

Cellulosic Fiber Gels Prepared from Cell Walls of Maize Hulls

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ABSTRACT

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Cellulosic fiber gel substances were prepared from maize hulls by chemically and physically treating the pericarp cell-wall substrate in a multistage process to disintegrate morphological cellular structure. Thermal alkaline degradation was used with impact shear in the first stage, followed by alkaline peroxidation and shear in the second stage. The degradation products were removed in the liquid supernatants at each stage. The maize fiber gels are characterized by high viscosities and hydration capacities. After drying, they can be reconstituted to the original gel

form by using shear forces. The fiber gels were also dried together with hydrophilic materials to more easily reconstitute the gel form. The alkaline-extracted hemicellulose effluents from the fiber gel preparation, including the second stage peroxidized extract, were purified and used as hydrophilic material. Oat flour hydrolyzate, an amylopectin, also was used as a hydrophilic material. The physical properties of the gels were characterized by scanning electron microscopy and rheological evaluations.

Trowell (1972) and Burkitt et al (1972) were pioneers who related the importance of dietary fibers to gastrointestinal health. Today, it is well established that insoluble dietary fibers can contribute to important gastrointestinal functions, and some of the soluble fibers can contribute to other health benefits. Wheat bran decreases transit time (Anderson and Chen 1979) and other fibers lower blood cholesterol (Munoz et al 1979). Coarse-textured maize and soybean hulls pass through the gastrointestinal tract without appreciable disruption of the rigid morphological pericarp cell-wall structures (Dintzis et al 1979). Other researchers (Gould et al 1989) altered the lignocellulose contents of some fibers, which preserved skeletal structures, using alkaline hydrogen peroxide without thermomechanical shear processing. Over the years, many milled cereal flours have been refined to reduce the husk and bran fibers to give these flours greater smoothness in the gastrointestinal tract.

In contrast to the coarse fiber hulls (Dintzis et al 1979), maize fiber gels are very smooth textured and should not cause gastrointestinal problems. Furthermore, these gels can increase health benefits when combined with soluble fibers. Numerous studies show that some soluble fiber sources such as oat flour lower blood cholesterol (Ripsin et al 1992).

Hydrolyzed oat flour (also called Oatrim) is an oat amylopectin product that is used as a source of soluble β -glucan (Inglett, 1991). The soluble β -glucan found in hydrolyzed oat flour retains health benefits related to heart disease, glucose tolerance, and weight control (Behall et al 1993; Scholfield et al 1993; Inglett and Newman 1994; Hallfrisch et al 1995; Behall et al 1997).

The objective of this study was to describe the previously unknown physical properties of the maize cellulosic gels and to recap the known nutritional properties of maize fiber gum and β -glucan.

MATERIALS AND METHODS

Maize hulls (corn bran) were obtained from ConAgra Corn Processing (Atchison, KS). The corn bran was composed of finely ground hulls from dry-milling of maize with >90% passing through a U.S. #60 sieve. Another type of maize hulls (wet-milled maize hulls) was obtained from CereStar (Hammond, IN). After the hulls were

removed from the wet process stream, they were air-dried and ground to a particle size of ≤ 1 mm. Hydrolyzed oat flour (Oatrim-5) was obtained from Quaker Oats Food Ingredients Co. (Chicago, IL). Taka-Therm L-340, a thermostable bacterial α -amylase was supplied by Genencor International (formerly Solvay Enzymes, Elkhart, IN).

Viscosities were measured by viscometer (model LVT, Brookfield Engineering Laboratories, Stoughton, MA). Spindles #3 and 4 were used. Three replicates were made, and an average value, along with the associated standard deviation, was calculated.

Rheological properties were measured using a controlled-strain rheometer (ARES Series IV, Rheometrics Scientific, Piscataway, NJ) capable of measuring torques 0.2–2,000 g-cm. All the rheological studies were conducted using 50-mm diameter parallel plate fixtures. The measured torques were monitored to ensure that the detection limits of the rheometer were not exceeded for our samples. The temperature of the samples was controlled using an air-circulating oven equipped with a mechanical chiller, which enabled the chamber of the rheometer to be controlled to within $\pm 0.1^\circ\text{C}$. Thixotropic loop experiments were conducted over a shear rate range of 0–250/sec with a 4-min loop time. The upward and downward time segments of the thixotropic loop were kept equal for all the experiments. Stress relaxation experiments were conducted with applied strains in both the linear and nonlinear viscoelastic regimes. Oscillatory shear experiments were conducted over a frequency range of 0.1–100 rad/sec with an applied strain of 0.1%, which was well within the linear viscoelastic limits. For the rheological measurements, three replicates were made for each sample, and the average values, along with the standard deviations, were reported. The experimental data were analyzed using Rheometrics Orchestrator 6.5.2 and Rheometrics IGOR PRO 3.15 software.

Hydration capacities of the products were measured using Approved Method 56-20 (AACC 2000) for pregelatinized cereal products. Moisture content, protein, ash, and lipid levels were measured using AACC Approved Methods; and total dietary fiber was measured using Official Methods (AOAC 1990). To observe the microstructure of the products, dried films of the maize fiber gel were sputter-coated with 20-nm thick gold-palladium covering and micrographs were obtained using a Jeol 6400 V scanning electron microscope.

Maize Cellulosic Gel Preparation

Maize cellulosic gel was prepared using a multistage process. In a 20-L plastic tank, 1 kg of fine ground maize hulls was mixed with 11 L of water for the first-stage treatment. Approximately 10 g of a 50% sodium hydroxide solution (specific gravity 1.52; 13.3% w/v, alkali concentration) was added to adjust to pH 6.8. After heating the slurry to 90–94°C, 2.4 mL of α -amylase (Taka Therm 240) was added to the mixture, giving a concentration of 204 material weight units/g of substrate. The mixture was sheared in a dispersator

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(model 90, Premier Mill Corp., Reading, MA) equipped with a Hi-Vis colloid mill head at a rotation speed of 12,000 rpm at 90–98°C. After 15 min, 175 mL of a 50% sodium hydroxide solution was added to adjust to pH > 12 and the shearing was continued for 45 min. The solids were collected by decanting and washed two times with 200 L of deionized water in a 300-L vat before collecting the solids on a 25- μ m filter bag. The pH level of the solids was \approx 7.

In the second-stage treatment, the wet solids (volume amount 6 L) were adjusted to approximately pH 10 using a 50% sodium hy-

droxide solution. To this mixture, 500 mL of a 30% hydrogen peroxide solution was added, and the mixture was sheared in a colloid mill at 12,000 rpm and 90–98°C for 45 min. The slurry was stirred with mild agitation for 36 hr, and the wet solids were collected on a 25- μ m filter bag. The maize fiber gel had a viscosity of 18 ± 2 Pa-sec before drying. The composition of the dried maize fiber gel was moisture 7.17%, total dietary fiber 86.21%, crude fat 0.20%, crude protein 1.28%, and minerals 0.86%.

Preparation of Soluble Hemicellulose from Alkaline Peroxidized Extracts

In a 20-L mixing tank, 1 kg of finely ground maize hulls was treated as described above. All supernatants, following both the first and second stages, were combined to yield a yellow liquid containing the solubles from the two treatments. The combined liquids were passed through six layers of cheesecloth before undergoing ultrafiltration (Prostak 10K module, Millipore Corp., Bedford, MA). The 21-L volume of liquid was diluted to 500 L by adding reverse osmosis water. The resulting solution was pH 12.4. Ultrafiltration was continued for 2 hr until the color was substantially removed and reduced to approximately pH 9.4. A total of \approx 500 L of membrane filtrate was discarded. Concentrated yellow liquid (16 L) was recovered for further purification by activated charcoal treatment (Dowex 50W (H), Dow Chemical, Midland, MI) in a 50-mesh (280 μ m) sieve to adjust to pH 6–7. The resulting clear liquid was freeze-dried to yield a white powder of the purified, peroxidized, soluble maize hemicellulose fraction.

Drying Cellulosic Fiber Gel with Soluble Fiber Material

The maize cellulosic gel was dried together with hydrolyzed oat flour or soluble hemicellulose. The liquid gel was mixed with an equal weight of the hydrophilic solids. Either drum or oven-drying was used to dry the slurries. These gels were easily reconstituted at the 6% solids level with only moderate shearing force.

RESULTS AND DISCUSSION

Fiber gels were prepared from maize hulls by chemically and physically altering the pericarp cellular structures, as opposed to earlier work (Dintzis et al 1979, Gould et al 1989). A two-stage operation requiring elevated temperatures and intense shear force was essential for obtaining the desired functional gel properties. The dried gel products are readily dispersible in water and could be rehydrated to give high viscosity gels using general vigorous high shear procedures such as colloid mills and homogenization (Inglett 1998). The influence of shear time and water temperature of 25°C on the rehydration of a 3% dried maize fiber gel is shown in Table I.

The viscosity increased from 9.4 ± 0.9 Pa-sec to 12.6 ± 1.1 Pa-sec when the shear time was extended from 2.5 to 10 min at 25°C. The colloid mill was used for achieving sufficient shear. Repeating the same experiment, except using water at 50°C indicated only a modest increase in viscosity from 9.5 ± 0.9 Pa-sec to 10.7 ± 1.1 Pa-sec. There was not sufficient change in the hydration capacities; both stayed between 24 and 26. The untreated maize bran did not demonstrate either the viscosity ($\approx 0.004 \pm 0.0004$ Pa-sec) or the hydration capacity of the maize fiber.

The dried gel material rehydrated more readily in a hydrophilic substance, particularly a hydrophilic carbohydrate. The hydrophilic substance was added to the gels before drying to achieve the greatest degree of rehydration. The added hydrophilic substance also required less shear to achieve gel reconstitution. A suitable hydrophilic substance was hydrolyzed oat flour (Oatrim). Drying this hydrophilic carbohydrate with maize fiber gel in equal proportions gave a product with different viscosities and hydration capacities depending on the method of drying (drum or oven) and the degree of shear used to regenerate the gel. Reconstitution studies of the maize fiber and oat flour hydrolyzate composite gels, dried together using drum and oven procedures, are shown in Table II.

TABLE I
Influence of Shear Time (min) and Water Temperature (°C) on Reconstituting 3% Maize Fiber Gel and Bran

Shear Time	Temp	Viscosity		Hydration Level ^a	
		Fiber Gel (Pa-sec)	Bran (Pa-sec)	Fiber Gel (%)	Bran (%)
2.5	25	9.4 \pm 0.9	0.004 \pm 0.0004	25	4
5.0	25	11 \pm 1.1	0.017 \pm 0.00017	27	4
10.0	25	12.6 \pm 1.3	0.014 \pm 0.0014	26	5
2.5	50	9.5 \pm 0.9	0.010 \pm 0.001	26	4
5.0	50	7.7 \pm 0.7	0.015 \pm 0.0015	25	4
10.0	50	10.7 \pm 1.1	0.021 \pm 0.002	24	4

^a Measured using Approved Method 56-20 (AACC 2000).

TABLE II
Reconstitution Studies of Dried^a Maize Fiber and Oat Flour Hydrolyzate Composite Gels Using Colloid Mill Shear Conditions^b

Sample	Viscosity (Pa-sec)		Hydration Level ^c (%)	
	Drum	Oven	Drum	Oven
Maize fiber gel (3%)	6.2 \pm 0.6	7.3 \pm 0.7	23	19
Maize fiber gel (3%) and oat flour hydrolyzate ^d	8.5 \pm 0.8	15.5 \pm 1.5	28	21

^a Drum drying with 280 kPa of steam. Oven drying with forced air at 65°C.

^b Duplex head mill for 5 min.

^c Measured using Approved Method 56-20 (AACC 2000).

^d Oat flour hydrolyzate without maize fiber gel at 3% solids had a viscosity of 0.008 ± 0.0008 Pa-sec. Sample evaluated at 6% solids to maintain 3% maize fiber gel concentration.

TABLE III
Reconstitution of Maize Fiber Gels Prepared with Hydrophilic Carbohydrates and Dried Together^a by Drum and Oven Procedure Using Blender Shear Conditions^b

Sample	Viscosity (Pa-sec)		Hydration Level ^c (%)	
	Drum	Oven	Drum	Oven
Maize fiber gel (3%)	1.1 \pm 0.1	0.055 \pm 0.005	23	19
Maize fiber gel (3%) and oat flour hydrolyzate ^d	3 \pm 0.3	0.100 \pm 0.01	28	21

^a Drum drying with 280 kPa of steam. Oven drying with forced air at 65°C.

^b High speed for 5 min.

^c Measured using Approved Method 56-20 (AACC 2000).

^d Oat flour hydrolyzate without maize fiber gel at 3% solids had a viscosity of 0.008 ± 0.0008 Pa-sec. Sample evaluated at 6% solids to maintain 3% maize fiber gel concentration.

TABLE IV
Reconstitution of Maize Fiber Gel Prepared with Maize Hemicellulose and Dried Together by Drum Drying^a Using Colloid Mill Shear Conditions^b

Dietary Fibers	Solids (%)	Viscosity (Pa-sec)	Hydration Level ^c (%)
Hemicellulose fraction	3	0.008 \pm 0.0008	Soluble
Maize fiber gel (3%) and hemicellulose ^d (1:1)	6	4.2 \pm 0.4	30
Maize fiber gel (3%)	3	6.2 \pm 0.6	23

^a With 280 kPa of steam.

^b High speed for 5 min.

^c Measured using Approved Method 56-20 (AACC 2000).

^d Sample evaluated at 6% solids to maintain 3% maize fiber gel concentration.

The drum-dried samples viscosities on gel regeneration were 6.2 ± 0.6 Pa-sec for maize gel compared with 8.5 ± 0.8 Pa-sec for the composite. For the oven-dried samples, the regenerated viscosities were 7.3 ± 0.7 Pa-sec for maize gel compared with 15.5 ± 1.6 Pa-sec for the composite. The higher viscosities are probably related to the lower drying temperatures. Also, the high viscosity of the composite (15.5 ± 1.6 Pa-sec) suggests synergism between the components and possibly some protective colloid influence.

The importance of the shear influence on the regeneration of the fiber gels is shown in Table III. In this study, the same compositions used for the data in Table II (colloid mill shear force) were used to regenerate gels with only a blender (model 37BL84, Waring Products, New Hartford, CN) for the shear force. Under these experimental conditions, the oven-dried samples had viscosities of 0.055 ± 0.005 Pa-sec for maize fiber and 0.10 ± 0.01 Pa-sec for the composite. The drum-dried materials had higher viscosities of 1.1 ± 0.1 Pa-sec for maize fiber and 3.0 ± 0.3 Pa-sec for the composite. It is difficult to explain the magnitude of the difference between the oven- and the drum-dried samples. Mild impact shear generated by an ordinary blender was not sufficient to obtain the very high viscosities of the fiber gels. The addition of the oat flour improved the level of viscosity achieved for the drum- and oven-dried composites in comparison with the maize fiber gel alone. The study

characterized the importance of using adequate shear force to regenerate the maize fiber gels.

Maize hemicellulose is another source of hydrophilic material for combination drying. Maize hemicellulose can be extracted from corn hulls using alkali methods (Hespell 1998; Doner et al 1998; Leathers and Dien 2000). In the maize fiber procedure, hemicellulose is extracted in both stages. A composite containing this extracted, purified hemicellulose in combination with the maize fiber gel was dried and evaluated. The results (Table IV) did not show any increased viscosity over the maize fiber gel without hemicellulose. In fact, the composite was only 4.2 ± 0.4 Pa-sec and the maize fiber gel alone was 6.2 ± 0.6 Pa-sec. These results suggest that there is considerable variation in different hydrophilic materials and their function is related to maize gel rehydration.

Scanning Electron Micrographs

The maize gel products are characterized by several unique structural properties observed by scanning electron microscopy. The drum-dried maize fiber gels powder shows pieces of sheet-like or film-like morphology. At magnifications of 500 \times , scanning electron micrographs of the dried and fractured gel reveal fiber particles are flat and exist largely as smooth sheet-like pieces of mica (Fig. 1A). When water was added to hydrate the gel without shearing, a smooth

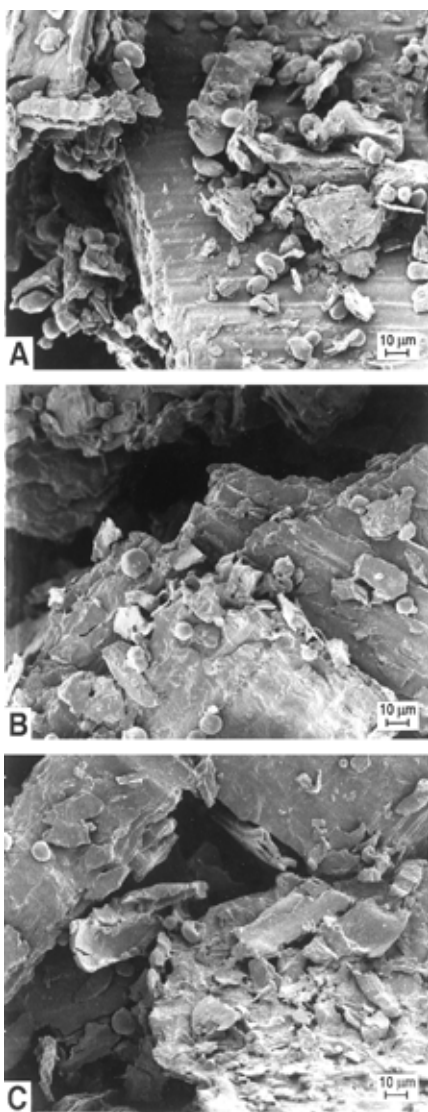


Fig. 1. Scanning electron micrographs (500 \times) of maize bran samples. **A**, dried powder brushed on surface; **B**, mixed with water and air-dried on surface; **C**, 3% slurry sheared in a colloid mill (6,300 rpm, 1 min) and air-dried on surface.

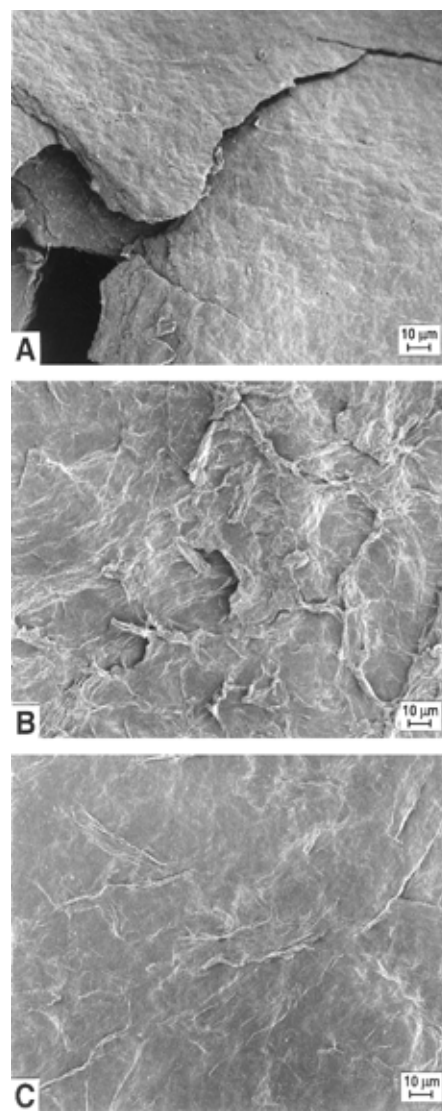


Fig. 2. Scanning electron micrographs (500 \times) of maize fiber gel samples. **A**, dried powder brushed on surface; **B**, mixed with water and air-dried on surface; **C**, 3% slurry sheared in a colloid mill (6,300 rpm, 1 min) and air-dried on surface.

structure emerged with fairly large deformable bodies ≈ 25 to 50 μm in each dimension with a wave-like surface (Fig. 1B). Using adequate shear forces to regenerate the gel, a smooth surface resembling a slightly disturbed water surface (Fig. 1C) was produced.

These micrographs are in sharp contrast to the initial maize bran before processing into gel form. Using magnifications of $500\times$, there is visual evidence of rough morphological cell-wall material, along with some spherical structures of ungelatinized starch (Fig. 2A). When hydrated by adding water alone without shear, the cellular structure become slightly more evident along with the ungelatinized starch bodies (Fig. 2B). By applying shear forces to the hydrated form of the unprocessed maize bran, the cellular particles remain

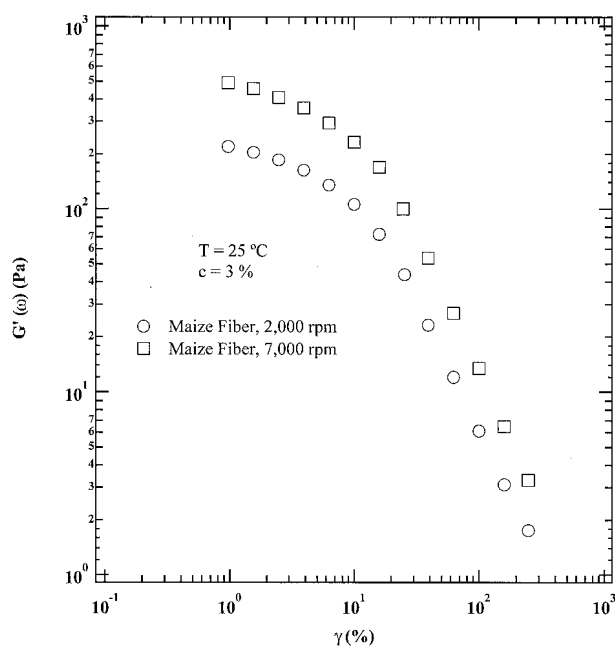


Fig. 3. Effect of strain on oscillatory shear storage modulus $G'(\omega)$ for 3% solids maize gel processed at 2,000 and 7,000 rpm in a colloidal mill for 5 min. Measured at 25°C using a controlled-strain rheometer.

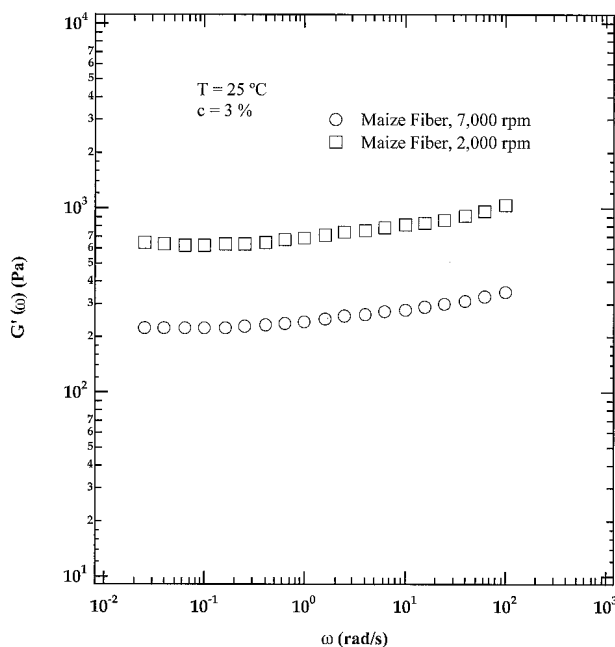


Fig. 4. Effect of frequency on oscillatory shear storage modulus $G'(\omega)$ for 3% maize gels processed at 2,000 and 7,000 rpm in a colloidal mill for 5 min. Measured at 25°C and a strain of 0.1% using a controlled-strain rheometer.

essentially unchanged (Fig. 2C), except for the starch granules. Some of the starch granules assumed flat and donut shapes, indicating partial gelatinization resulting from the heat produced during shearing.

Rheological Properties

The viscosity of the maize fiber gels at 3% solids can vary considerably depending on the preparation variables and the degree of shear used in regenerating the gel from dried materials (Inglett 1999). Maize fiber gels, as prepared before drying, were characterized with viscosities as high as 24 ± 2 Pa-sec. Generally, the viscosity of a typical preparation was ≈ 18 Pa-sec. Viscosities of the reconsti-

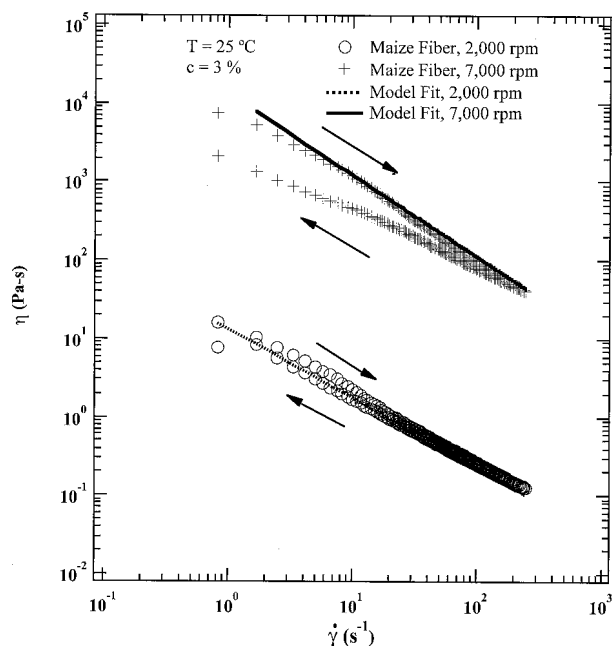


Fig. 5. Effect of shear rate on shear viscosity during a thixotropic loop experiment for 3% maize gels processed at 2,000 and 7,000 rpm in a colloidal mill for 5 min. Arrows are direction of applied shear rate. Solid and dotted lines are the fits to the data using a power law constitutive equation. Measured at 25°C using a controlled-strain rheometer.

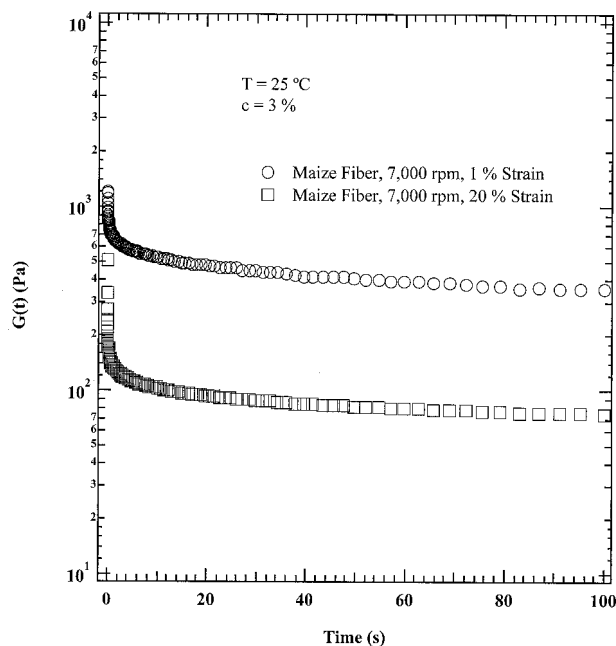


Fig. 6. Effect of applied strain on stress relaxation modulus $G(t)$ of a 3% maize gel processed at 7,000 rpm. Measured at 25°C using a controlled-strain rheometer.

tuted gels after drying were also highly variable. The viscosity was related to the quality of the gel before drying, the manner of drying the gel, drying materials together to aid in rehydration, and the type and force of shear used to regenerate the gel structure.

Preliminary studies of the rheological performance of the maize gels were conducted. For these studies, maize gels were reconstituted at 3% solids content in deionized water using a colloidal mill (Polytron PT6000, Kinematica GmbH, Kriens-Luzen, Switzerland) at mixing speeds of 2,000 or 7,000 rpm for 5 min. The viscoelastic properties of the gels were measured immediately after reconstitution to eliminate any effects from sample degradation with time.

The effect of strain on the oscillatory storage modulus $G'(\omega)$ of the maize gels is shown in Fig. 3. At strains $>1\%$, the moduli dropped rapidly with increasing strain, indicating nonlinear viscoelastic behavior. The transition region from linear to nonlinear viscoelastic behavior was not affected by the mixing speed. The effect of oscillatory shear rate on the shear storage modulus for 3% maize gels is shown in Fig. 4. The oscillatory data were obtained at a shear strain of 0.1%, which is within the region of linear viscoelastic behavior. Both of the gels display a plateau region at oscillatory shear rates <1 rad/sec. This plateau behavior is indicative of the gel-like nature of these materials. The maize gel processed at 7,000 rpm has a slightly higher plateau modulus than does gel processed at 2,000 rpm. This result indicates that the higher processing rate produces a gel with a more densely entangled structure; however, further research is required to verify this result.

The behavior of the 3% maize gels processed at 2,000 and 7,000 rpm during a thixotropic loop experiment is shown in Fig. 5. Both of the gels display shear-thinning behavior across the shear rate range studied. The maize gel processed at 7,000 rpm displayed a higher viscosity over the shear rate than the gel processed at 2,000 rpm, again indicating the more densely entangled nature of the gel processed at 7,000 rpm. The ending viscosity of the gel processed at 7,000 rpm was significantly lower than its starting viscosity, indicating some shear degradation of the network structure. This effect was not observed for the gel processed at 2,000 rpm. The rheological behaviors of the two gels were characterized using a power law constitutive equation which may be expressed as

$$\eta = K \dot{\gamma}^{m-1} \quad (1)$$

where η is the shear viscosity, $\dot{\gamma}$ is the shear rate, K is the front factor and m is the power law exponent (Bird et al 1977). The majority of fluids exhibit pseudoplastic behavior with m values of 0.15 to 0.6. Equation 1 was fitted to the initial upward cycle of the thixotropic loop experiment data for each of the two gels and yielded $K = 10.2 \pm 0.2$ and $m = 0.114 \pm 0.002$ for the gel processed at 2,000 rpm, and $K = 1,352 \pm 16$ and $m = 0.418 \pm 0.004$ for the gel processed at 7,000 rpm (Fig. 5). From the power law exponents, it is evident that all the suspensions exhibit pseudoplastic behavior. The gel processed at 2,000 rpm displays slightly stronger shear-thinning behavior over the shear rate range than does the gel processed at 7,000 rpm, as seen in the lower value for the power law exponent.

The effect of strain on the stress relaxation behavior for the 3% maize gel processed at 7,000 rpm is shown in Fig. 6. At the two strain levels, 1% (linear viscoelastic regime) and 20% (nonlinear viscoelastic regime), the stress relaxation modulus $G(t)$ decays to a stable plateau that is indicative of a gel-like structure. The value for the plateau is dependent on the strain level, again indicating the nonlinear viscoelastic behavior of these materials at strains $>1\%$.

CONCLUSIONS

Cellulosic fiber gels could be prepared from maize hulls obtained from maize processing industries. The fiber gels, in sharp

contrast to dietary fiber hulls, had high viscosities at low solids levels with the ability to retain large amounts of water in smooth deformable particles. Reconstitution of the dried fiber gel to the liquid form required elevated shear forces obtained from colloid mills or homogenization impact levels. Reconstitution was made easier and more efficient by drying fiber gel together with hydrophilic materials, including hemicellulose, gums, and maltodextrins.

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