Viscoelastic Properties of Rice-Flour Pastes and Their Relationship to Amylose Content and Rice Quality

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ABSTRACT

The dynamic viscoelastic behavior of nonwaxy rice-flour pastes (15 varieties) was studied using oscillatory and stress-relaxation tests. When cooked to 95°C, the storage (elastic, G'), loss (viscous, G''), and relaxation (G) moduli of the pastes, as well as their relaxation time ($T_{0.75}$) increased with rise in paste concentration. The increase was greater in high-amylose equivalent (AE) varieties than in low-AE varieties. In pastes of high concentration (13%), G', G'', G, and $T_{0.75}$ were highest in type I (high AE) and lowest in type VII (low AE) rice. Upon continued cooking at 95°C for 60 min, G', G, and $T_{0.75}$ increased in dilute pastes (7%), but decreased in concentrated (>7%) pastes. Again, these changes were greater in low AE rice. G', G'', and G were highly correlated with total AE, as well as with water-insoluble AE content of rice, but not with the water-soluble AE content. The data suggested that starch granules of high AE rice were relatively rigid, elastic, and strong, while those of low AE rice were soft, inelastic, and weak, and broke down more easily. This difference in starch-granule rigidity was probably the cause of varietal difference in cooked rice texture. Relaxation time, as well as G''/G' (tan δ) data, suggested that concentrated rice pastes were more predominantly elastic than viscous.

Results of previous studies (Chinnaswamy and Bhattacharya 1986; Takeda et al 1987, 1989; Hizukuri et al 1989; Radhika Reddy et al 1993) suggested that cooked rice texture was largely determined by the fine structure of amylopectin molecules. The chain-length distribution and the external-internal disposition of the long chains especially, seemed to be the cause of variation in rice quality. How does amylopectin structure affect rice quality? This study of the rheology of rice-flour pastes was conducted to provide an answer.

Sandhya Rani and Bhattacharya (1985, 1989, and *in press*) studied the apparent viscosity of rice-flour pastes under different paste concentrations, temperatures, and cooking times. They observed that paste viscosity was inversely proportional to the amylose equivalent content of rice at low (<7%) paste concentrations but directly proportional at high (>7%) paste concentrations. [As explained by Takeda et al (1987, 1989) and Radhika Reddy et al (1993), amylose determined by iodine reaction of starch is always an overestimation. Referred to here as amylose equivalent (AE), it comprises the contribution of true amylose (or soluble AE) and amylopectin (or insoluble AE).] Also, paste viscosity increased with cooking time at 95°C for low paste concentrations, but it decreased with cooking time at high concentrations. Both these effects were greater in low AE rice than in high AE rice. From this, we concluded that starch granules in high AE rice were probably strong and rigid; they resisted swelling as well as disintegration (giving low viscosity in dilute pastes but high viscosity in concentrated pastes). Low AE starch granules were probably weak and fragile; they swelled and disintegrated easily (giving high viscosity in dilute pastes but low viscosity in concentrated pastes). These conclusions were supported by microscopic observations of Sandhya Rani and Bhattacharya (*in press*).

Clearly, a rheological approach seemed to be a promising tool for the study of rice quality. The rheological response of a system can be resolved into an elastic and a viscous component, the complete characterization of which provides a better idea of the behavior of starch gels (Eliasson 1986). Therefore, an attempt was made here to characterize various rice types for the viscoelastic properties of their pastes, using oscillatory and stress-relaxation tests.

MATERIALS AND METHODS

Rice

Rice samples and milling and grinding procedures were the same as those described previously (Radhika Reddy et al 1993). Fifteen varieties with a graded content of total and insoluble AE were selected for the study and classified according to the scheme of Bhattacharya et al (1982) for convenience and identification. Waxy varieties were excluded from the study; Sandhya Rani and Bhattacharya (*in press*) observed that starch granules of these varieties disintegrated extensively even before the paste reached 95°C. Total (Juliano 1971, Sowbhagya and Bhattacharya 1979) and hot-water-insoluble AE (Shanthy et al 1980) of the varieties were determined as previously described. Varieties, classifications, and AE content are shown in Table I.

Preparation of Rice-Flour Paste

Rice flour was pasted under a controlled heating regime in a Brabender viscograph using a microbowl attachment as previously reported (Sandhya Rani and Bhattacharya 1985). Briefly: ~120 g of slurry in the inner microbowl was heated by the water

TABLE I
Experimental Rice Samples and Their Characteristics

Type	Variety	Amylose Equivalent Content (% db)[a] Total	Insoluble
I	Jaya	27.7 ± 0.4	16.9 ± 0.1
	T (N) 1	28.6 ± 0.2	17.8 ± 0.3
	IR8	28.1 ± 0	16.8 ± 0.2
II	Co32	28.1 ± 0.1	14.5 ± 0.2
	S701	28.0 ± 0.2	13.8 ± 0.1
III	Jhona	28.9 ± 0.2	12.8 ± 0.1
	Madhu	28.9 ± 0.1	14.1 ± 0.1
	S317	29.4 ± 0.1	13.8 ± 0.3
IV	Basmati370	25.4 ± 0.2	10.9 ± 0.1
	Br9	24.0 ± 0	10.4 ± 0.2
V	Intan	26.0 ± 0.2	10.5 ± 0.4
VI	Rojolele	26.2 ± 0.1	11.3 ± 0.1
	Sukhanandi	24.8 ± 0.5	9.6 ± 0.3
VII	Changlei	19.7 ± 0.4	10.3 ± 0.1
	T65	19.7 ± 0.1	8.5 ± 0.2

[a] Mean of duplicate values ± standard deviation.

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bath in the outer bowl at a controlled rate from room temperature to 95°C within 40 min (designated as 0 min of holding). After quickly removing 50 g of the paste (sample 95°–0 min), the remaining portion was cooked at the same temperature for another hour (sample 95°–60 min).

The paste was stored in a 100-ml beaker covered with a wet cotton-padded petri dish and was cooled in an air-conditioned room (25°C) for 1 hr until used. All dilute pastes were pourable when placed on the rheometer plate. But most concentrated (>13%) pastes were not pourable after cooking.

Measuring Viscoelastic Properties

A Bohlin VOR rheometer (Bohlin Rheologi, Lund, Sweden) was used. The general methods used by Eliasson and Bohlin (1982), Mita and Bohlin (1983), and Bohlin and Eliasson (1986) were followed, but preliminary experiments were made to select the best test conditions. A cone-plate (30 mm diameter, cone angle 2.5°) system and a torque element of 90 cmg were chosen after much experimentation. The excess paste expelled upon lowering of the cone was trimmed off, and a thin film of silicone oil was applied to the edge to prevent drying. All measurements were done at 25° ± 0.5°C. A strain value of 0.04 was chosen because preliminary tests showed that the viscoelastic response became nonlinear beyond a strain value of 0.06–0.07.

In the oscillation experiments, measurements were made at frequencies of 0.001, 0.02, and 1 Hz, with appropriate delay periods for each. At higher frequencies, the slope of the frequency sweep curve showed a steep change; the preset amplitude and strain were also altered.

In stress-relaxation experiments, the paste was allowed to rest for 30 min after loading to equilibrate before starting the test; otherwise the reproducibility was poor. Time to reach the required strain was 0.2 sec and measurements were made at 0.01–1,000 sec. The initial relaxation modulus (G_0), as well as the modulus after various times (G_t) were noted. After completion of an experiment, the relative G_t/G_0 versus the natural logarithm of time was recorded.

Relaxation time (at a constant strain) of a Maxwell body is defined as the time needed for the stress to relax to 1/eth of its initial value. For complex materials, where the stress does not relax fully, researchers define the parameter arbitrarily as the time at which the stress relaxes to 50% of its original value ($T_{0.5}$). In the present case, the G values were more than 50% of the initial values, even after 1,000 sec of relaxation. $T_{0.75}$ (i.e., the time in seconds at which stress relaxed to 75% of the initial value) was arbitrarily defined as the relaxation time.

Reproducibility and the Effect of Paste Aging

Repeated observations during the long preliminary experimentation indicated that reproducibility of the measurements was very good. For example: six to nine replicates of Jaya rice, 13% paste, for 95°C–0 min and 95°C–60 min, respectively, gave the following results: G_0 2,435 ± 108 and 2,420 ± 66 Pa; G' 1,724 ± 42 and 1,629 ± 132 Pa. This gave a coefficient of variation of 4.4, 2.7, 2.4, and 8.1%, respectively. Most data reported here are therefore of single determinations.

Because the work protocol involved cooking the paste in a Brabender viscograph for two time periods, followed by measurement in the relaxation (1,000 sec) and oscillation modes, measurement of all viscoelastic parameters soon after pasting was next to impossible. Similarly, it was difficult to maintain a constant time interval between pasting and measuring. Preliminary data suggested that cooling the paste in the air-conditioned room for 1 hr was necessary for reproducible results. This matter was extensively tested during the preliminary experimentation, and variation in the storage time at 25°C between 1 and 10 hr seemed to have no measurable effect on the rheological parameters of the pastes. Four to six replicates of one sample (Co32, 12% paste, 95°–0 min), measured on the day of cooking and again the next day, gave the following results: G_0 1,510 ± 65 and 1,553 ± 90 Pa. This gave a coefficient of variation of 4.3 and 5.8%, respectively. Therefore, all measurements were made between 1 and 10 hr after pasting; the majority were made between 1 and 6 hr. Recently, Biliaderis and Zawistowski (1990) observed that wheat starch paste at concentrations <20% gave insignificant increase in G' values for storage up to 24 hr at 25°C.

RESULTS

Two sets of experiments were done: one to study the effect of paste concentration (with three selected varieties), and the other to study the effect of rice variety (for all 15 varieties) at a constant concentration of 13% (wb).

Effect of Paste Concentration

Three rice varieties were used: high (type I, Jaya), intermediate (type IV, Br9), and low (type VII, T65) AE content. Effects of paste concentration on storage (G') and loss (G'') moduli at 0.02 Hz, as well as on relaxation modulus (G) and relaxation time ($T_{0.75}$) are shown in Tables II and III. A representative pattern for the values of G' is shown in Figure 1.

G', G, and $T_{0.75}$ all increased similarly with increased concentration for both 95°–0 min and 95°–60 min pastes. Clearly, the elasticity of the pastes increased as the concentration increased, showing the effect of increasing filler content in a given volume. Significantly, the increase in these parameters with increase in paste concentration was the highest in Jaya, followed by Br9 and T65. Clearly, high AE rice starch granules were harder and more rigid than intermediate AE granules, which, in turn, were more rigid than low AE starch granules. G'' also increased with increasing paste concentration (Table II). But its value was low and the pattern was somewhat erratic.

TABLE II
Effect of Concentration on Rheological Parameters of High, Intermediate, and Low Amylose Equivalent Rice Pastes[a,b]

Type	Variety	Concentration (% wb)	Storage Modulus, G' (Pa) 0 min	Storage Modulus, G' (Pa) 60 min	Loss Modulus, G'' (Pa) 0 min	Loss Modulus, G'' (Pa) 60 min	Tan δ 0 min	Tan δ 60 min
I	Jaya	7	20	224	13	28	0.63	0.13
		10	871	826	109	68	0.12	0.08
		13	1,840	1,710	203	124	0.11	0.07
		15	2,560	2,410	205	180	0.08	0.08
IV	Br 9	7	42	121	13	11	0.30	0.10
		10	456	223	63	30	0.14	0.14
		13	1,190	561	143	43	0.12	0.08
		16	1,720	973	112	62	0.06	0.06
VII	T65	9	241	114	70	12	0.29	0.11
		13	587	231	49	29	0.08	0.13
		16	1,050	278	87	38	0.08	0.14
		19	1,010	318	346	51	0.19	0.16

[a]Oscillatory test at 0.02 Hz.
[b]Pastes cooked at 95° for 0 and 60 min.

Effect of Varietal Difference

Effect of varietal difference at any given paste concentration can be perceived from data in Tables II and III and Figure 1. At higher concentrations, rice with higher AE clearly gave pastes with higher elasticity (and greater rigidity of starch granules). This conclusion was confirmed when 15 varieties with a wider representation of total as well as water-insoluble AE contents were studied at a constant, but moderately high, concentration of 13% (wb) (Tables IV and V).

The G'' values were low and somewhat erratic. The type-to-type gradation was not as clear-cut for relaxation time ($T_{0.75}$), but the overall trend was similar: types I and II showed higher relaxation time and type VII the least. Paste and granule elasticity were proportional to the AE content.

TABLE III
Effect of Concentration on Relaxation Modulus (G_o) and Relaxation Time ($T_{0.75}$) of Rice-Flour Pastes[a]

Type	Variety	Concentration (% wb)	G_o (Pa) 0 min	G_o (Pa) 60 min	$T_{0.75}$ (sec) 0 min	$T_{0.75}$ (sec) 60 min
I	Jaya	7	62	244	0.8	2.0
		10	1,360	1,260	5.3	13.0
		13	2,610	2,480	51.7	78.7
		15	3,190	3,090	161.3	152.0
IV	Br 9	7	112	161	0.5	3.3
		10	568	350	8.1	1.7
		13	1,740	972	22.4	5.0
		16	1,840	1,120	46.0	5.0
VII	T65	9	430	188	4.0	2.3
		13	827	302	10.3	0.5
		16	1,420	367	16.6	0.4
		19	1,810	492	30.2	0.4

[a]Cooked at 95°C.

Effect of Continued Heating (95°-60 min)

When the rice pastes were cooked at 95°C for an additional 60 min, the effect varied with the paste concentration and content of AE. At moderate-to-high concentrations, G', G, and $T_{0.75}$ tended to decrease in samples at 95°-60 min, as compared to those at 95°-0 min, in all three varieties (Fig. 1, Tables II and III), but the decrease was clearly greater for low AE varieties. At a low (7%) concentration, G', G, and $T_{0.75}$ tended to increase after continued heating (95°-60 min) as compared to 95°-0 min. Values of G'' were again erratic but similar. The results of the constant-concentration (13%) study with 15 varieties were also similar (Tables IV and V), except that the trend of $T_{0.75}$ data was not as clear-cut.

One can surmise that the granules swelled rather freely in the

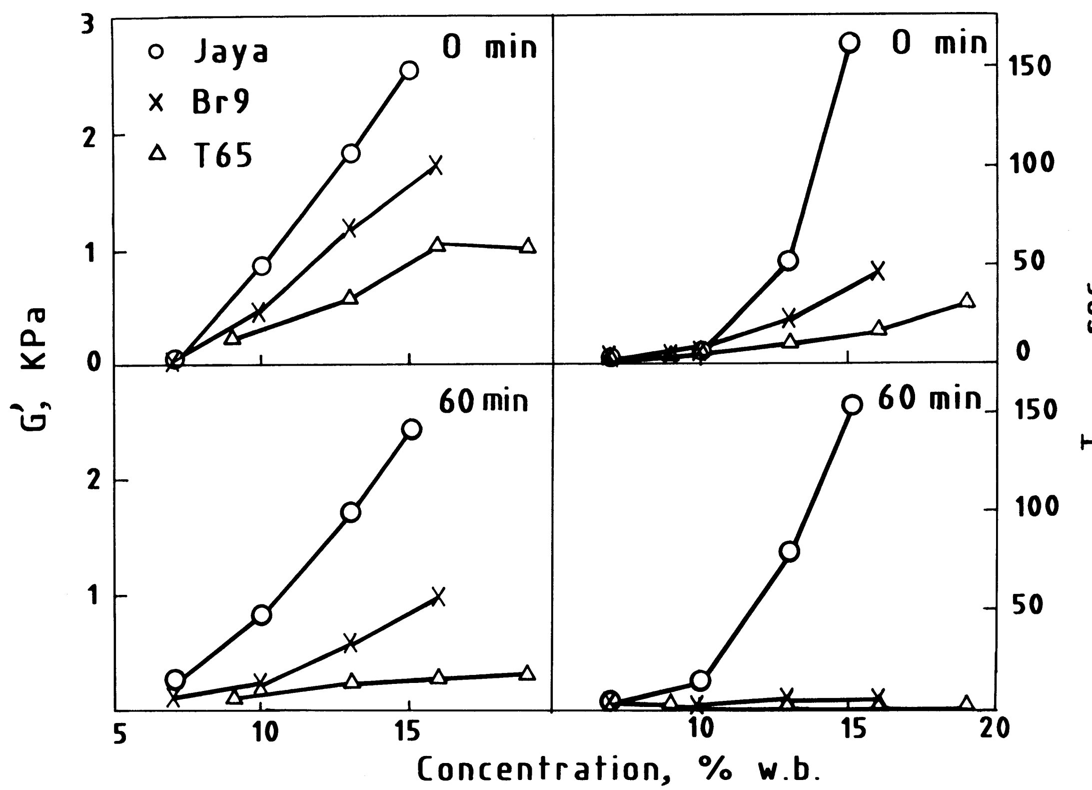

Fig. 1. Effect of paste concentration on storage modulus (G') and relaxation time ($T_{0.75}$) of three rice varieties: Jaya (high amylose equivalent [AE] content); Br9 (intermediate AE); T65 (low AE).

TABLE IV
Effect of Varietal Difference on Rheological Parameters of Rice Pastes Under Oscillatory Test[a,b]

Type	Number of Varieties	Storage Modulus, G' (Pa) 0 min	Storage Modulus, G' (Pa) 60 min	Loss Modulus, G'' (Pa) 0 min	Loss Modulus, G'' (Pa) 60 min	Tan δ 0 min	Tan δ 60 min
I	3	1,830 ± 110	1,727 ± 185	143 ± 17	117 ± 13	0.08 ± 0.01	0.07 ± 0.01
II	2	1,785 ± 55	1,355 ± 55	135 ± 9	95 ± 4	0.08 ± 0.01	0.07 ± 0
III	3	1,740 ± 29	1,083 ± 58	169 ± 3	102 ± 6	0.10 ± 0.01	0.10 ± 0.01
IV	2	1,340 ± 30	628 ± 33	126 ± 2	56 ± 8	0.10 ± 0.01	0.09 ± 0.01
V	1	1,220	596	98	48	0.08	0.08
VI	2	1,315 ± 55	549 ± 9	84 ± 7	39 ± 1	0.07 ± 0.01	0.07 ± 0
VII	2	728 ± 17	213 ± 28	75 ± 12	30 ± 8	0.11 ± 0.02	0.14 ± 0.02

[a]Frequency 0.02 Hz. Pastes (13% concentration, wb) were cooked at 95°C for 0 and 60 min.
[b]Values reported are means ± standard deviation.

TABLE V
Effect of Varietal Difference on Relaxation Parameters of Rice-Flour Pastes[a,b]

Type	Number of Varieties	Relaxation Modulus, G_o (Pa)		Relaxation Time, $T_{0.75}$ (sec)		$G_{1,000}/G_o$[c] (%)	
		0 min	60 min	0 min	60 min	0 min	60 min
I	3	2,277 ± 246	2,150 ± 251	61.5	100.0	66 ± 5	67 ± 7
II	2	2,100 ± 10	1,315 ± 15	61.5	18.3	70 ± 2	58 ± 3
III	3	2,113 ± 25	1,443 ± 59	33.5	20.6	64 ± 2	60 ± 2
IV	2	1,720 ± 100	967 ± 93	31.6	29.7	61 ± 4	62 ± 2
V	1	1,520	788	23.3	20.6	59	62
VI	2	1,565 ± 35	697 ± 97	33.5	20.6	62 ± 0	62 ± 1
VII	2	909 ± 55	307 ± 3	11.2	1.3	52 ± 1	37 ± 7

[a] Pastes (13% concentration, wb) were cooked at 95° for 0 and 60 min.
[b] Values reported are means ± standard deviation.
[c] Relaxation modulus after 1,000 sec as a percent of initial modulus.

relatively free space of a dilute paste, so the paste became thicker and more elastic. The greater swelling of the low AE granules implied that they were less rigid and weaker in organization than high AE starch granules. The decrease in elasticity in the concentrated pastes indicated softening or partial or extensive disintegration of the starch granules as a result of overcrowding and shear. The greater decrease in these parameters for starch granules of low AE varieties again point to such granules being weaker and more fragile than those in high AE rice.

These results were very similar to those reported by Sandhya Rani and Bhattacharya (1989 and *in press*) of an increase or decrease in apparent viscosity of rice-flour pastes due to continued cooking. Clearly, the AE content affected the swelling as well as stability of the starch granules to continued heating and shear. In agreement with this qualitative trend, various rheological parameters were very significantly correlated with both the total and insoluble AE content of the rice samples (Table VI). Soluble AE was not correlated with any parameter.

G''/G' (tan δ)

The G''/G' ratio generally decreased with increasing paste concentration in 0-min and 60-min cooked samples (Table II). When heated at high concentrations, starch granules may have swelled and filled up the volume and absorbed all the water, and the paste became predominantly elastic (solid). At low concentrations, some water or the dispersing phase remained, giving the paste a predominantly viscous behavior.

The decrease in the ratio with increasing concentration was highest in Jaya and lowest in T65; Br9 was intermediate, which supports the trend seen earlier.

The G''/G' ratio at the same high concentration (13%) in 15 varieties remained nearly constant among all the rice types, except type VII (Table IV). This suggested that the relative proportions of the elastic and viscous elements were more or less constant in all the pastes. The ratio was also very low, indicating that the pastes at this high concentration showed predominantly elastic behavior. Only the low AE varieties gave slightly more viscous paste, possibly because of extensive granule disintegration.

Residual Stress

All the samples showed a fairly large amount of residual stress, even after 1,000 sec (over 16.5 min) of relaxation (Table V, Fig. 2).

TABLE VI
Correlation of Rheological Parameters of 13% (wb) Rice Paste to Amylose Equivalent (AE) Content of Rice[a]

	r Value (×1,000)[b]					
	G'-0	G''-0	G-0	G'-60	G''-60	G-60
Total AE	928	877	891	771	845	779
Insoluble AE	847	762	829	957	926	944
Soluble AE	206	246	176	−168	−19	−139

[a] G'-0, G''-0, G-0 = G', G'', and G value of 95°-0 min paste, G'-60, G''-60, G-60 = G', G'', and G value of 95°-60 min paste.
[b] All r values of the first two rows were significant at 0.1% level (n = 15). Those of the third row were not significant.

Moreover, the $G_{1,000}/G_o$ values of all the 13% pastes tended to fall in a rather narrow range, except perhaps the two extreme types. This is somewhat similar to the rather constant G''/G' values of these pastes (Table IV).

The partial relaxation and the high residual stress again show that concentrated rice-flour pastes were more predominantly elastic than viscous.

This data, as well as the shape of the curves in Figure 2, suggest that a concentrated rice-flour paste can be visualized, albeit in a simplified way, as a Burger's model: a Kelvin element (a spring and a dash pot coupled in parallel) joined in series with a Maxwell element (a dash pot and a spring coupled in series). Stress relaxation, caused in a simple Maxwell element due to displacement in the dash pot, is obstructed by the spring component in the Kelvin element.

DISCUSSION

Our studies (Bhattacharya et al 1978, 1982; Sowbhagya et al 1987) showed that the hot-water-insoluble AE was a key deter-

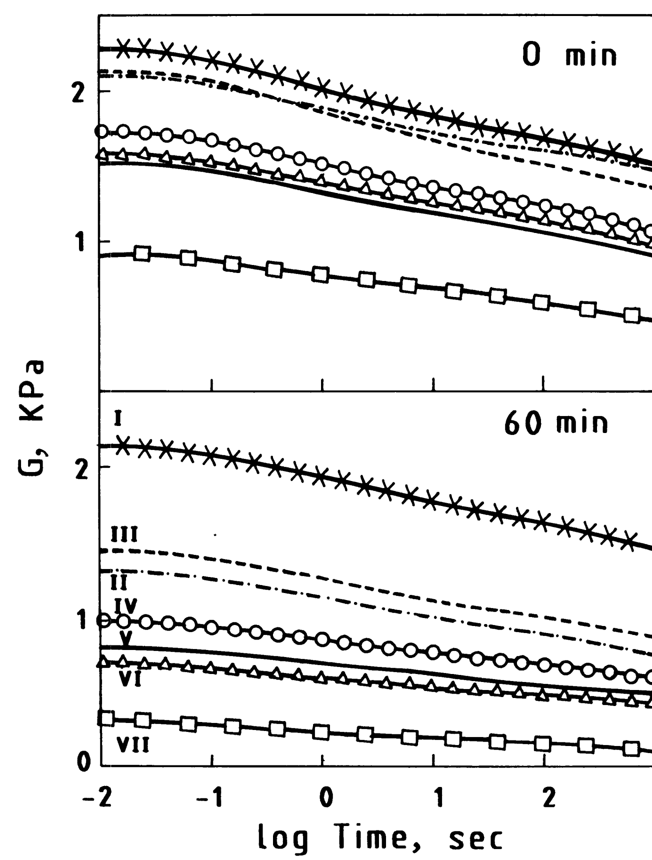

Fig. 2. Stress-relaxation curves of rice pastes, 13% concentration (wb), cooked at 95°C for 0 min and 60 min.

minant of rice texture. Also, we (Radhika Reddy et al 1993) showed that the insoluble AE was an index of the iodine-binding amylopectin component of starch, depending upon its long-B chains and their external/internal disposition, while the soluble AE was an index of the true amylose. These and other recent studies (Takeda et al 1987, 1989; Hizukuri et al 1989) strongly suggested that chain-length distribution and external/internal disposition of the long chains of amylopectin were indeed largely, if not solely, responsible for the varietal difference in rice texture.

Rheological studies (Sandhya Rani and Bhattacharya 1985, 1989 and *in press*) have shown that starch granules of high AE rice were relatively strong, rigid, and elastic, while those of waxy and low AE rice were relatively weak, inelastic, and fragile. This fact was clearly related to the cooked rice texture. It seemed varieties with strong and rigid starch granules maintained their granule integrity even upon cooking and gave firm, dry, and nonsticky cooked rice, just as they gave a strong, elastic paste. Varieties with weak granules, on the other hand, had extensive granule disintegration when cooked and gave moist, sticky, soft cooked rice, just as they gave a thin, weak paste.

With the newer knowledge of rice AE, we may ask: How do amylopectins with more long and external B chains render the starch granule strong and elastic?

Several authors (Hizukuri et al 1983, French 1984, Hizukuri 1985, Eliasson et al 1987, Zobel 1988, Manners 1989) have demonstrated that it is primarily the amylopectin component that contributes to the crystallinity of the starch granule. Therefore, it may follow that more insoluble AE (amylopectin with more long and external B chains), by virtue of strong intermolecular interaction, rendered the starch granule strong and elastic. Low insoluble AE varieties, with more short chains and an inability to mutually interact, yielded only weak starch granules. Soluble AE, as an index of the true amylose of starch, correlated neither with rheological parameters of rice nor with cooked rice texture.

Thus, a relative abundance and external orientation of long-B chains of amylopectin, through their effect on starch-granule organization, seemed to be the fundamental factor in determining the varietal difference in cooked rice texture.

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