

# Rheological Properties of Sago Starch

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The swelling characteristics and rheological properties of 11 sago starches have been studied and compared to a number of commonly used starches. The swelling power of the sago starches were all very similar except for two samples which were known to contain a lower molecular mass amylose component. The swelling and solubility for these two samples were higher than the rest. The values for sago starches were similar to that for potato and tapioca starch, but higher than maize and pea starch. The storage modulus,  $G'$ , of the sago starches as studied by small deformation oscillation measurements showed a rapid initial increase in  $G'$  followed by the development of a pseudoplateau. The dependence of  $G'$  on the molecular mass of the amylose followed the relationship  $G' \propto M^{-0.4}$ . Gels were formed only at sago starch concentrations of  $>3.5\%$ , corresponding to an amylose concentration of  $\sim 1.0\%$  and varied with concentration according to the relationship  $G' \propto C^{1.8}$  and  $G' \propto C^{2.5}$  for high and low amylose molecular mass samples, respectively. Gel strength (GS) measurements also confirmed that the minimum concentration for gelation was  $\sim 3.5\%$  and that  $GS \propto C^{2.0}$ . Sago starches showed good freeze–thaw stability compared to other starch types.

**Keywords:** *Sago starch; swelling; solubility; gelatinization; gelation; rheology; freeze–thaw stability*

## INTRODUCTION

Starch is composed of amylose, which is a linear molecule consisting of (1→4)-linked  $\alpha$ -D-glucopyranose units and amylopectin which is a highly branched molecule consisting of short chains of (1→4)-linked  $\alpha$ -D-glucose with (1→6)- $\alpha$ -linked branches. Although amylose accounts for only 20–32% of the total in the case of many common starches (Doublie and Choplin, 1989) it makes a major contribution to the overall properties. Starch occurs in the form of granules which are insoluble in cold water and when aqueous suspensions are heated above a certain temperature, swelling and dissolution occurs. This process is, perhaps inappropriately, referred to as gelatinization. The degree of swelling, granule disintegration, and release of amylose depends on various factors such as type of starch, starch concentration, temperature, presence of solute, and also shear or agitation during heating (Biliaderis, 1991). If the concentration of the starch paste is high enough (normally  $> 6\%$ ), on cooling opaque gels are formed as a consequence of the aggregation of amylose chains forming ordered junction zones based on amylose double helices (Clark and Ross-Murphy, 1987). Starch gels undergo further crystallization during storage resulting in an increase in the rigidity and syneresis usually occurs (Biliaderis, 1991). This process which is commonly known as retrogradation depends on various factors such as the botanical origin of the starch, the concentration of the starch paste, the heating and cooling conditions, pH, and the presence of solutes such as lipids, salts, and sugars (Swinkels, 1985). Starch gels can be regarded as composites where the swollen granules (amylopectin) are embedded into a continuous matrix of aggregated amylose molecules (Ott and Hester, 1965; Ring, 1985; Miles et al., 1985b; Carnali and Zhou, 1996).

Sago starch is isolated from sago palm, *Metroxylon* spp. which is distributed throughout Southeast Asia. About  $3 \times 10^5$  tonnes of sago starch are produced annually in Malaysia. In a previous paper, we reported the physicochemical properties of sago starch obtained from Malaysia, Thailand, and Indonesia (Ahmad et al., 1998). The amylose contents were  $27 \pm 3\%$  and the average particle size was around  $30 \mu\text{m}$ . The gelatinization temperature,  $T_g$ , was close to  $70^\circ\text{C}$  which is high compared to maize ( $68^\circ\text{C}$ ), potato ( $63^\circ\text{C}$ ), or tapioca starch ( $66^\circ\text{C}$ ). The gelatinization enthalpy was around  $15\text{--}17 \text{ J/g}$ . The starch was found to have a C-type X-ray diffraction pattern consisting of about 65% A-type and 35% B-type crystalline forms. Very few reports appear in the scientific literature regarding the rheological properties of sago starch. The only study of which we are aware concerned with the mechanical spectroscopy of sago starch is that of Akuzawa et al. (1995). These workers compared the dynamic viscoelasticity and stress relaxation characteristics of 4% dispersions of starch from a wide variety of sources with their spinnability.

The purpose of the work presented here was to determine the swelling characteristics and rheological properties of a range of sago starches obtained from different sources.

## MATERIALS AND METHODS

**Materials.** Eleven sago samples isolated from *Metroxylon sago* ROTTB. have been used in this study. Sago 1 to sago 9 were obtained from various manufacturers in Sarawak, Malaysia, sago 10 was obtained from Pekan Baru, Riau, Indonesia, and sago 11 was obtained from Bangkok, Thailand. Sago 2 and sago 5 were food grade while the other samples were industrial grade. All the samples were used as provided without any further treatment. The percentage amylose and the molecular mass of the amylose fractions of the sago starches used have been reported previously and details are given in Table 1. Pea, potato, tapioca, and maize starch were

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**Table 1. Percentage Amylose and Amylose Viscosity Average Molecular Mass in Sage Starch Samples<sup>a</sup>**

| starch              | amylose (%) | $M_v \times 10^{-6}$  |
|---------------------|-------------|-----------------------|
| sago 1              | 28 (±1)     | 1.4                   |
| sago 2 <sup>b</sup> | 30 (±1)     | 0.79                  |
| sago 3              | 26 (±1)     | 1.7                   |
| sago 4              | 25 (±2)     | 1.8                   |
| sago 5              | 26 (±1)     | 1.9                   |
| sago 6              | 25 (±2)     | 1.7                   |
| sago 7              | 28 (±1)     | 1.8                   |
| sago 8 <sup>b</sup> | 26 (±2)     | 0.88                  |
| sago 9              | 28 (±2)     | 1.6                   |
| sago 10             | 24 (±1)     | 2.0                   |
| sago 11             | 27 (±1)     | 1.8                   |
| potato              | 22 (±2)     | 0.14–3.6<br>(av 1.0)  |
| tapioca             | 18 (±1)     | 0.1–3.7<br>(av 1.1)   |
| pea                 | 40 (±2)     | 0.21                  |
| maize               | 28 (±1)     | 0.06–2.1<br>(av 0.42) |

<sup>a</sup> For amylose and molecular mass determination refer to Ahmad et al. (1998); weight average molecular masses for potato, tapioca, and maize are from Hizukuri et al. (1984) and for pea, from Sosulski et al. (1997). <sup>b</sup> The initial amylose molecular mass for sago 2 and sago 8 when the samples were first obtained was  $1.2 \times 10^6$  and  $1.4 \times 10^6$ , respectively. The molecular mass reduced during storage due to acidic degradation.

a gift from Cerestar, Trafford Park, U.K., and were used as received for comparison.

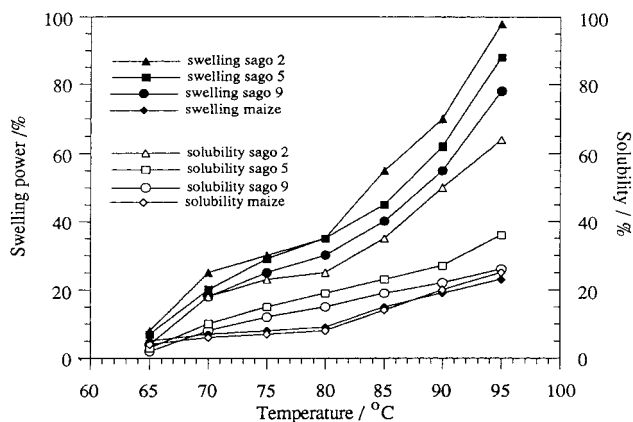
**Swelling and Solubility.** Swelling and solubility were determined according to the procedure of Schoch (1964). The concentrations used for the various starches were sago and tapioca 0.5%, potato 0.25%, and maize/pea 1%. The solubility and swelling power were obtained using the following equations:

$$\text{solubility, \%} = \frac{\text{wt of soluble starch/wt of sample, db}}{100} \times 100$$

$$\text{swelling power, \%} = \frac{\text{wt of sedimented paste}}{\text{wt of sample, db} \times (100 - \% \text{ solubles, db})} \times 100$$

**Mechanical Spectroscopy.** The storage and loss moduli ( $G'$  and  $G''$ ) and dynamic viscosity,  $\eta'$  were monitored using a Carri-Med CSL500 controlled stress rheometer (TA Instruments, Leatherhead, U.K.). Measurements were performed on 6% w/v starch samples, and  $G'$  and  $G''$  were monitored as a function of time. The concentration dependence of shear modulus was determined for sago 2 and sago 9. The starch suspensions were adjusted to pH 5.5 using dilute HCl or dilute NaOH prior to heating. All the samples were heated for 30 min at 95 °C with constant stirring (400 rpm). The beaker was reweighed, and the volume was corrected for evaporation loss on heating by the addition of hot distilled water. Hot samples were placed immediately into the rheometer measuring system (cone and plate, 4 cm diameter, 2° angle) which was equilibrated to 25 °C. All measurements were performed in the linear viscoelastic region which was determined earlier. For all the samples, measurements were made at a frequency of 1.0 Hz with an amplitude of 1 mrad over a 6 or 12 h period, and for sago 2 and sago 9, the measurements were carried out up to 68 h without disturbing the samples. A solvent trap was used to eliminate the evaporation of water. At the end of the run a frequency sweep was performed in the range between 0.1 and 10.0 Hz. All measurements were undertaken twice and agreed within ±2%. Results reported are an average of the two measurements.

**Gel Strength.** Sample preparation was similar to above. Hot samples were transferred into storage jars and cooled to 25 °C in a circulating water bath and stored at that temperature for 6 h before the measurement of the gel strength. Measurement was performed using a texture analyzer (Stevens, Leatherhead Food Research, Surrey, U.K.) using a 1 cm probe.

**Figure 1.** Effect of temperature on the swelling and solubility of sago and maize starch.**Table 2. Swelling and Solubility for Sage and Some Other Starches at 95 °C**

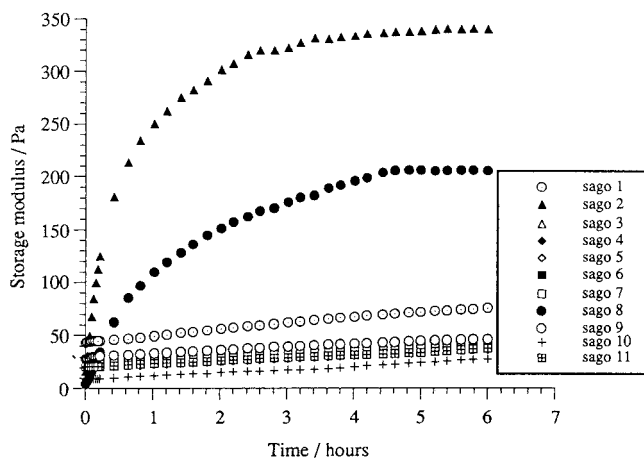
| starch  | swelling power | solubility = (%) |
|---------|----------------|------------------|
| sago 1  | 94 (±1)        | 40 (±1)          |
| sago 2  | 99 (±1)        | 64 (±2)          |
| sago 3  | 75 (±2)        | 24 (±1)          |
| sago 4  | 76 (±2)        | 32 (±1)          |
| sago 5  | 87 (±2)        | 36 (±1)          |
| sago 6  | 82 (±2)        | 36 (±1)          |
| sago 7  | 81 (±2)        | 27 (±1)          |
| sago 8  | 84 (±1)        | 55 (±1)          |
| sago 9  | 77 (±1)        | 27 (±2)          |
| sago 10 | 72 (±1)        | 32 (±2)          |
| sago 11 | 75 (±2)        | 27 (±2)          |
| potato  | 100 (±1)       | 45 (±2)          |
| tapioca | 70 (±1)        | 40 (±1)          |
| pea     | 24 (±2)        | 24 (±1)          |
| maize   | 20 (±2)        | 21 (±1)          |

Triplicate measurements were performed and agreed within ±2%. Results reported here are an average of the measurements.

**Freeze–Thaw Stability.** Sample preparation was as above. Measurements were performed on 6% w/v samples. The samples were transferred to storage jars and cooled at 25 °C in a circulating water bath. The gels obtained were subjected to cold storage at 4 °C for 16 h. To measure freeze–thaw stability the gels were frozen at –16 °C for 24 h, then thawed at 25 °C for 6 h, and then refrozen at –16 °C (Hoover and Vasanthan, 1992). Five freeze–thaw cycles were performed. The thawed samples were centrifuged at 2200 rpm for 20 min and the exuded water was determined. Triplicate measurements were performed, and the results are reported here as an average. All the measurements agreed within ±2%.

## RESULTS AND DISCUSSION

Figure 1 shows the effect of temperature on the swelling power and solubility for sago and maize starches, and Table 2 gives the swelling power and solubility at 95 °C. The results for tapioca, maize, pea, and potato starch are included for comparison. The sago starches show a two-stage swelling, similar to cereal starches (Leach et al., 1959; Rasper, 1969; Madamba et al., 1989; Delpeuch and Favier, 1980). It has been shown by others that potato and tapioca starch show a one-step swelling (Leach et al., 1959; Rasper, 1969; Madamba et al., 1989; Delpeuch and Favier, 1980). For sago starches, the first stage of swelling and solubility occurs at the gelatinization temperature, about 70 °C (Ahmad et al., 1998) while the second stage of swelling occurs at about 80 °C. The swelling and solubility increase with temperature, and the magnitude is quite similar to potato and tapioca starches but higher



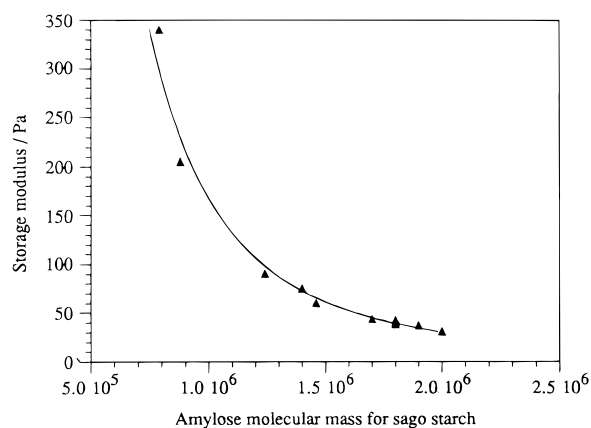
**Figure 2.** Storage modulus (frequency 1 Hz, temperature 25°C) as a function of time for 6% sago starches.

compared to maize or pea starches. The values for sago starch did not vary significantly between samples except for sago 2 and sago 8. The higher solubility for these is most probably due to the lower molecular mass of their amylose fraction (Table 1).

On the molecular level, the swelling and solubility of the starch granule is influenced by several factors including the ratio of amylose to amylopectin, molecular mass of each fraction, degree of branching, conformation, length of outer branches in amylopectin, and also on the presence of non-carbohydrate components such as proteins and lipids (Leach, 1967). Certain starches, for example, maize, rice, and sorghum show restricted swelling compared to the waxy types due to the presence of stronger and a greater number of intermolecular bonds (Leach, 1967). On the basis of the results in Table 2 and Figure 1, it is apparent that sago starch granules have a lower degree of intermolecular association compared to maize or pea and are comparable to potato and tapioca.

Figure 2 shows  $G'$  for the sago starches as a function of time. Sago samples 2 and 8 show the expected behavior (Clark et al., 1989) with an initial steep rise in  $G'$  followed by a much slower increase and attainment of a pseudoplateau value. This value remained almost constant after  $\sim 12$  h (not shown in Figure 2), and this is consistent with gel strength data reported previously for samples stored up to 7 days (Ahmad et al., 1998). The other sago samples attained values close to the  $G'$  maximum values at the start of the experiment, and the increase in  $G'$  thereafter was minimal. Sago samples 2 and 8 have higher pseudoplateau values compared to the other sago samples and are similar to maize starch. The gelation of starch is due mainly to the aggregation of amylose chains (Miles et al., 1985a; Ring et al., 1987) and will, therefore, be dependent on the starch amylose content and its molecular mass. Pfanmuller et al. (1971) reported that maximum retrogradation as monitored by light-scattering measurements was reached for amylose with an optimum molecular mass of  $1.3 \times 10^4$ , and above or below this, lower values were obtained. Whistler and Daniel (1984) also reported that retrogradation of amylose depends on molecular mass and was maximum in the molecular mass range of 6500–160 000 but was dependent on polydispersity.

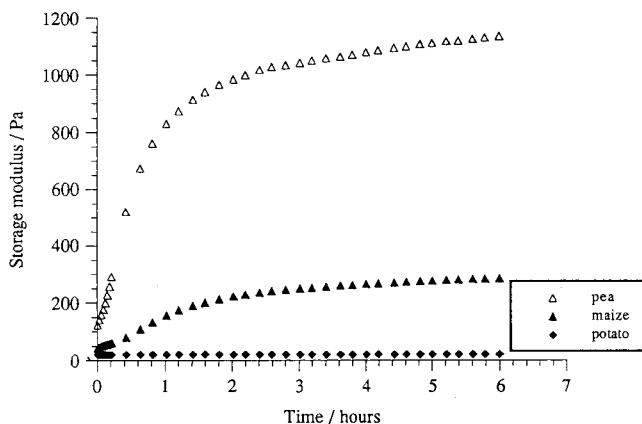
Since the amylose contents of the various sago starches are quite similar, the higher  $G'$  pseudoplateau values for sago 2 and sago 8 are most probably due to



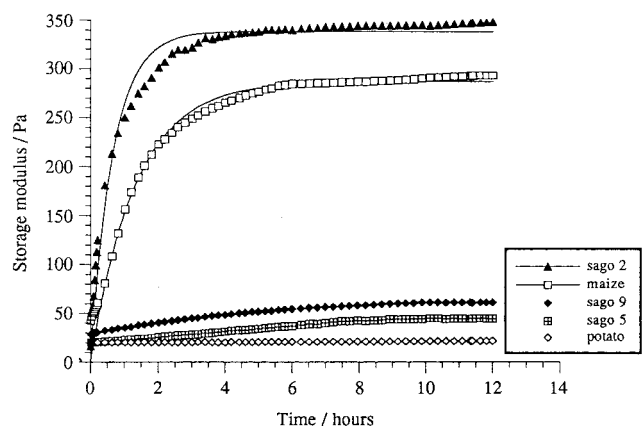
**Figure 3.** Storage modulus (frequency 1 Hz, temperature 25°C) after 6 h as a function of amylose viscosity average molecular mass,  $M_v$ , for 6% sago starches.

the lower molecular mass of their amylose fractions (Table 1). The relationship between  $G'$  (after 6 h and frequency 1 Hz) and molecular mass of the amylose fraction for the sago starch samples is illustrated in Figure 3 and clearly demonstrates that  $G'$  decreases with increasing molecular mass of the amylose ( $G' \propto M_v^{-0.4}$ ). Ellis and Ring (1985) also showed  $G'$  to be inversely proportional to molecular mass in studies using polydisperse fractions of amylose of molecular mass  $4.9 \times 10^5$  to  $1.1 \times 10^6$ . Clark et al. (1989) and Gidley (1990) found more complex behavior with pseudoplateau  $G'$  values attained after up to 33 h increasing with molecular mass for monodisperse amylose fractions of molecular mass between  $0.41 \times 10^5$  and  $1.8 \times 10^5$  but then decreasing for amylose of molecular mass  $4.2 \times 10^5$ . At longer time scales i.e.,  $> 60$  h the pseudoplateau  $G'$  values were then greater for the sample of higher molecular mass. These workers concluded that the initial rise in  $G'$  was due to the rapid establishment of a three-dimensional network structure due to amylose chain aggregation. Lower molecular mass molecules diffused faster in solution and hence the aggregation process and attainment of the pseudoplateau  $G'$  value were achieved more rapidly. The subsequent slower rise in  $G'$  was reported to be as a consequence of rearrangement of cross-links and lateral chain aggregation. Biliaderis (1992) argued that for long amylose chains, the initial formation of a few cross-links will retard the chain mobility and delay subsequent cross-linking.

For starch the situation will be even more complex. This is exemplified by the studies on mixed amylose–amylopectin systems by Parovuori et al. (1997). They found that the rate of gelation was faster than for pure amylose alone and that the plateau  $G'$  values obtained were greater for mixtures at amylose:amylopectin ratios less than  $\sim 3:5$  but less for mixtures above this ratio. This behavior may be due to the tendency for amylose and amylopectin to segregate (Kalichevsky et al., 1986; Kalichevsky and Ring, 1987; German et al., 1992) at higher concentrations and form two liquid layers each enriched in one or other of the polysaccharides. The position of the binodal separating the one-phase and two-phase regions in the phase diagram will depend on the concentration and molecular mass of both the amylose and amylopectin. In the two-phase region, amylose–amylopectin segregation, and amylose self-association will occur simultaneously, but at low temperatures association will be rapid and complete segregation will be inhibited. At higher temperatures ( $\sim 80$



**Figure 4.** Storage modulus (frequency 1 Hz, temperature 25°C) as a function of time for pea, maize, and potato starches. Starch concentration used was 6%.



**Figure 5.** Storage modulus (frequency 1 Hz, temperature 25°C) as a function of time for sago 2, sago 5, sago 9, maize, and potato starches. Starch concentration used was 6%. Symbols in the figure represent the experimental data points while the solid lines for sago 2 and maize starch represent the calculated values using a first-order rate equation.

°C) the reverse will be true (Kalichevsky et al., 1986; Kalichevsky and Ring, 1987; German et al., 1992). Self-aggregation of amylopectin will also occur over longer time scales.

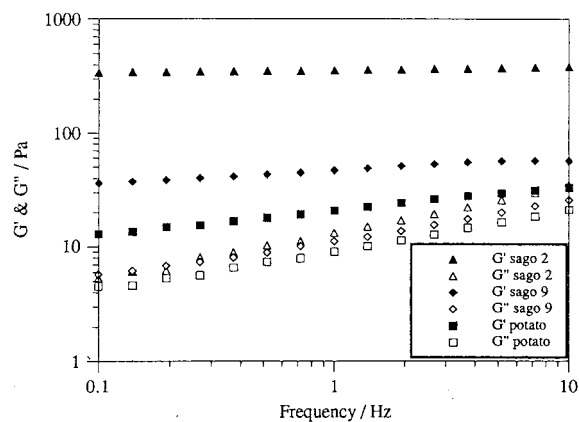
Figure 4 shows the development of  $G'$  with time for other starch types. For pea starch the higher value is probably due to its much higher amylose content and possibly lower amylose molecular mass while the lower value for potato starch is due to the lower amylose content and possibly higher amylose molecular mass.

Figure 5 shows the evolution of  $G'$  for sago samples 2, 5, and 9 and maize and potato starch over a 12 h period. The symbols represent the experimental data points while the solid lines for sago 2 and maize starch have been calculated using the first-order rate equation (Yoshida et al., 1990):

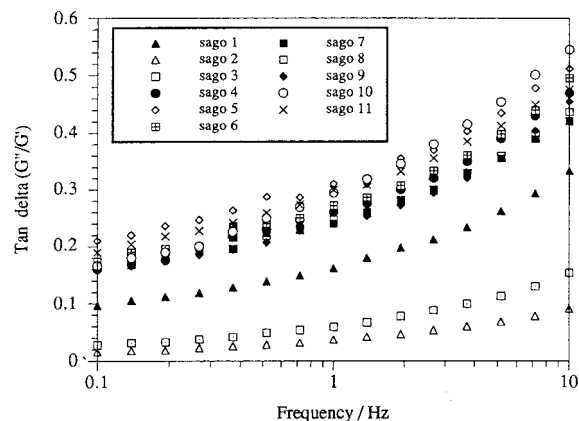
$$G_t = G_{\text{sat}} (1 - e^{-kt})$$

where  $G_{\text{sat}}$  = plateau value of  $G'$ ,  $k$  = rate constant for gelation, and  $t$  = time. The rate constant for sago 2 and maize starch was found to be 1.49 and 0.75  $\text{h}^{-1}$  respectively. The equation could not be used to calculate the rate constants for the other samples presumably because  $G_{\text{sat}}$  was achieved at or soon after  $t = 0$  and had a very low value.

Figure 6 shows the frequency dependence of  $G'$  and



**Figure 6.** Mechanical spectra for 6% sago and potato starch gels at 25°C.

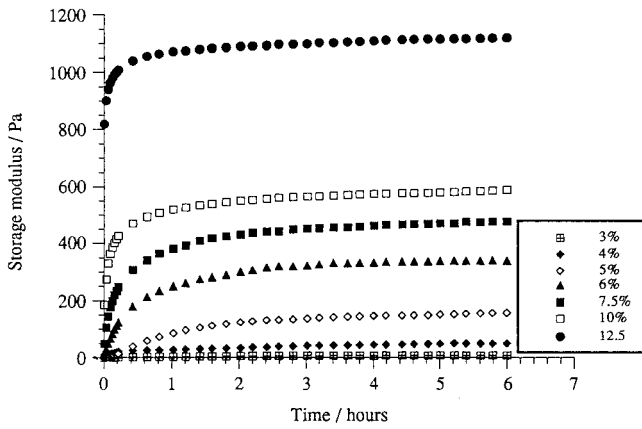


**Figure 7.**  $\tan \delta$  as a function of frequency for 6% sago starches.

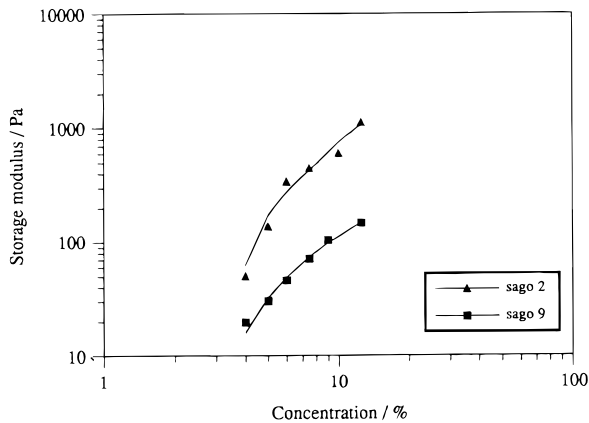
$G'$  for sago 2, sago 9, and potato starch. For the two sago samples,  $G'$  was found to be significantly greater than  $G''$  over the entire frequency range and this was particularly so for sago 2.  $G'$  was almost independent of frequency while  $G''$  was slightly frequency dependent. Potato starch showed similar behavior to sago starches although the  $G'$  values for potato starch were lower compared to sago starches. All the other sago starches and pea and maize starch showed similar mechanical spectra (data not shown). Akuzawa et al. (1995) reported similar frequency dependence of  $G'$  and  $G''$  for 4% sago starch dispersions. Muhrbeck and Eliasson (1987) and Svegmarm and Hermansson (1990) have also reported similar behavior for potato starch in the concentration range between 4 and 8%.

Figure 7 shows  $\tan \delta$  as a function of frequency for the sago starches. Only sago 2 and sago 8 give  $\tan \delta$  values below 0.1 at 10 Hz, indicating that these two starches form the strongest three dimensional gel network and, as discussed above, is presumably a consequence of the fact that the molecular mass for the amylose fraction in these two starches is significantly lower than in the other samples. For sago 1 the  $\tan \delta$  at 10 Hz was close to 0.3 while for other sago samples the  $\tan \delta$  at 10 Hz ranged between 0.4 and 0.5. The  $\tan \delta$  values for sago starches was largely dependent on frequency where the  $\tan \delta$  increases with frequency. Similar observation on  $\tan \delta$  as a function of frequency was reported for 4% sago starch dispersions by Akuzawa et al. (1995).

Figure 8 shows the increase in  $G'$  as a function of time for increasing concentrations of sago 2. All curves show

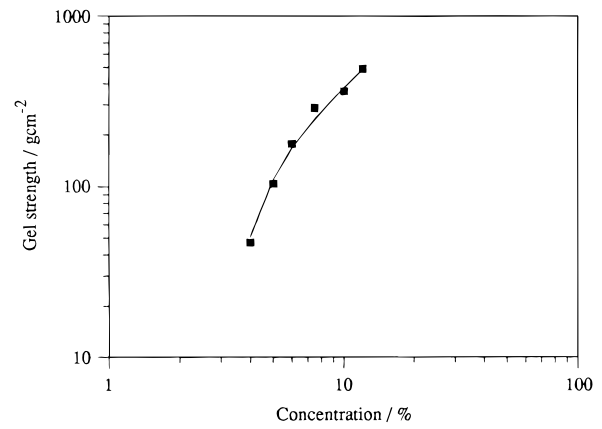


**Figure 8.** Effect of starch concentration on the storage modulus for sago 2 measured at frequency 1 Hz and at 25°C.

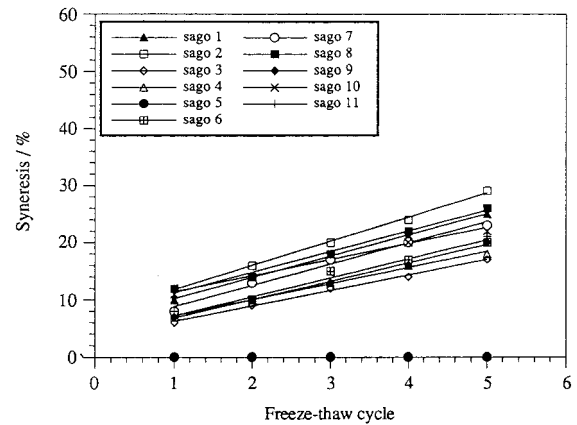


**Figure 9.** Storage modulus (frequency 1 Hz, temperature 25°C) after 6 h as a function of starch concentration for sago 2 and sago 9.

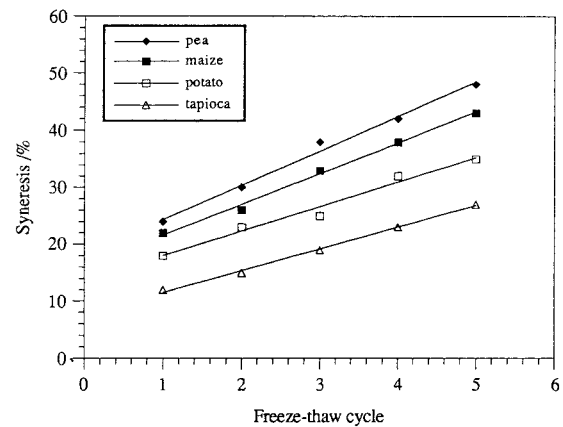
the same general shape but the pseudoplateau region is reached at shorter times as the concentration increases. Figure 9 shows the relationship between the final  $G_{\text{sat}}$  (at 6 h) and concentration for sago 2 and sago 9. For sago 2 it was found that  $G \propto C^{2.5}$ , while for sago 9 the relationship was  $G \propto C^{1.8}$ . It is evident that gelation only occurs above a critical minimum value of about 3.5% starch which corresponds to an amylose concentration of  $\sim 1.0\%$ . Other workers have shown that for amylose, gelation will occur above a minimum concentration of between 0.9% and 1.5% (Miles et al., 1985a; Ellis and Ring, 1985; Clark et al., 1989; Doublier and Choplin, 1989). For starch, the gelation normally occurs at a concentration of  $>6\%$  (Ring, 1985), and this will depend on the starch amylose content. A linear or almost linear concentration dependence on  $G$  has been reported for potato, wheat, and maize starch gels at concentrations up to 10% (Evans and Haisman, 1979); for pea, maize and potato starch gels in the concentration range from 6 to 30% (Ring, 1985) and for waxy maize amylopectin in the concentration range from 10 to 25% (Ring et al., 1987). Biliaderis and Tonagai (1991) reported  $G \propto C^{2.3}$  for garbanzo bean starches and  $G \propto C^{2.4}$  for rice starches, while Biliaderis and Juliano (1993) found  $G \propto C^{2.2-2.9}$  for rice starches. The dependence of  $G$  on concentration for sago starches are close to the theoretical limiting relationship  $G \propto C^{2.0}$  predicted for biopolymers (Clark and Ross-Murphy, 1987). Pure amylose gels have been shown to have a strong concentration dependence, with the exponent ranging from 4.4 to 7 (Ellis and Ring, 1985; Miles et al., 1985a; Clark et



**Figure 10.** Gel strength after 6 h as a function of starch concentration for 6% sago 2.



**Figure 11.** Freeze-thaw stability for 6% sago starch gels.



**Figure 12.** Freeze-thaw stability for 6% maize, pea, potato, and tapioca starch gels.

al., 1989). The higher power dependence of  $G$  on concentration for sago 2 compared to sago 9 is presumably attributable to the lower molecular mass for amylose. Figure 10 shows the gel strength (GS) as a function of concentration for sago 2. Again it is apparent that the minimum concentration for gelation is  $\sim 3.5\%$  and  $GS \propto C^{2.0}$  consistent with the small deformation oscillation results.

Figure 11 shows the freeze-thaw stability of the sago starches, and Figure 12 gives the freeze-thaw stability of maize, pea, potato, and tapioca starch for comparison. The results show that sago starches generally have better freeze-thaw stability compared to other starch varieties (White et al. 1989; Sosulki et al., 1997).

Syneresis is caused by increasing aggregation of amylose and amylopectin chains and will be influenced particularly by the amylose content and molecular mass. Sago starch samples 2 and 8 give rise to the highest syneresis, and this is attributed to the lower molecular mass of the amylose component. The resistance of sago 5 to undergo syneresis is not clear and further work is required to elucidate this point.

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