

Solution Viscoelastic Properties of OATRIM-10 and Cooked Oat Bran

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ABSTRACT

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The solution rheological behaviors of OATRIM-10 and cooked oat bran were investigated. The rheological properties of the materials were investigated using both thixotropic loop and small-amplitude oscillatory shear experiments. The cooked oat bran exhibited shear-thinning behavior during a thixotropic loop experiment over a shear rate range of 0–250/sec. The shear-thinning behavior was reproduced during the measurement of a second thixotropic loop. In contrast, OATRIM-10 exhibited an unexpected region of shear-thickening behavior at 20–80/sec. The shear-thickening and subsequent shear-thinning regions for OATRIM-10 could be described by a transient network model indicating that the shear-thickening behavior is caused by a shear-induced entangled network

that is partially disentangled at higher shear rates. Subsequent thixotropic loop experiments displayed the shear-thickening region for OATRIM-10, indicating that the network structure can be reformed during the imposition of a shear field. Small-amplitude oscillatory shear data for cooked oat bran can be described reasonably well using a generalized linear viscoelastic (GLV) model. The oscillatory shear data obtained for OATRIM-10 could not be described by the GLV model. OATRIM-10 exhibited a distinctive plateau centered at 10/sec, and the low frequency response of storage modulus G' decreased with a much larger slope in frequency than was predicted by the GLV model.

Oat bran and other oat materials are used in numerous food applications where high-fiber content is desired in food products. The soluble fiber of oats is (1–3),(1–4)- β -D-glucan, which is highly regarded for contributing health benefits to food products (Inglett 1997). Numerous studies have shown that soluble fibers obtained from sources such as oat flours benefit health by lowering blood cholesterol (AHA 1980, Topping 1991, Malkki et al 1992, Uusitupa et al 1992, FDA 1997a,b). Consequently, the utilization of oat (1–3),(1–4)- β -D-glucan in the food industry has been an area of much active research recently.

A characterization of the rheological behavior of the systems is required to develop and produce materials efficiently. Rheological properties are of import in governing many industrial processes such as extrusion, injection molding, and baking. In addition, rheological measurements can be used to distinguish the effects of different processing conditions on the flow properties of materials. Application of rheological theories can aid in understanding of the conformational dynamics of the macromolecules and can be used to predict the flow behavior of the materials in processing. Validated rheological models can aid in the timely and cost-efficient development of new processes as well as in the development of new die and screw designs. Despite the importance of rheological properties in many industrial processes, a limited number of studies on the viscoelastic properties of oat-based biopolymers has been reported in literature (Wood 1984; Autio et al 1987, 1992; Autio 1988, Doublier and Wood 1995). Semidilute solutions of oat gums have displayed shear-thinning behavior at shear rate ranges of 0–1,500/sec. The limited data on the oscillatory shear viscoelastic properties of oat (1–3),(1–4)- β -D-glucan appear to display classic non-Newtonian flow behavior.

In this study, initial investigations of the solution viscoelastic properties of OATRIM-10 and cooked oat bran are reported. The materials were subjected to both thixotropic loop and small-am-

plitude oscillatory shear experiments. Cooked oat bran exhibited shear-thinning behavior over the shear rate range investigated. In contrast, OATRIM-10 exhibited an unexpected region of shear-thickening behavior. The data obtained from the thixotropic loop experiments for both of the materials was interpreted using a power law model. The oscillatory shear rheological properties of the cooked oat bran and OATRIM-10 were compared to the predictions developed from a generalized linear viscoelastic (GLV) model.

MATERIALS AND METHODS

Materials and Sample Preparation

The oat bran material used in this study was obtained from Grain Millers (Saint Ansgar, IA). The oat bran was designated as lot number IN-046-97-408 and was used as received. The cooked oat bran sample was prepared by creating a 5% (w/w) aqueous slurry of the oat bran in water. The slurry was brought to a boil in a microwave oven and cooked for 5 min. The solution was then cooled to room temperature.

The procedure to produce the OATRIM-10 sample was as discussed previously (Inglett 1993, Inglett and Newman 1994): 4 kg of the oat bran was slurried in 28 L of water containing 50 ppm of calcium. The slurry was adjusted to pH 5–8 and passed through a steam-injection cooker and collected in a 30-gal steam-heated kettle. An α -amylase enzyme preparation was added to the slurry (Inglett 1993). The solution was stirred at 80–90°C for 5 min, and the enzyme was deactivated by passing the slurry through a steam-injection cooker. The slurry was then centrifuged at 15,000 rpm in a Sharples centrifuge to separate the soluble and insoluble components. The soluble materials were isolated and the water was removed by freeze-drying the solution. The resulting material was designated as OATRIM. Degrees of polymerization and number average molecular weights of the amyloextrins produced by the enzymatic treatment are summarized in Inglett (1993). In this study, the specific OATRIM used was designated OATRIM-10 and contains (w/w): 81% amyloextrins, 10% (1–3),(1–4)- β -D-glucan, 4% various proteins, 4% ash minerals, and 1% crude fat.

The OATRIM-10 solution used in this work was prepared in a manner identical to that used to prepare the oat bran samples. OATRIM-10 was dissolved in water at a concentration of 5% (w/w). The solution was brought to a boil in a microwave for 5 min and then cooled to room temperature.

Rheological Measurements

Rheological properties were measured using a controlled-stress rheometer (CSL² 500, CarriMed, Dorking, England) with a cone-and-plate fixture. All the rheological studies were conducted using

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a 6-cm diameter, 4° cone. The temperature of the sample was controlled using a Peltier plate which enabled the chamber of the viscometer to be controlled to within $\pm 0.1^\circ\text{C}$. For all the data discussed in this report, the temperature was maintained at 25°C . For each solution, a small-amplitude oscillatory shear experiment was first conducted using frequencies of 0.01–10/sec. The oscillatory experiments were performed using a constant shear strain of 50%. Prior to the oscillatory experiments, a torque sweep was conducted on each sample to ensure that the applied shear strain was within the linear viscoelastic regime of the material. From the oscillatory experiments, the shear storage and loss moduli (G' and G'') were measured. After the oscillatory experiment, a thixotropic loop experiment was conducted by accelerating the cone uniformly from rest to a shear rate of 250/sec and back to rest over a 2-min period. The thixotropic loop experiment was repeated immediately. Each of the experiments were repeated a minimum of three times. The average relative standard deviation of the measurements was $\pm 10\%$.

RESULTS AND DISCUSSION

Thixotropic Loop Experiments

The effect of shear rate for the first cycle of the thixotropic loop experiment on the measured shear viscosity (η) for OATRIM-10 and the cooked oat bran is illustrated in Fig. 1. The data for the first cycle of the thixotropic loop experiment for oat bran exhibited shear-thinning behavior over the entire shear rate range investigated. The rheological behavior of OATRIM-10 differed markedly from that of the cooked oat bran. It exhibited an initial region of shear-thinning behavior followed by a region of shear-thickening behavior at $\approx 20\text{--}80/\text{sec}$ and then by another region of shear-thinning behavior. We believe that this is the first reported evidence for shear-thickening behavior in solutions containing (1–3),(1–4)- β -D-glucan and amylopectin biopolymers. The exact cause of the shear-thickening behavior for OATRIM-10 has yet to be determined. However, published work on (1–3),(1–4)- β -D-glucan biopolymers in solution indicates shear-thinning of these materials over the shear rate range examined in this work, so it is possible that the shear-

thickening behavior was caused by the high molecular weight amylopectins present in the sample. Studies of the rheological properties of the individual components of OATRIM-10 will be needed to establish the exact cause of this unexpected behavior.

The effect of a second thixotropic loop experiment on cooked oat bran and OATRIM-10 is illustrated in Figs. 2 and 3. For cooked oat bran, the data for the second thixotropic loop overlapped the data obtained during the initial cycle. This result indicates the absence of any flow-induced structure in the fluid caused by the imposition of the shear field. During the second thixotropic loop, OATRIM-10 again exhibited a region a shear-thickening behavior over the same shear rate range. This result indicates that the flow-induced structure formed during the shear-thickening regime is transient and is reversible at higher shear rates, but it can be reformed with a second application of the shear field. This is in contrast to recent findings for semidilute solutions of waxy maize starch which exhibited shear-thickening behavior over a similar shear rate range but produced a stable flow-induced structure (Dintzis and Bagley 1994, 1995; Dintzis et al 1996)

Modeling Shear-Thinning and Shear-Thickening Behavior

The effect of shear rate on the viscosity of the materials was characterized using a power law model (Macosko 1994) which can be expressed as:

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

where η is the viscosity of the solution, K is the front factor, and n is the power law coefficient. If n is greater than unity, the solution is defined as exhibiting shear-thickening behavior. If n is less than unity, the solution is defined as exhibiting shear-thinning behavior. Eq. 1 was fitted to the experimental data illustrated in Fig. 1 using K and n as input parameters. The standard deviation between the curve generated using Eq. 1 and the experimental data was defined as the difference between the log of the experimental viscosity and the log of the calculated viscosity. The best fit was defined as that set of input parameters that minimized the fit deviation. The computations were conducted using a Macintosh 9600/200MP using Wavemetrics IgorPro software. For cooked oat bran, the fit

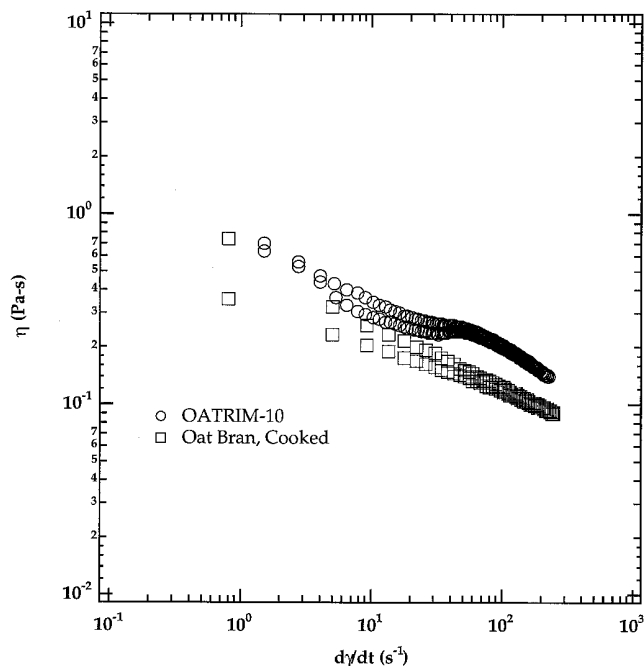


Fig. 1. Effect of shear rate ($d\gamma/dt$) on measured shear viscosity (η) during first cycle of a thixotropic-loop experiment for OATRIM-10 and cooked oat bran at 25°C . Shear-thickening behavior for OATRIM-10 is evident at 20–80/sec.

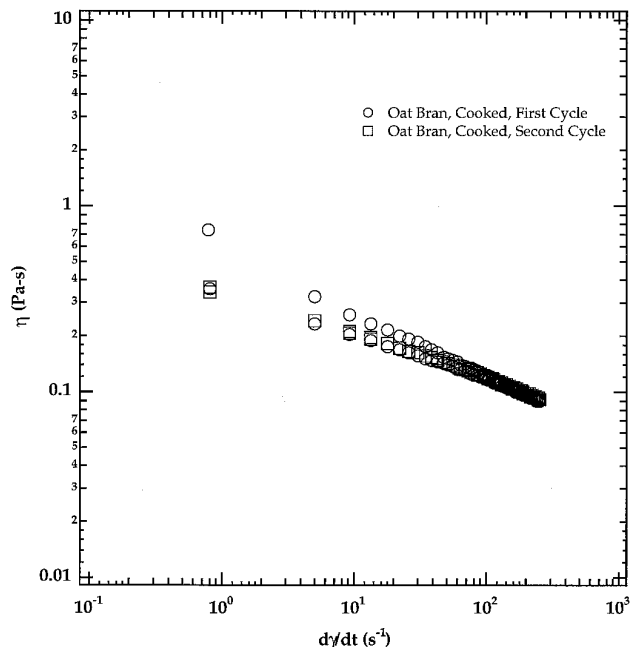


Fig. 2. Effect of shear rate ($d\gamma/dt$) on the measured shear viscosity (η) during first and second cycles of a thixotropic-loop experiment for cooked oat bran at 25°C . Solution exhibits shear-thinning behavior in both cycles.

was limited to the data obtained for the first upward cycle of the thixotropic loop experiments. For OATRIM-10, the model fit was limited to the shear-thickening regime (20–80/sec) during the first upward shear cycle of the thixotropic loop experiment.

Values of K and n obtained from the data of the model fit are summarized in Table I. For cooked oat bran, the values for n are less than unity, indicating the shear-thinning behavior of the solutions over the shear rate range of 0–250/sec. The model fit to the experimental data for cooked oat bran is illustrated in Fig. 4. For OATRIM-10 in the shear-thickening regime (20–80/sec), $n = 1.07$. This value indicates the moderate shear-thickening behavior of the solution.

Various theories that describe shear-thickening behavior in solution have been advanced by a number of different authors over the years (Bird and Marsh 1968, Marsh 1968, Ballard et al 1988, Petruccione and Biller 1988, Tanaka and Edwards 1992, Wang 1992, Marrucci et al 1993, Ahn and Osaki 1994, Hess and Hess 1994, Hatzikiakos and Vlassopoulos 1996). The theories range from phenomenological approaches to those based on model networks. I. M. Krieger (*private communication*) has suggested that shear-thickening behavior can arise in fluids as a consequence of the viscoelastic response of the system over certain shear-rate regimes.

Of the various approaches to model shear-thickening behavior that are based on model networks, the theory proposed by Wang (1992) is of particular interest. Wang's theory, describing both shear-thickening and shear-thinning behavior, is based on a transient network model. Wang's approach makes use of the concept of a

TABLE I
Summary of Power Law Coefficients for OATRIM-10^a
and Cooked Oat Bran at 25°C

Material	Shear Rate Range (sec)	K (Pa-sec) ^b	n ^b
OATRIM-10*	20–80	0.184 ± 0.006	1.07 ± 0.001
Cooked oat bran	1–250	0.661 ± 0.026	0.629 ± 0.025

^a K = front factor, n = power law coefficient. Model fit limited to shear-thickening regime.

^b Errors represent ± 1 standard deviation from the mean fit value.

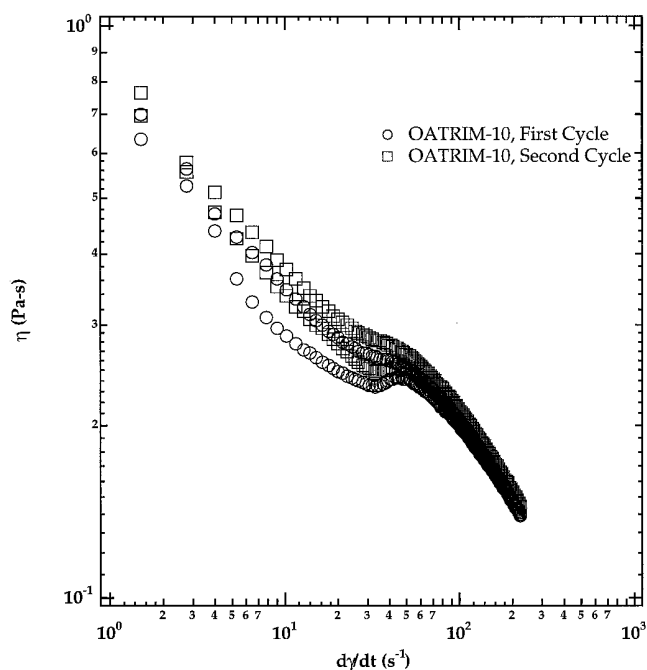


Fig. 3. Effect of shear rate ($d\gamma/dt$) on the measured shear viscosity (η) during first and second cycles of a thixotropic-loop experiment for OATRIM-10 at 25 °C. Shear-thickening behavior is evident at 20–80/sec in both cycles.

weakly joined network advanced by Tanaka and Edwards (1992) as well as Petruccione and Biller (1988). Shear thickening in this model is described by the development of a shear-induced entangled network. More chains join the network as the shear rate is increased above some critical value. The chain association can be either through chemical interactions such as hydrogen bonding or through chain entanglements. Shear-thinning after the shear-thickening regime is explained in this model as the subsequent dissociation of the flow-induced network. The expression for the shear viscosity in Wang's model can be expressed as:

$$\eta(\dot{\gamma}) = \eta_0 \frac{f(\dot{\gamma}, \delta)(1 + r\dot{\gamma}^2)[\bar{q} + g(0, \delta)]}{f(0, \delta)\{\bar{q} + [1 + g(\dot{\gamma}, \delta)](1 + r\dot{\gamma}^2)\}} \quad (2)$$

where η_0 is the zero-shear viscosity, $\dot{\gamma}$ is the reduced shear rate, \bar{q} is a model parameter related to initial free chain density, r is a model parameter related to the critical shear rate where shear-thickening commences, δ is a model parameter related to the disentanglement time of the network, and the integral functions f and g are as defined in Wang (1992). Eq. 2 was fitted to the experimental data using r , \bar{q} , and δ as input parameters. The calculations were conducted using Mathcad Pro software running on an Apple Macintosh 9600/200MP computer. The standard deviation was defined as the difference between the calculated viscosity and the experimental viscosity. The best fit was defined as the set of input parameters that produced the lowest standard deviation.

A comparison of the model prediction with experimental data is illustrated in Fig. 5. The model prediction was calculated using $r = 10$, $\bar{q} = 4.5$, and $\delta = 0.6$. From Fig. 5, it is evident that the transient network model does a reasonable job of describing both the shear-thickening region as well as the subsequent shear-thinning regime. Before the shear-thickening regime, the model assumes that the solution behaves as a Newtonian fluid and, thus, it is not able to describe the initial shear-thinning regime. This is a serious assumption in many of the models describing shear-thickening behavior in solutions. The model fit was limited to the shear-thickening and subsequent shear-thinning regions. The best fit for the model to the experimental data was obtained with $\delta = 0.6$. In the theory, δ describes the stability of the entangled network. For permanent networks $\delta = 0$; for systems that disentangle easily $\delta = 1$.

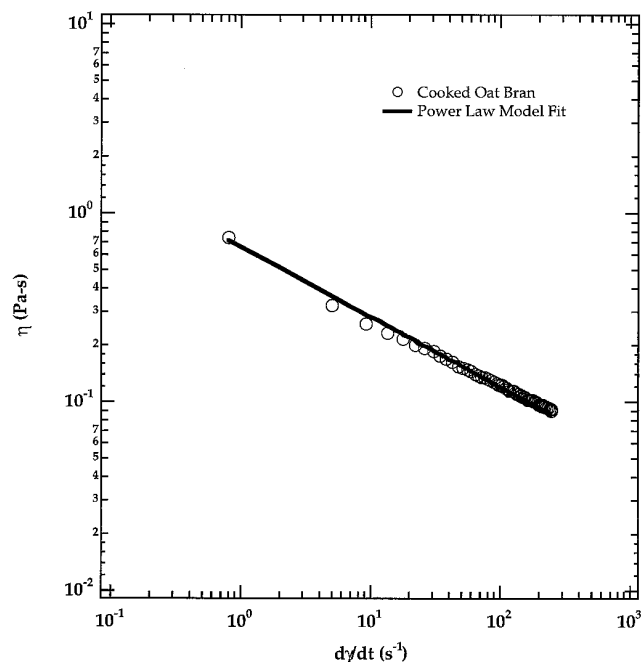


Fig. 4. Comparison of the experimental data for cooked oat bran obtained at 25°C during the first upward cycle of a thixotropic loop experiment with a power law model.

The network structure formed by OATRIM-10 is of intermediate stability according to the model. In addition, the agreement between the model and the experimental data adds credibility to the assumption that the shear-thickening behavior in OATRIM-10 is caused by a transient shear-induced associated network. However, additional work is needed to verify this proposal.

Oscillatory Shear Experiments

The dynamic viscoelastic properties of OATRIM-10 and cooked oat bran were investigated using small-amplitude oscillatory shear experiments. The oscillatory shear experiments were conducted before the shear-loop experiments. The measured oscillatory shear storage and loss moduli (G' and G'') for cooked oat bran and OATRIM-10 are illustrated in Figs. 6 and 7, respectively. For the data presented in Figs. 6 and 7, the contributions to G'' from the solvent ($\omega\eta_s$ where ω is the oscillation frequency and η_s is the solvent viscosity) have been eliminated (Ferry 1980).

The small-amplitude oscillatory shear data obtained for cooked oat bran are indicative of the behavior expected for a flexible coil in solution and do not provide evidence for any gel-like behavior. For linear chains in solution, the viscoelastic behavior is commonly described using the bead-spring model theories advanced by Rouse (1953) and Zimm (1956). The theories proposed by Rouse and Zimm were formulated for dilute solutions where the probability of chain overlap is negligible. For semidilute and concentrated solutions, such as those investigated here, the relaxation times are affected by chain-chain interactions. Corrections for the relaxation times in semidilute solutions have been developed and verified experimentally (Muthukumar and Freed 1978). The application of these molecular models is complicated for the systems discussed here because of the complex mixture of components in the solutions.

As an initial attempt to describe the solution viscoelastic properties of these materials, a general linear model was used (Bird et al 1977). In the GLV model, elements consisting of springs, which represent the elastic storage, and dashpots, which represent viscous dissipation, are added together in series to produce a multielement

mechanical presentation of a linear viscoelastic fluid. For small-amplitude oscillatory shear, G' and G'' can be expressed as:

$$G'(\omega) = \frac{\tau_1 \omega^2 \eta_0}{\zeta(\alpha)} \sum_{p=1}^N \frac{1}{k^{2\alpha} + \omega^2 \tau_p^2} \quad (3)$$

$$G''(\omega) - \omega\eta_s = \frac{\omega\eta_0}{\zeta(\alpha)} \sum_{p=1}^N \frac{k^\alpha}{k^{2\alpha} + \omega^2 \tau_p^2} \quad (4)$$

where τ_1 is the longest relaxation time, η_0 is the zero-shear viscosity, N is the total number of elements, $\zeta(\alpha)$ is the Riemann zeta function, and k and α are model parameters. The higher order relaxation times in the model are related to τ_1 as:

$$\tau_p = \frac{\tau_1}{k^\alpha} \quad (5)$$

It should be noted that when $\alpha = 2$, the GLV model becomes numerically equivalent to the Rouse model.

The GLV model, using Eqs. 3 and 4, was fitted to the experimental data of cooked oat bran using τ_1 , η_0 , and α as the input parameters. The calculations were conducted using Mathcad Pro software running on an Apple Macintosh 9600/200MP computer. The standard deviation of the fit was defined as the difference between the log of the measured storage moduli and the log of the predicted storage moduli. The best fit was chosen as the set of input parameters that minimized the standard deviation.

The results of the data fit are illustrated in Fig. 8. The experimental data were described to a reasonable degree by the GLV model with $\tau_1 = 0.28 \pm 0.03$ sec, $\eta_0 = 1 \pm 0.1$ Pa-sec, and $\alpha = 2.00 \pm 0.08$. The higher frequency G' data are well described by the GLV model; however, the lower frequency data deviate slightly from the model predictions. The G'' data are also reasonably well described by the GLV model over the frequency range studied. The quality of the fit between the experimental data and the GLV model could no doubt be improved by introducing the effects of molecular weight distribution (Carriere 1985) into Eqs. 3 and 4. In addition, the lower frequency G' data appear to possess a small plateau at ≈ 0.1 /sec. Oat bran is actually a mixture of several bio-

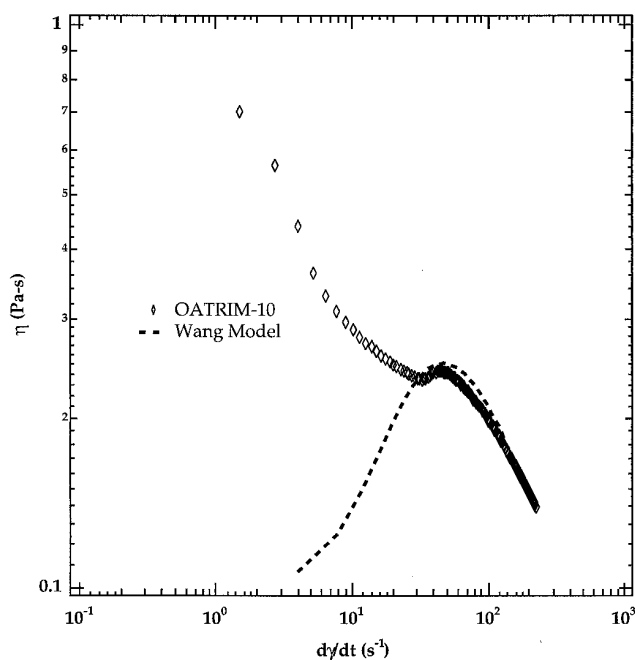


Fig. 5. Comparison of predictions of a theory of shear thickening (Wang 1992) with experimental data for OATRIM-10 at 25°C. Model predictions calculated using $r = 10$, $\bar{q} = 4.5$, and $\delta = 0.6$. Region before the shear-thickening regime is treated as a Newtonian fluid in the theory and cannot predict the initial shear-thinning region.

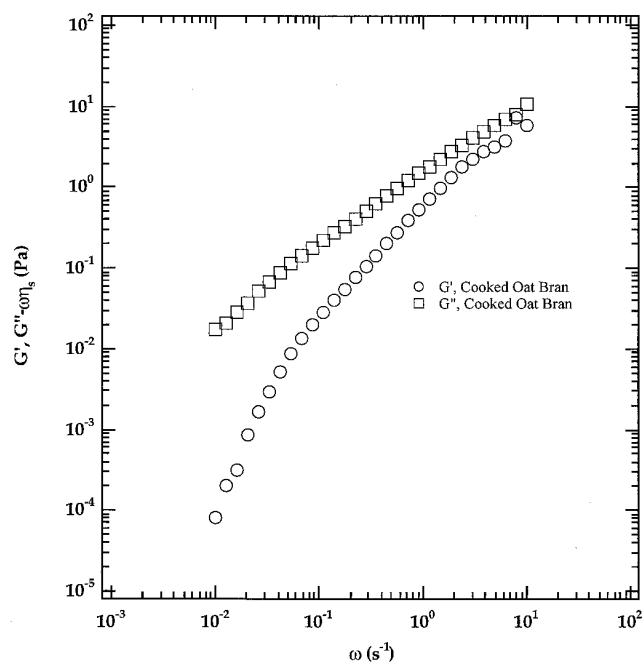


Fig. 6. Small-amplitude oscillatory shear data for cooked oat bran solutions at 25°C. Results for cooked oat bran resemble those expected for a flexible coil in solution. G' = elastic storage modulus, G'' = loss modulus, ω = oscillation frequency.

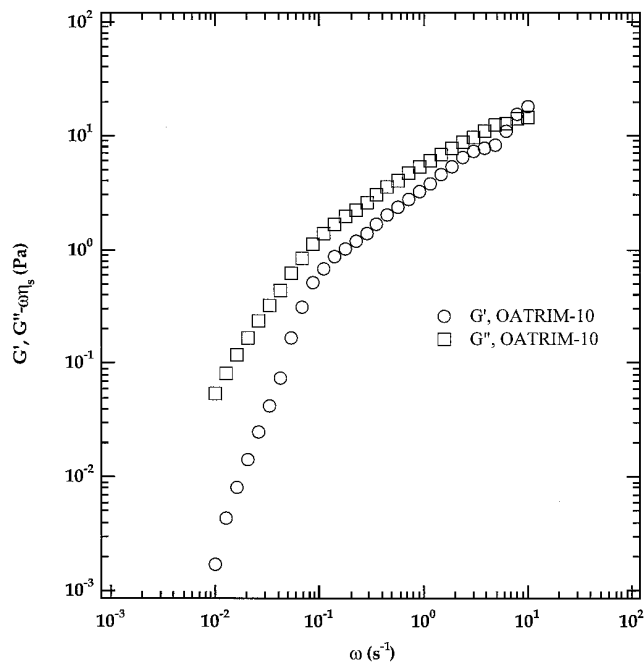


Fig. 7. Small-amplitude oscillatory shear data for OATRIM-10 solutions at 25°C. G' = elastic storage modulus, G'' = loss modulus, ω = oscillation frequency.

logical macromolecules, and the plateau is probably indicative of the longest relaxation time of one of the components.

The small-amplitude oscillatory shear data for OATRIM-10 are illustrated in Fig. 7. In contrast to the data obtained for cooked oat bran, the data for OATRIM-10 could not be fit using the GLV model. The frequency dependence of G' could be reproduced using a three-element Maxwell model, but the frequency dependence of G'' could not be replicated. OATRIM-10 exhibits a distinctive plateau centered at 10/sec, and the low frequency response of G' decreases with a much larger slope in frequency than is predicted by the GLV model. The plateau is caused by a distinct spacing in the relaxation time spectrum, in accord with that found, for example, with semiflexible macromolecules (Amis et al 1985; Carriere et al 1985, 1993). In this interpretation, the initial relaxation time constant represents the end-over-end translational mode of motion of the macromolecule, and the subsequent time constants represent the normal bending modes of motion of an elastic cylinder. For OATRIM-10, the plateau is probably caused by a spacing in the longest relaxation times of the constituent biopolymers that make up the mixture.

CONCLUSIONS

The solution rheological behavior of OATRIM-10 and cooked oat bran were investigated using both thixotropic loop and small-amplitude oscillatory shear experiments. The cooked oat bran exhibited shear-thinning behavior during a thixotropic loop experiment over a shear rate range of 0–250/sec. The shear-thinning behavior was reproducible during the application of a second thixotropic loop. In contrast, OATRIM-10 exhibited a region of shear-thinning behavior followed by shear-thickening behavior at 20–80/sec. At shear rates >80/sec, OATRIM-10 again behaved as a shear-thinning fluid. The shear-thickening and subsequent shear-thinning regions for OATRIM-10 could be described by a transient network model, indicating that the shear-thickening behavior is caused by a shear-induced entangled network, which is subsequently destroyed at high shear rates. Repeated thixotropic loop experiments displayed the shear-thickening region for OATRIM-10, indicating that the network structure formed is, at least par-

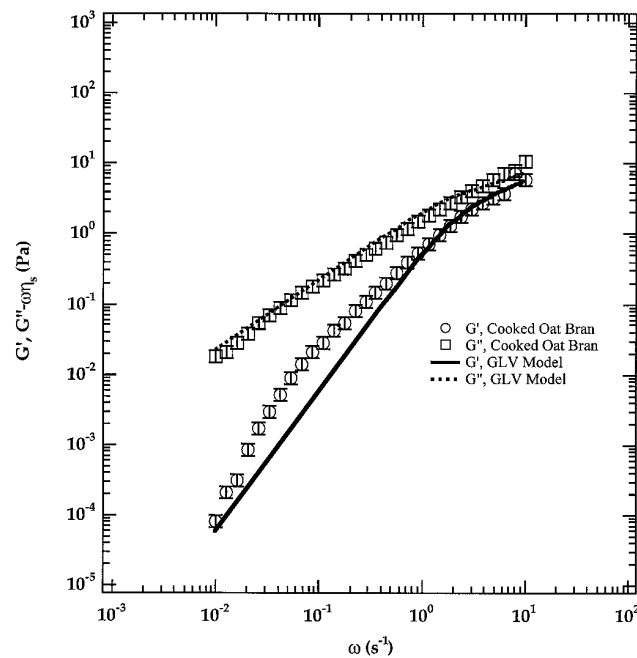


Fig. 8. Comparison of the experimental data for cooked oat bran obtained at 25°C from small-amplitude oscillatory shear experiments with the predictions from the generalized linear viscoelastic (GLV) model. Model predictions generated using $\tau_1 = 0.28 \pm 0.03$ sec, $\eta_0 = 1 \pm 0.1$ Pa-sec, and $\alpha = 2.00 \pm 0.08$. G' = elastic storage modulus, G'' = loss modulus, ω = oscillation frequency.

tially, disentangled at high shear rates and can be reformed during the imposition of a shear field.

Small-amplitude oscillatory shear data indicated that the component biopolymers in cooked oat bran behave as random coils in solution. The oscillatory shear data for cooked oat bran can be described reasonably well using a GLV model, but the oscillatory data obtained for OATRIM-10 could not. OATRIM-10 exhibits a plateau region that could be interpreted as a spacing between the longest relaxation times for the various biopolymers that constitute the material. Additional work is required to understand the complicated rheological behavior of (1–3),(1–4)- β -D-glucan and amylopectin solutions such as OATRIM-10.

The research described here constitutes a starting point for understanding the flow behavior of these complicated oat-based systems. Future research will be directed at investigating the cause of the observed shear-thickening behavior of the (1–3),(1–4)- β -D-glucan and amylopectin solutions. In addition, future efforts will also explore the utility of molecular-based models to predict the solution viscoelastic properties of complicated solution mixtures such as OATRIM-10.

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