.

Table 1
Iodine affinity, blue value and λ_{max} of rice amyloses

Property	KSS7	TCS10	TNu67
Iodine affinity (%, w/w)	19.0 ± 0.3[a]	17.6 ± 0.2	18.6 ± 0.4
Blue value	1.09 ± 0.02	1.06 ± 0.03	1.10 ± 0.03
λ_{max} (nm)	630 ± 1	633 ± 0	638 ± 0

[a] Mean ± SD; $n = 3$.

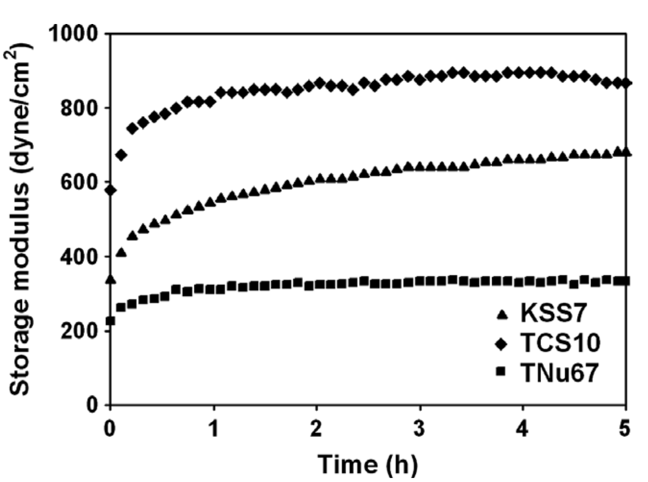

Fig. 1. Storage modulus (G') of 3% rice amylose gels aging at 4 °C for 5 h.

Table 2
Rheological parameters for amylose gels aging at 4 °C for 5 h

Parameter	KSS7	TCS10	TNu67
$G'_{initial}$ (Pa)	335.7 ± 92.0[a]	577.8 ± 50.9	227.1 ± 15.0
G'_{final} (Pa)	647.8 ± 82.4	834.4 ± 4.3	271.0 ± 94.9
$G''_{initial}$ (Pa)	56.6 ± 1.3	41.7 ± 0.2	25.3 ± 6.6
G''_{final} (Pa)	59.5 ± 2.6	38.8 ± 1.0	26.1 ± 1.4
$\tan\delta_{initial}$	0.181 ± 0.046	0.073 ± 0.006	0.110 ± 0.022
$\tan\delta_{final}$	0.093 ± 0.008	0.046 ± 0.012	0.084 ± 0.016

[a] Mean ± SD; $n = 3$.

Rheological parameters of amylose gels during 5 h aging at 4 °C are summarized in Table 2. The order of both $G'_{initial}$ and G'_{final} of the amylose gels studied was TCS10 > KSS7 > TNu67. The $G'_{initial}$ values were less than the G'_{final} values for all three gels, the difference being least for the TNu67 gel. The $G''_{initial}$ and G''_{final} values of amylose gels were all lower than 60 Pa, and were obviously less than their counterpart $G'_{initial}$ and G'_{final} values (Table 2). As compared to the obvious change of G' during aging, much less difference between $G''_{initial}$ and G''_{final} of each amylose gel was found. This result indicated that the G' value could be used as a good index on the rheological change of amylose gel during aging. For KSS7 amylose gel, the increasing ratio of G' $((G'_{final} - G'_{initial})/G'_{initial} \times 100\%)$ after 5 h of aging was 93.0%, while the increasing ratio for TCS10 and TNu67 were 44.4% and 19.3%, respectively. This revealed that the increment of gel strength for KSS7 amylose was more profound than those of other amylose gels.

The $\tan\delta_{initial}$ and $\tan\delta_{final}$ values for the amylose gels ranged from 0.05 to 0.18 (Table 2). The fact that all were <1 agrees with the results of Doublier and Choplin (1989). TCS10 amylose gel had the lowest values of $\tan\delta_{initial}$ and $\tan\delta_{final}$, which indicated that TCS10 amylose gel was more elastic than were the other two amylose gels. Although both $G'_{initial}$ and G'_{final} values of KSS7 were obviously greater than those of TNu67, the $\tan\delta_{initial}$ and $\tan\delta_{final}$ values of KSS7 were also higher than those of TNu67 amylose gel. This implied that the TNu67 amylose gel was more elastic than was the KSS7 gel.

3.3. Molecular characteristics of amyloses

Takeda et al. (1984) suggested that GPC performance on TOYOPEARL HW-75F was a sensitive and useful method for examining the purity of amylose. The GPC profiles of rice amyloses on the TSK HW-75F column are illustrated in Fig. 2. Only one asymmetric peak was observed on fraction numbers 55–91 for TCS10 and on numbers 63–92 for KSS7 and TNu67. This result suggests that all extracted amylose samples are free of amylopectin. The GPC profiles of rice amyloses were similar to amyloses from lily, wheat, kudzu, tapioca and potato starches reported by Takeda et al. (1984). The λ_{max} of GPC fractions of amylose ranged from 620 to 660 nm, which were

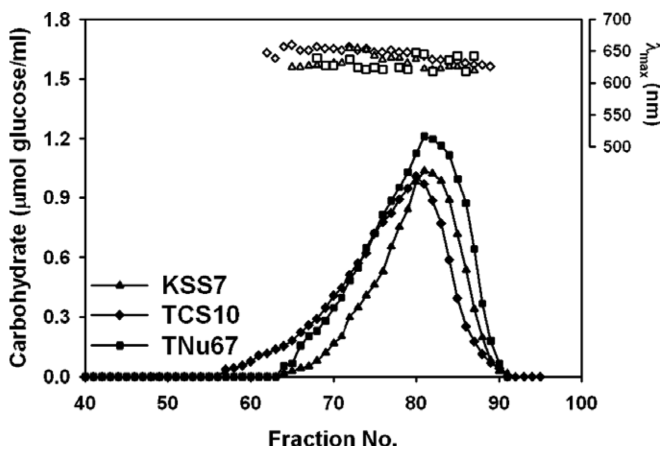

Fig. 2. GPC profiles of rice amyloses on TSK HW-75F column. Solid and hollow symbols are total carbohydrate and λ_{max}, respectively.

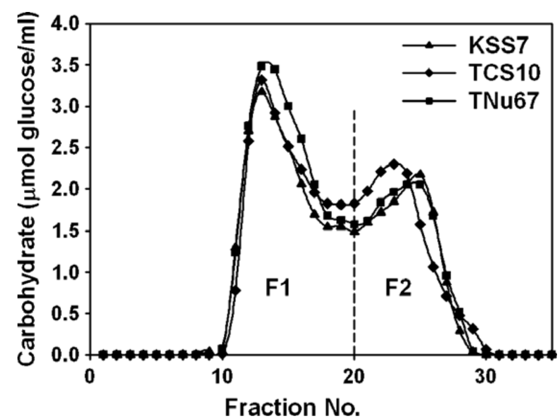

Fig. 3. GPC profiles of rice amyloses on TSK HW-65F column.

in line with the typical λ_{max} of amylose (Thitipraphunkul et al., 2003; Hizukuri, 1996); this result reconfirms the purity of amyloses extracted in this study.

The DP_n of rice amyloses studied ranged from 1004 to 1289 (Table 3), and was in the order of TCS10 > KSS7 ≈ TNu67. The DP_n values of rice amyloses studied are similar to those of other cereals, such as wheat, barley, normal maize, but greater than that of amylomaize, and much less than those of most root, tuber and legume starches (Hizukuri, 1996). The average chain length (CL) of amyloses was in the order of TCS10 > KSS7 > TNu67. KSS7 and TCS10 amyloses had similar average chain numbers (NC, 2.5 for KSS7 and 2.4 for TCS10), while TNu67 had a greater NC (3.5) value. The β-amylolysis limit of KSS7 (83.5%) and TCS10 (84.2%) amylose were also similar, and a lower value (79.8%) was found for TNu67. This result indicated that TNu67 amylose was more branched; for a greater the degree branching, the lower was the β-amylolysis.

Table 3
Structural characteristics of rice amyloses

Characteristic	KSS7	TCS10	TNu67
Number-average DP (DP_n)	1075 ± 44[a]	1289 ± 23	1004 ± 44
Average chain length (CL)	430	537	287
Average chain number (NC)	2.5 ± 0.2	2.4 ± 0.2	3.5 ± 0.3
β-Amylolysis limit (%)	83.5 ± 0.4	84.2 ± 0.3	79.8 ± 0.4

[a] Mean ± SD; $n = 3$.

3.4. Fractionation and molecular characteristics of amylose subfractions

The fractionation of the rice amyloses was carried out by GPC on a TSK HW-65F column. Similar GPC profiles were observed for the three amyloses studied, which were bimodal distributions (Fig. 3). From the GPC profiles, rice amylose could be separated into two fractions as *F*1 (amylose with larger molecular weight) and *F*2 (amylose with smaller molecular weight). The result was different from the report on an IRRI indica rice amylose which had only one asymmetric peak on tandem columns of HW-65F and HW-60F (Takeda et al., 1992). This discrepancy may result from the differences of rice variety and column system used.

The content and molecular characteristics of amylose subfractions of each rice amylose are summarized in Table 4. Results indicated that TNu67 amylose had a slightly higher *F*1 fraction content than had KSS7 and TCS10 amyloses. The DP_n of *F*1 and *F*2 fractions of TCS10 were obviously greater than those of KSS7 and TNu67, while KSS7 and TNu67 amyloses had similar DP_n values for both *F*1 and *F*2 fractions. For the same amylose, the CL of the *F*1 fraction was longer than that of the *F*2 fraction. The CL of both *F*1 and *F*2 were in the order of TCS10 > KSS7 > TNu67.

Table 4
Structural characteristics of subfractions of rice amyloses

Characteristic	KSS7		TCS10		TNu67	
	*F*1	*F*2	*F*1	*F*2	*F*1	*F*2
Weight (%)	65.3	34.7	63.0	37.0	67.6	32.4
Number-average DP (DP_n)	1486 ± 78[a]	436 ± 8	1730 ± 48	530 ± 10	1472 ± 73	441 ± 9
Average chain length (CL)	346	291	412	353	294	152
Average chain number (NC)	4.3 ± 0.4	1.5 ± 0.1	4.2 ± 0.4	1.5 ± 0.2	5.0 ± 0.3	2.9 ± 0.3
β-Amylolysis limit (%)	76.8 ± 0.1	91.0 ± 0.2	78.3 ± 0.2	90.3 ± 0.4	72.1 ± 0.3	87.6 ± 0.1

[a] Mean ± SD; $n = 3$.

The NC of the $F1$ fraction was greater than that of the $F2$ fraction for the same amylose. This result was in line with the previous report that amylose subfractions with larger molecular weight had relatively the greater NC (Takeda et al., 1992). The NC of $F1$ and $F2$ fractions for TNu67 amylose were greater than those of KSS7 and TCS10 amyloses, while no differences were found between the NC of both fractions of KSS7 and TCS10 starches. The β-amylolysis limits of amylose subfractions were in reverse order to the NC values, as should be the case (Thitipraphunkul et al., 2003; Takeda et al., 1992).

3.5. Relationships between rheological properties of amylose gels and their molecular characteristics

Among the rice amyloses examined in this study, TCS10 amylose gel had the greatest values of G' and the smallest values of $\tan\delta_{\text{initial}}$ and $\tan\delta_{\text{final}}$ (Table 2), implying a higher gel strength and more elastic gel properties than for the other two amyloses. This result could be attributed to the greatest molecular size (DP_n) and longest chain length (CL) of TCS10 amylose (Table 3) and its subfractions (Table 4) among the three amyloses studied. Ellis and Ring (1985) indicated that 3.5% amylose gel, made from the leaching-out fractions of pea, potato and maize starches, with greater molecular weight (M_w in range of 5×10^5–1.245×10^6 or DP in the range of 3100–7600) did not had stronger gel strength. This could be resulted from the slow and limited aggregation of amylose with greater DP (DP > 2000), (Gidley & Bulpin, 1989). However, the DP_n of rice amylose used in this study were all less than 1300, therefore the greater DP and longer CL in rice amylose led to the higher G' of the amylose gel.

Although KSS7 and TNu67 amyloses had similar DPs (Table 3), the strength and the development during aging of KSS7 amylose gel were more profound than were those of TNu67 amylose gel (Fig. 1). KSS7 and TNu67 amyloses also had similar molecular sizes in amylose subfractions (both $F1$ and $F2$); the higher gel strength of KSS7 amylose could be attributed to its longer chain length and smaller NC. This suggests that the amylose with longer chain length and smaller NC retrogrades more easily and fast, and enhances the strength of gel and its development during aging.

4. Conclusions

Amyloses from KSS7, TCS10 and TNu67 rice starches were isolated and fractionated in this study. By using a TSK HW-65F column, amylose could be separated into large ($F1$) and small ($F2$) molecular weight fractions. The structure of amylose would affect the rheological properties of 3% amylose gel. The greatest DP_n, the longest CL and the smallest NC of TCS10 amylose and its two subfractions resulted in the greatest G' value and the smallest $\tan\delta$ value of gel during aging, while the TNu67 amylose, with the smallest DP_n, the shortest CL and the greatest NC of amylose and its subfractions, formed the weakest gel. Results suggest that the structures of amylose subfractions affect the rheological properties of amylose gel, and that rice amylose and its subfractions, with greater DP_n, longer CL and smaller NC, form gels with greater G' values.

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References

Biliaderis, C. C., Grant, D. R., & Vose, J. R. (1979). Molecular weight distributions of legume starches by gel chromatography. *Cereal Chemistry, 56*, 475–480.

Clark, A. H., Gidley, M. J., Richardson, R. K., & Ross-Murphy, S. B. (1989). Rheological studies of aqueous amylose gels: the effect of chain length and concentration on gel modulus. *Macromolecules, 22*, 346–351.

Doublier, J. L., & Choplin, L. (1989). A rheological description of amylose gelation. *Carbohydrate Research, 193*, 215–226.

Dubois, M., Gilles, K. A., Hamilton, J. K., Rebers, A. P., & Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry, 28*, 350–356.

Ellis, H. S., & Ring, S. G. (1985). A study of some factors influencing amylose gelation. *Carbohydrate Polymers, 5*, 201–213.

Gidley, M. J., & Bulpin, P. V. (1989). Aggregation of amylose in aqueous systems: the effect of chain length on phase behavior and aggregation kinetics. *Macromolecules, 22*, 341–346.

Gilbert, G. A., & Spragg, S. P. (1964). Iodimetric determination of amylose. In R. L. Whistler (Ed.), *Method in carbohydrate chemistry* (4th ed., pp. 168–169). New York: Academic Press.

Hizukuri, S. (1984). Estimation of the distribution of molecular weight for amylose by the low-angle laser-light, scattering technique combined with high-performance gel chromatography. *Carbohydrate Research, 134*, 1–10.

Hizukuri, S. (1996). Starch: analytical aspects. In A. C. Eliasson (Ed.), *Carbohydrates in food* (pp. 362–393). New York: Marcel Dekker.

Hizukuri, S., Kaneko, T., & Takeda, Y. (1983). Measurement of the chain length of amylopectin and its relevance to the origin of crystalline polymorphism of starch granules. *Biochimica Et Biophysica Acta, 760*, 188–191.

Hizukuri, S., & Osaki, S. (1978). A rapid smith-degradation for the determination of non-reducing terminal residues of (1 → 4)-α-D-glucans. *Carbohydrate Research, 63*, 261–264.

Hizukuri, S., Takeda, Y., Shitaozono, T., Abe, J., Ohtakara, A., Takeda, C., et al. (1988). Structure and properties of water chestnut (*Trapa natans* L var bispinosa Makino) starch. *Starch, 40*, 165–171.

Hizukuri, S., Takeda, Y., Yasuda, M., & Suzuki, A. (1981). Multi-branched nature of amylose and the action of debranching enzymes. *Carbohydrate Research, 94*, 205–213.

Kodama, M., Noda, H., & Kamata, T. (1978). Conformation of amylose in water. I. Light-scattering and sedimentation-equilibrium measurements. *Biopolymers, 17*, 985–1002.

Lu, T. J., Chen, J. C., Lin, C. L., & Chang, Y. H. (2005). Properties of starches from cocoyam (*Xanthosoma sagittifolium*) tubers planted in different seasons. *Food Chemistry, 91*, 69–77.

Miles, M. J., Morris, V. J., Orford, P. D., & Ring, S. G. (1985). The roles of amylose and amylopectin in the gelation and retrogradation of starch. *Carbohydrate Research, 135*, 271–281.

Miles, M. J., Morris, V. J., & Ring, S. G. (1985). Gelation of amylose. *Carbohydrate Research, 135*, 257–269.

Mua, J. P., & Jackson, D. S. (1997). Relationships between functional attributes and molecular structures of amylose and amylopectin fractions from corn starch. *Journal of Agricultural and Food Chemistry, 45*, 3848–3854.

Pfannemüller, B., Mayerhöfer, H., & Schultz, R. C. (1971). Conformation of amylose in aqueous solution: optical rotatory dispersion and circular dichroism of amylose–iodine complexes and dependence on chain length of retrogradation of amylose. *Biopolymers, 10*, 243–261.

Schoch, T. J. (1964). Iodimetric determination of amylose. In R. L. Whistler (Ed.), *Method in carbohydrate chemistry* (4th ed., pp. 157–160). New York: Academic Press.

Suzuki, A., Hizukuri, S., & Takeda, Y. (1981). Physicochemical studies of kuzu starch. *Cereal Chemistry, 58*, 286–290.

Swinkels, J. J. M. (1985). Composition and properties of commercial native starches. *Starch, 37*(1), 1–5.

Takeda, Y., & Hizukuri, S. (1987). Structures of branched molecules of amyloses of various origins, and molar fractions of branched and unbranched molecules. *Carbohydrate Research, 165*, 139–145.

Takeda, Y., Hizukuri, S., & Juliano, B. O. (1986). Purification and structure of amylose from rice starch. *Carbohydrate Research, 148*, 299–308.

Takeda, Y., Maruta, N., & Hizukuri, S. (1992). Structures of amylose subfractions with different molecular sizes. *Carbohydrate Research, 226*, 279–285.

Takeda, Y., Shirasaka, K., & Hizukuri, S. (1984). Examination of the purity and structure of amylose by gel permeation chromatography. *Carbohydrate Research, 132*, 83–92.

Takeda, C., Takeda, Y., & Hizukuri, S. (1983). Physicochemical properties of lily starch. *Cereal Chemistry, 60*, 212–216.

Thitipraphunkul, K., Uttapap, D., Piyachomkwan, K., & Takeda, Y. (2003). A comparative study of edible canna (*Canna edulis*) starch from different cultivars. Part II. Molecular structure of amylose and amylopectin. *Carbohydrate Polymers, 54*, 489–498.

Yang, C. C., Lai, H. M., & Lii, C. Y. (1984). The modified alkaline steeping method for the isolation of rice starch. *Food Science (Taiwan), 11*, 158–162.