Viscoelasticity of Cowpea Starch Gels

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ABSTRACT

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The mechanical behavior of cowpea starch gels (10%, w/v) at small and large deformations was investigated in comparison with acorn, corn, and potato starches in storage at 4°C for seven days. The rapid viscograms of starch paste (7%, w/v) revealed that cowpea starch had a larger setback (1,135 cP) than other starches (465–830 cP), although peak viscosity (1,723 cP) and pasting temperature (76°C) were between those of corn and potato starches. Texture profile analysis of cowpea starch gel showed exceptionally higher values for hardness, gumminess, chewiness and initial modulus than other starch gels. Cowpea starch gel also exhibited higher G' and smaller tan δ compared with other starch gels, regardless of the storage time. A creep test revealed that the cowpea starch gel could remain highly resistant to stress, showing the least deformation among the tested starch gels during storage up to seven days. The overall results disclosed that cowpea starch was capable of forming exceptionally strong and elastic gels with good storage stability.

Starch gels are often consumed in the forms of desserts or side dishes in many countries. In Korea, cowpea starch, crude or purified, is currently used in place of mung bean starch for the preparation of a traditional starch gel dish called *mook* (Moon et al 1977). Not only cowpea and mung bean starches, but also the starches or flours from buckwheat and acorn could be used for the same purpose. Some of these starches have been characterized with elastic, stable, and irreversible gels different from ordinary cereal starch gels (Lee and Kim 1992).

Cowpea (*Vigna unguiculata*) is one of the legumes harvested mainly in Western African and South Asian countries. The seeds contain a relatively large quantity (66.5%, db) of carbohydrates, mainly starch, and the rest is mainly protein and lipids (28.5, and 3.5%, respectively, db) (Lee and Kim 1993, Chung et al 1998).

Several researchers have isolated starch from cowpea and have reported the pasting and gelling properties of the starch (Kim et al 1977, Yoon and Sohn 1988, Yoon 1992, Kweon et al 1993, Chung et al 1998). Kweon et al (1993) compared the pasting and gelling properties of the legume starches of different origins and found that the gels prepared with cowpea and mung bean starches had relatively higher hardness and cohesiveness than those of others. The harder and chewier texture of cowpea starch gel in comparison with corn and potato starches was also reported by Chung et al (1998). They also found that the cowpea starch gel had a better stability during repeated freeze-thaw cycles than potato starch gel.

A molecular feature of the cowpea starch was investigated by the same researchers (Chung et al 1998). Cowpea starch has a greater percentage (46%) of short chains (A and B1) in amylopectin than corn (41.3%) or potato starch (37.5%) but fewer (27.6%) longer B chains than corn (40.4%) and potato starch (45.0%). The amylose content, determined by gel filtration, was 27.9%, in between those of potato and corn starches. However, the amylose chains in cowpea starch were much longer than potato or corn amyloses. The authors hypothesized that the long amylose chains contributed to the high gel strength of cowpea starch (Chung et al 1998).

Despite the many mechanical analyses of cowpea starch gels with large strain, few studies have been conducted on the dynamic or static viscoelastic properties. In addition, effects of storage on the cowpea starch gel properties have not been investigated. In this study, not only the texture profiles but also the dynamic and creep properties of cowpea starch gel were measured during cold storage (4°C for seven days), and compared with the gels prepared with acorn starch which is another material used for Korean gel dishes, and corn and potato starches which are the popular commercial starches.

MATERIALS AND METHODS

Materials

Cowpea and acorn starches were isolated from the corresponding flours (Pulmuone Chanmaru Foods Co., Seoul, Korea) by using an aqueous NaOH solution (Chung et al 1998). Commercial corn and potato starches were provided by Samyang Genex Co. (Seoul, Korea), and Handuk Avebe (Seoul, Korea), respectively. Nitrogen analysis for protein content in starch was performed using an automatic Kjeldahl system (Kjeltec 1026, Tecator, Hoganas, Sweden), and crude lipids and ash were analyzed using AOAC Methods No. 920.85 and 923.03, respectively (AOAC 1995). Moisture content was measured using an infrared moisture analyzer (MB200, Ohaus, Florham Pk, NJ).

The pasting viscosity of starch was measured using a Rapid Visco Analyser (Newport Scientific, Australia). An aqueous dispersion of starch (7%, w/v) was heated from 25 to 95°C at a heating rate of 3.5°C /min, held for 10 min, and then cooled to 50°C at the same rate.

Starch gels were prepared following the procedure of Park et al (1999). Starch dispersion (10%, w/v) was heated in a boiling water bath for 15 min with mechanical stirring. The paste was then poured into petri dishes (50 mm diameter, 10 mm deep) and taped around the edge to accommodate excess paste. After cooling at room temperature for 1 hr, the paste was covered and then stored at 4°C for one to seven days.

Texture Profile Analysis

The texture profile of the starch gels was measured using a texture analyzer (TA-XT2, Stable Microsystems, Surrey, UK). After the tape was removed from the edge of petri dish, the top gel portion above the edge was carefully removed using a wire cutter. Then a gel column (35 mm diameter, 10 mm deep) was obtained from the middle portion of the gel with a cork borer. With the gel column, compression testing was repeated twice at 2.0 mm/sec with a cylinder-type probe (40 mm diameter) at 30% of the compression ratio where no gel fracture occurred. From the texture profile curve, hardness, cohesiveness, springiness, gumminess, chewiness, and initial modulus were calculated. Hardness was defined as the force necessary to attain a given deformation. Cohesiveness was the quantity necessary to simulate the strength of the internal bonds making up the sample. Springiness was the rate at which a deformed sample goes back to its undeformed condition after the deforming force was removed. Gumminess was the quantity necessary to simulate the energy required to disintegrate a semisolid

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sample to a steady state of swallowing. Chewiness was the quantity necessary to simulate the energy to masticate a semisolid sample to a steady state of swallowing. Initial modulus was the initial force at 0.5–1.5 sec divided by the contact area. The breaking force was also measured under the same conditions, but with the compression ratio increasing until gel breakage.

Dynamic and Creep Tests

Dynamic and creep viscoelasticity of the starch gels were analyzed with a rheometer (RS150, Haake Inc., Karlsruhe, Germany) equipped with a temperature control unit. As the specimens for tests, gel disks (35 mm diameter, 3 mm high) were prepared by carefully slicing the gel column using the method of Choi and Lee (1998). The disk was placed between two parallel plates with serrated surfaces to prevent surface slippage. Silicon oil was applied on the exposed surface of the specimen to prevent moisture evaporation.

The dynamic and creep tests were performed at 25°C in a linear viscoelastic range (30 Pa) where the moduli were independent of stress amplitude. The oscillatory shear range was 0.01–6.81 Hz. The creep test was performed by measuring the time-dependent strain of the gel (deformation) while applying a constant load (30 Pa) for 300 sec on the disk specimen. The measurements were repeated at least five times for each sample, and significance in difference was identified using Duncan's multiple range test (SAS Institute, Cary, NC).

RESULTS AND DISCUSSION

Minor Components

The cowpea and acorn starches isolated from the flours contained minor amounts of protein (≈0.4 or 0.3%, respectively, db), which were similar to the amounts found in the commercial corn or potato starch (Table I). The lipid content appeared relatively higher in acorn starch (0.26%) but was <0.3% in all starches. Cowpea and acorn starches contained ≈0.1% ash, which was significantly less than that of corn or potato starch. The overall contents of the minor components were low enough for the cowpea and acorn starches to compare with commercial corn and potato starches.

TABLE I
Proximate Contents (db) of Impurities in Cowpea, Acorn, Corn, and Potato Starches

Starches	Protein	Lipids	Ash
Cowpea	0.41	0.17	0.09
Acorn	0.28	0.26	0.10
Corn	0.48	0.15	0.30
Potato	0.20	0.09	0.65

TABLE II
Pasting Temperature (T) and Viscosity of Cowpea, Acorn, Corn, and Potato Starches[a]

		Viscosity (cP)				
Starches	**T (°C)**	**Peak**	**Through**	**Breakdown**	**Setback**	**Final**
Cowpea	76bc[b]	1,723b	1,086b	637b	1,135a	2,221a
Acorn	77b	1,255c	766c	489bc	781b	1,547b
Corn	81a	1,167c	774c	393c	465c	1,239c
Potato	63d	7,613a	1,620a	5,993a	830b	2,449a

[a] 7% starch (w/v).
[b] Values followed by the same letter are not significantly different ($P < 0.05$).

Pasting Temperature and Viscosity

Pasting temperature and viscosity of cowpea and other starches are summarized in Table II. Among the four starches tested, corn starch showed the highest pasting temperature (80.8°C) whereas potato starch showed the lowest (62.3°C). Peak viscosity appeared in inverse order to pasting temperature. Cowpea starch had a pasting temperature and peak viscosity in between those of corn and potato starches. However, cowpea starch displayed a higher breakdown than acorn or corn starch, indicating relative weakness of the swollen starch granules against hot shearing. It was also noteworthy that the starch paste had exceptionally high setback (1,135 cP), which contributed to the high final viscosity of >2,000 cP. This result indicates that cowpea starch molecules dispersed in hot paste reassociate more readily in the cooling stage than other starch molecules.

Texture Profile of Gels

After storage for one, three, or seven days at 4°C, starch gels were mechanically tested for the texture profile (hardness, cohesiveness, springiness, chewiness, gumminess, initial modulus, and breaking force). Most of the testing parameters, except cohesiveness and springiness, increased as storage time increased (Table III).

Among the tested starches, cowpea starch displayed the highest values for gel hardness, gumminess, chewiness, and initial modulus, regardless of the storage period. Springiness of cowpea starch gel, which represents recovery from deformation, was significantly ($P < 0.05$) higher than those of other starch gels on the first day of cold storage, but it tended to decrease as the storage continued (Table III). But potato starch gel showed an increasing tendency of springiness with storage time. The slow but continuous increase in gel strength for potato starch has been observed by other researchers (Orford et al 1987).

The exceptionally high initial modulus and breaking force that cowpea starch gels displayed indicate that the starch can form gels with significantly firm and surface-resistant texture against compression force. As the storage period increased, the rate of modulus and breaking force increase were greater for cowpea starch than for the other starch gels. The gel firmness and resistance might be desirable attributes when the cowpea starch is used for the gel-type dishes. The readiness for retrogradation that cowpea starch paste showed in the pasting curve could be one of the major contributors to making gel firm and rigid.

Potato starch gel showed breaking forces as high as that of cowpea starch gel although it had significantly less hardness (Table III). Chung et al (1998) also reported that hardness of cowpea starch gel was greater than that of potato starch gel, but cohesiveness of cowpea gel was similar to that of potato. Despite the low value of hardness, the potato starch gel was not readily broken and this property might be due to the relatively high cohesiveness.

TABLE III
Textural Properties of Starch Gels[a] Stored at 4°C for 1, 3, and 7 Days

	Hardness (kg$_f$)			Cohesiveness			Springiness			Gumminess (kg$_f$)			Chewiness (kg$_f$)			Init. Mod. (N/cm^2)			Break Force (kg$_f$)		
Starches	**1**	**3**	**7**	**1**	**3**	**7**	**1**	**3**	**7**	**1**	**3**	**7**	**1**	**3**	**7**	**1**	**3**	**7**	**1**	**3**	**7**
Cowpea	2.20a[b]	4.13a	5.11a	0.91a	0.90c	0.90c	0.94a	0.91a	0.89c	1.96a	3.33a	4.40a	1.90a	3.43a	4.22a	2.87a	6.22a	7.75a	5.88b	8.94a	10.07a
Acorn	0.65b	1.08b	2.03b	0.86b	0.92bc	0.91c	0.90bc	0.92a	0.95a	0.53b	1.07b	1.83b	0.56b	0.97b	1.74b	0.97b	1.45b	2.74b	3.16c	3.62b	4.65b
Corn	0.56b	1.07b	1.62b	0.90ab	0.94a	0.93b	0.91ab	0.94a	0.94a	0.50b	0.91b	1.49b	0.46b	0.85b	1.40b	0.89b	1.33b	1.98b	2.93c	2.96b	3.33c
Potato	0.61b	1.11b	1.69b	0.80c	0.93ab	0.94a	0.86c	0.91a	0.92ab	0.37b	1.03b	1.58b	0.31b	0.98b	1.47b	0.78b	1.76b	2.74b	7.59a	8.92a	10.77a

[a] 10% starch (w/v).
[b] Values followed by the same letter are not significantly different ($P < 0.05$).

Dynamic Viscoelastic Properties

As one of the variables for dynamic viscoelasticity, the storage modulus (*G*′) characterizes the elastic behavior of the material, the ability to restore energy during one deformation cycle, and the loss modulus (*G*″) refers to the liquid-like properties or energy loss (Ferry 1980). The loss modulus characterizes the ability to dissipate the energy applied on sample during one deformation cycle. The loss factor (tan δ) is measured as the ratio between *G*″ and *G*′. and the viscosity of sample increases, whereas the elasticity decreases in proportion to the loss factor.

Figures 1–3 show the dynamic viscoelastic properties of the starch gels at different storage periods. The starch gels tested showed the increased moduli (*G*′ and *G*″) as the storage period increased, which was common with many polymer gels. As the frequency increased, *G*′ was relatively constant but *G*″ varied significantly. As a whole, *G*′ was higher than *G*″ in the frequency range tested, characterizing the predominantly solid-like properties of the gels (Figs. 1 and 2). This made the loss factor <1 (Fig. 3). Yoon et al (1997) analyzed the dynamic viscoelasticity of the gels of Job's Tears flour, and reported similar tendencies. They claimed that the high ratio of *G*′/*G*″ was a typical phenomenon of polymers at a high concentration. They also found that at a moisture content higher than 75%, both *G*′ and *G*″ became more dependent on the frequency and tended to cross each other as the frequency increased.

Cowpea starch exhibited the higher values of *G*′ and *G*″ than other starches even on the first day of storage (Figs. 1 and 2). The *G*′ of cowpea starch, which was positively related to gel strength, was significant different from other gels. The rigidity of the cowpea starch gels shown by dynamic analysis agreed with the results from texture analysis (Fig. 1). Corn starch gel displayed the lowest *G*′ value that could be predicted from the lowest hardness from the texture analysis.

The *G*″ at low frequency range (<0.1 Hz) appeared decreasing with frequency for all starches. At >0.1 Hz, it varied depending on the starch type (Fig. 2). The cowpea starch gels showed steep reductions of *G*″ as the frequency increased, whereas other starch gels showed relatively constant or slightly increased *G*″. These phenomena also appeared in the work by Bello-Perez and Paredes-Lopez (1994) on starch and amylopectin gels. They claimed that the frequency dependence of *G*″ gave valuable information concerning the nature of the network structure of a starch paste or gel. The weak network interactions in the gel might result in the reduction

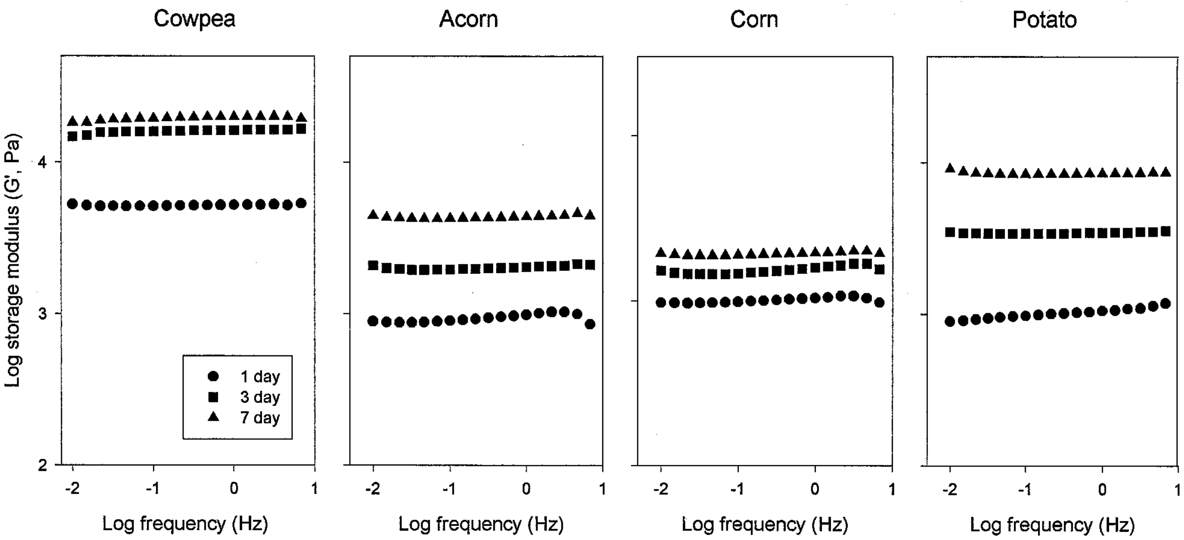

Fig. 1. Storage modulus (*G*′) vs. frequency for starch gels stored at 4°C for one, three, and seven days.

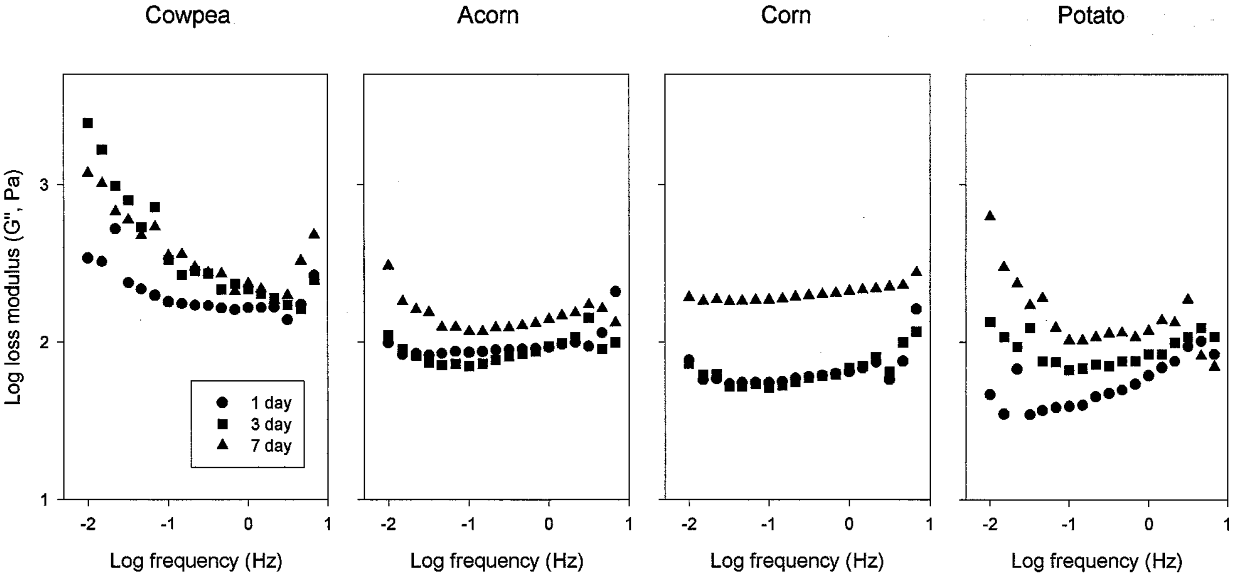

Fig. 2. Loss modulus (*G*″) vs. frequency for starch gels stored at 4°C for one, three, and seven days.

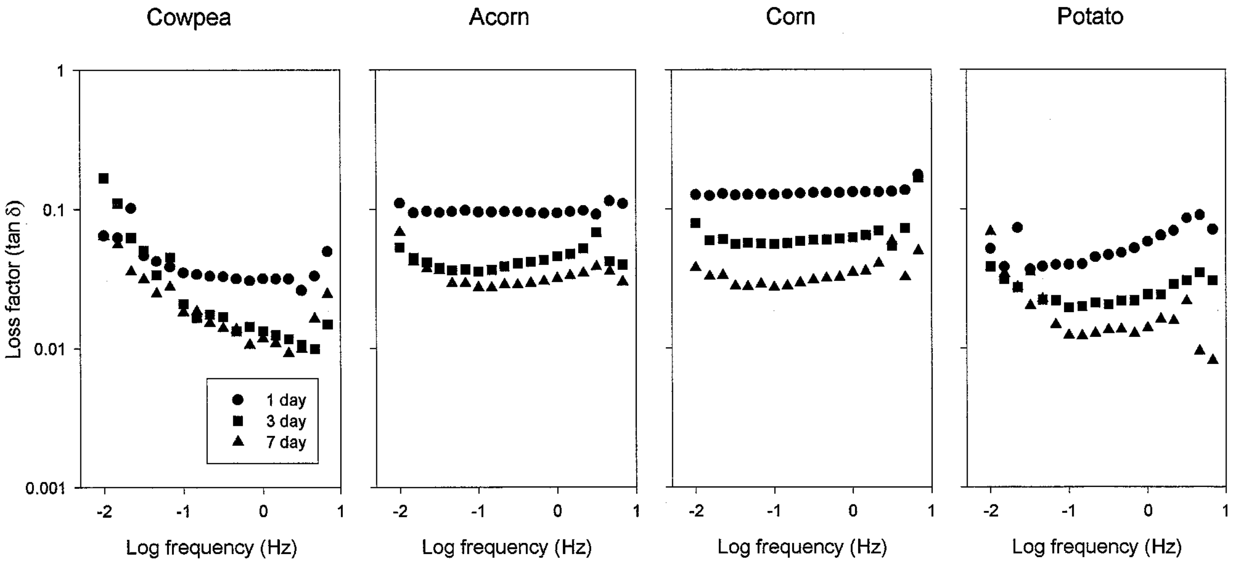

Fig. 3. Loss factor (tan δ) vs. frequency for starch gels stored at 4°C for one, three, and seven days.

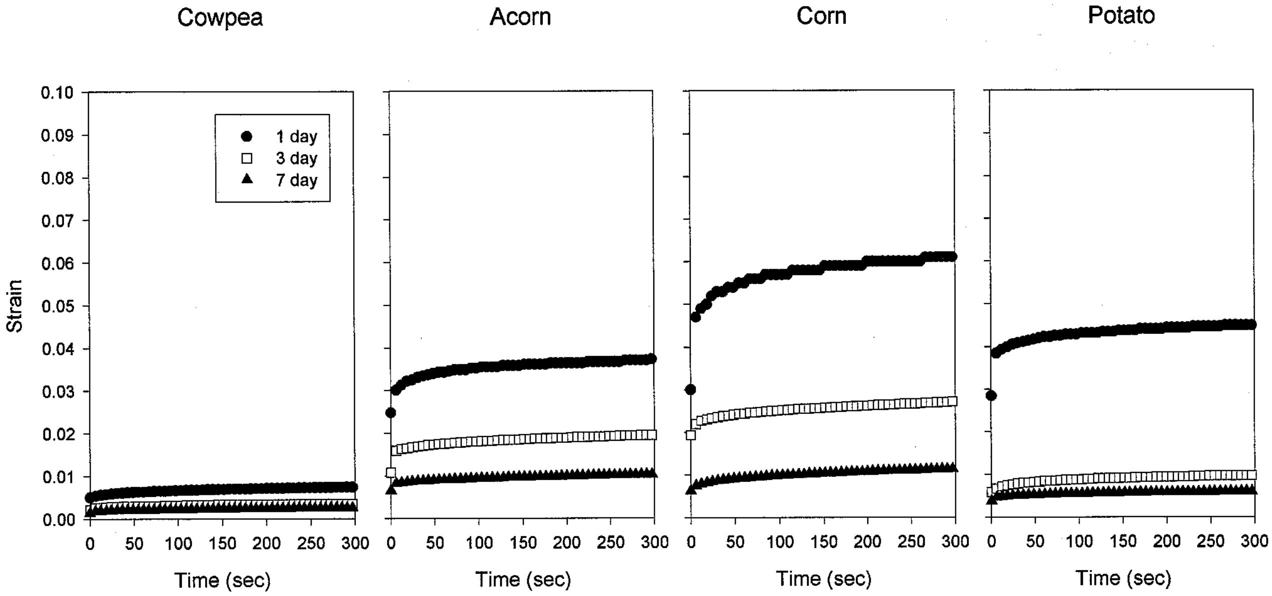

Fig. 4. Creep curves of starch gels stored at 4°C for one, three, and seven days.

of *G*″ with frequency. The cohesiveness change of cowpea gel over the storage period was possibly an indication that the gel had weak network interactions. And thus, the gel became less resistant to the oscillating flow at the higher frequency, resulting in the reduction of *G*″ (Fig. 3). More experiments should be conducted for clear understanding of this phenomenon.

During extended storage period, the *G*′ and *G*″ values changed in different ways, depending on the origin of starch, although most gels displayed the increasing trends for *G*′ and *G*″ (Figs. 1 and 2). Cowpea starch displayed a continuous increase in both *G*′ and *G*″ up to three days, but no significant increase was found thereafter. In contrast, potato starch gel displayed marginal and continuous increases in both moduli up to seven days. The continuous development of gel networks by potato starch has been reported by Orford et al (1987). Corn starch gel showed relatively small increments in *G*′ that were continuous for seven days. The *G*″ of corn starch gel, however, raised mostly at three to seven days. From this result, it was postulated that the substantial change in the corn starch gel matrix might occur in the later stage of storage (three to seven days). It was also noteworthy that the gels of corn starch were more stable with respect to frequency change (0.01–6.81 Hz) than other starch gels (Figs. 2 and 3).

These trends in *G*′ and *G*″ changes by frequency could be combined to with loss factor (tan δ, Fig. 3). Tan δ decreased as the storage time increased, which was typical for the starch or most polysaccharide gels. Cowpea starch showed smaller values of loss factor than other starches, indicating that the corresponding gel was more solid-like and elastic. As the frequency increased, tan δ of the cowpea starch gel decreased. This trend was attributed to the similar pattern of *G*″ versus frequency.

Creep Properties

Creep properties could be defined as the material deformation dependency on time when a constant stress or force was applied. Normally, the semisolid or solid food materials exhibit time-dependent deformations. This test provides information about the network formations and structural changes on the molecular level inside the material.

The strain changes (degree of deformation) versus time of the different starch gels while creeping for 300 sec with 30 Pa are given

TABLE IV
Creep Parameters[a] Analyzed by Inokuchi Method (1985) of Starch Gels Stored at 4°C for 1, 3, and 7 Days

	E_o (10^3 Pa)			E_r (10^3 Pa)			η_r (10^3 Pa • sec)			η_N (10^3 Pa • sec)		
Starches	1	3	7	1	3	7	1	3	7	1	3	7
Cowpea	5.7	11.8	16.8	22.6	38.3	59.3	331.8	441.0	713.9	6,876.0	14,565.8	16,776.5
Acorn	1.2	1.9	3.7	3.9	9.1	18.1	54.5	113.1	189.2	2,821.5	3,803.0	6,031.1
Corn	0.8	1.4	4.2	2.6	6.6	8.3	39.6	72.9	87.0	2,550.0	2,748.9	3,510.3
Potato	0.9	4.7	6.5	3.6	10.5	29.9	47.1	155.9	454.2	2,437.3	5,334.5	10,194.3

[a] Instantaneous elasticity (E_o), retarded elasticity (E_r), retarded viscosity (η_r), Newtonian viscosity (η_N).

TABLE V
Correlation Coefficients ($P < 0.05$) Between Creep and Texture Profile Analysis Parameters

	Hardness			Cohesiveness			Springiness			Gumminess			Chewiness			Initial Modulus			Breaking Force		
	1	3	7	1	3	7	1	3	7	1	3	7	1	3	7	1	3	7	1	3	7
E[a]	0.99	0.99	0.91	ns[c]	ns	ns	0.91	ns	ns	0.99	0.99	0.92	0.99	0.99	0.91	0.99	0.99	0.95	ns	ns	ns
η[b]	0.99	0.98	0.86	ns	ns	ns	ns	ns	ns	0.98	0.99	0.87	0.98	0.99	0.86	0.99	0.99	0.91	ns	ns	ns

[a] Instantaneous elasticity + retarded elasticity.
[b] Newtonian viscosity + retarded viscosity
[c] Not significant.

in Fig. 4. All starch gels displayed strain reduction as the storage time increased, showing the tendency for the gels to become stronger and more resistant to the stress. After one to three days, corn starch gel showed the highest strain (at 300 sec) whereas cowpea starch showed the least. But after seven days, the differences in final strain among the starches became minor. Potato starch showed a significant decrease in maximum strain during storage from one to three days, which could be expected from the dynamic moduli data. The cowpea starch gels maintained a strong and rigid texture, showing the least changes in strain during the storage. For the analysis of the creep curves, the Inokuchi method (1985) was used:

$$\varepsilon(t) = \sigma_o/E_o + \sigma_o/E_r\,(1 - e^{-t/Tr}) + \sigma_o \bullet t/\eta_N \qquad (1)$$

In the equation, $\varepsilon(t)$ was strain at time t, σ_o was constant stress, E_o was instantaneous elasticity, E_r was retarded elasticity, Tr was retardation time, and η_N was Newtonian viscosity.

The cowpea starch showed a significantly higher instantaneous elasticity than the other starches, indicating that the gel was little deformed at the initial stage of creeping (Table IV). And the E_o value increased continuously during the storage period. Lynch and Mulvihill (1994) reported that E_o was related to the stability of the network structure. Therefore, the cowpea starch was supposed to form a gel matrix with a substantially higher stability, whereas corn starch gel was least stable.

Retarded elasticity (E_r) and viscosity as two elements (Voigt body [η_r] and Newtonian viscosity [η_N]) also appeared significantly higher with cowpea starch gels than with the gels of other starches. Therefore, the cowpea starch formed a unique gel structure in which the retarded deformation and Newtonian flow were relatively small.

The results from the creep tests were consistent with the texture profile analysis as well as the dynamic viscoelasticity test. The overall results from these tests revealed that cowpea starch had the better ability to form gels with rigidity and stability.

The correlation analysis between the creep results (Table IV) and the texture profiles (Table III) is summarized in Table V. Creep test is normally conducted with a very small range of deformation measuring the structural changes, whereas the textural profile analysis, which is performed with in a wide range of deformation, is related rather to the sensory characteristics (Konstance 1991). It is important to observe the correlation between creep and TPA tests, although the strain regions were different because any change in molecular structure could influence the textural quality of the foods (Choi 1999). Regardless of the storage time, the sum of elastic properties (E_o plus E_r) as well as the sum of viscosity characteristics ($\eta_r + \eta_N$) measured by the creep test, had significant correlations with hardness, chewiness, gumminess, and initial modulus from the texture analysis (Table V).

The great capability that the cowpea starch displayed in the intermolecular network formation could be attributed to the starch's molecular structure and composition. It was reported that the cowpea amylopectin had more short chains (A and B1) than corn or potato starch. The relative content of amylose fraction in cowpea starch was between those of corn and potato starches, but the cowpea amylose had greater chain length (Chung et al 1998). The long amylose chains might contribute to the quick retrogradation and stable setting of the gel networks, as indicated in the pasting viscosity profile. The short chains of amylopectin could contribute to the gel rigidity and stability developed in the late stage of storage.

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