COMPLEX VISCOSITY-TEMPERATURE MASTER CURVE OF CORNSTARCH DISPERSION DURING GELATINIZATION

W.H. YANG and M.A. RAO[1]

Department of Food Science & Technology
Cornell University-Geneva
Geneva, NY 14456-0462

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ABSTRACT

Complex viscosity (η) of 8% cornstarch dispersion during gelatinization at oscillatory frequencies (ω): 0.63-47.12 (rad s^{-1}) was independent of the heating rates: 1.6-6.0 (°C min^{-1}) when temperature was the independent variable. The influence of ω on η* was scaled by a frequency shift factor resulting in reduced complex viscosity (η^*_R) versus temperature (60-95C) master curve. Similar η^*_R master curves were derived for data on 6% and 3.5% dispersions. A modified Cox-Merz rule correlated the complex and apparent viscosity data, and a power law model described the effect of concentration of the gelatinized structures.*

INTRODUCTION

Gelatinization of starch in excess water is encountered in many foods, such as soups, salad dressings, gravies, and sauces. Because of the drastic increase in magnitude of the apparent viscosity (η_a) during transition from a starch dispersion (STD) to a fully gelatinized starch, rheological behavior plays an important role in heat transfer and thermal processing of such foods. For example, Ball and Olson (1957) discovered that broken heating phenomena during thermal processing of starch containing foods in cans was related to increase in viscosity due to starch gelatinization. However, because viscosity data were obtained only at 60C (140F) with a Scott orifice viscometer, direct correlations between broken heating and starch gelatinization parameters were not established.

Under isothermal conditions, the apparent first-order reaction rate model and the Arrhenius equation described the rate of gelatinization and the influence of temperature on the rate, respectively (Kubota *et al.* 1979; Lund 1984; Okechukwu *et al.* 1991). Because of the important role of temperature history,

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[1] Corresponding author, Phone: (315) 787-2266, Fax: (315) 787-2397, E-mail: mar2@cornell.edu

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changes in η_a during gelatinization can not be described accurately by the models obtained under isothermal conditions. In addition to the temperature of the starch sample, shear rate or dynamic frequency has a significant effect on η_a or complex viscosity (η^*), respectively.

Dolan *et al.* (1989) developed a model for η_a during starch gelatinization under nonisothermal conditions that had an exponential function of the temperature-time history and Arrhenius equation to describe the gelatinization rate. The form of the model for constant shear rate and starch concentration is:

$$\eta_{dim} = \frac{\eta_a - \eta_{ug}}{\eta_\infty - \eta_{ug}} = [1 - \exp(-k\psi)]^\alpha \qquad (1)$$

where,

$$\psi = \int T(t) \exp\left(\frac{-E_g}{RT(t)}\right) dt \qquad (2)$$

where, η_{dim} is dimensionless apparent viscosity, η_a is apparent viscosity at a specific time during heating, η_{ug} is apparent viscosity of ungelatinized dispersion, and η_∞ is the highest magnitude of η_a during gelatinization. The model was based on earlier work on viscosity of protein doughs by Morgan *et al.* (1989) and was extended to include the influence of shear rate, temperature, concentration, and strain history (Dolan and Steffe 1990). Data obtained using back extrusion and mixer viscometers were used to evaluate the models. The activation energy of gelatinization (E_g) depended on the heating temperature (Dolan *et al.* 1989) and some of the factors affecting viscosity were negligible (Dolan and Steffe 1990). The studies of Dolan *et al.* (1989) and (Dolan and Steffe 1990) provide valuable guidelines on the factors affecting apparent viscosity of STDs during heating.

η_a versus temperature profiles of dispersions of a specific starch (e.g., corn, bean) are usually similar over narrow ranges of starch concentrations (Launay *et al.* 1986; Champenois *et al.* 1997), so that it may be possible to develop simple equations for estimating η_a over specific ranges of concentration. Because η^* data are obtained at low strains with minimal alteration of the STD structure, they provide unique opportunities for studying applicable models. Further, empirically obtained frequency shift factor (Ferry 1980) has been used successfully in time-temperature superposition studies on food polymer dispersions (Lopes da Silva *et al.* 1994), and the applicability of similar, if not identical, scaling of frequency needs to be explored for STDs.

For fluid dynamics and heat transfer investigations related to food processing, the necessary η_a data may be obtained from models developed for η^* data using relationships based on the Cox-Merz rule (Rao 1992; Rao and

Cooley 1992). Yang (1997) used these procedures to investigate the role of temperature and shear dependent viscosity on heating rates of a canned 3.5% corn STD. The objective of this study was to investigate the influence of different heating rates and dynamic frequencies on values of η^* during the heating phase (gelatinization) of 6% and 8% corn STDs, and to determine the relationship between η^* and η_a obtained from steady shear rate data. In addition, utilizing additional data on a 3.5% corn STD, the effect of starch concentration on η^* was evaluated.

MATERIALS AND METHODS

Corn STD

An unmodified corn starch (American Maize-Product Company, Hammond, IN) with the commercial specifications: 10.0% moisture, 5.5 pH, and 99.5% granulation through US 200 mesh was used. Starch dispersions, 8%, 6%, and 3.5% w/w, were prepared by mixing with distilled water and held overnight (~16h) to allow starch hydration. Extensive rheological tests were conducted on the 8% corn STD because the concentration was sufficiently high to obtain reliable magnitudes of η^* over the temperature range of interest: 60-95C. After the experimental and data analysis procedures were established with the 8% corn STDs, additional tests were conducted with 6.0% and 3.5% corn STDs.

Dynamic Rheological Data

Dynamic rheological data were obtained using 4 cm diameter parallel plate (gap 500 μm) geometry of a Carri-Med CSL-100 rheometer (TA Instruments, New Castle, DE). A plate-cone geometry was also tried, but the narrow gap (52 μm at the center) contributed to capillary suction of the few drops of paraffin oil that were placed on the exposed edge of the plate to minimize loss of water vapor during heating into the corn STD. In contrast, with the parallel plate geometry, the drops of paraffin oil placed soon after the storage modulus (G') reached values that were measurable did not penetrate into the corn STD. Magnitudes of the η^* were computed by the rheometer software and the parameters in Eq. 1 (Dolan *et al.* 1989) were calculated by nonlinear regression based on a modified Levenberg-Marquardt algorithm (subroutine ZXSSQ, IMSL, Inc., Houston, TX) and on minimum residual sum of squares.

Two critical factors for obtaining reliable η^* data were: (1) control of the sample volume (0.80 mL) that was loaded carefully on the bottom rheometer plate and removal of excess dispersion from the edge of the top plate, and (2) minimizing water evaporation from the sample when the temperature was high and avoiding displacement of paraffin oil by tiny air bubbles from the edge of the top plate. Because of the narrow gap (500 μm) between the parallel plates, settling of starch granules was not a problem as might have been in a concentric cylinder geometry and the structure formation was not disrupted because of the low magnitude of oscillatory strain.

Heating Rates

The effect of different heating rates on η^* of the 8% corn STDs was studied at 1.26 rad s^{-1}, 1% strain, as the corn STDs were heated from 60C to 92C at different heating rates: 1.6, 2.1, 2.5, 3.0, 4.2 and 6.0C min^{-1} and holding at 92C for about 2 min. Additional experiments were conducted by heating the corn STDs to 95C and holding for 5.5 min. The Peltier control system of the Carri-Med rheometer permitted reliable temperature control of the corn STD samples.

Dynamic Frequencies and Starch Concentrations

In order to examine the effect of ω, η^* data were obtained at: 0.63, 1.88, 5.34, 6.28, 7.85, 12.57, 18.85, 31.41, and 47.12 rad s^{-1} as the 8% corn STDs were heated from 60 to 92C over 15 min (2.1C min^{-1}). To confirm the validity of the rheological model over a wide range of test conditions, heating times other than 15 min and different dynamic frequencies were also employed: 5 min at 3.14 rad s^{-1}, 7.5 min at 3.14 rad s^{-1} and 4.71 rad s^{-1}, 10 min at 1.57 rad s^{-1}, 3.14 rad s^{-1}, and 4.71 rad s^{-1}, and 12.5 min at 3.14 and 4.71 rad s^{-1}. Experiments were also conducted at high ω values: 62.83 rad s^{-1} (10 Hz) and 78.54 rad s^{-1} (12.5 Hz) at the heating rate of 2.1C min^{-1} as the 8% corn STDs were heated from 60 to 95C. The effect of concentration was investigated by performing experiments with 6.0% and 3.5% corn STDs at 3.14, 12.57, and 47.12 rad s^{-1}.

Relationship Between η^* and η_a of Gelatinized Corn STDs

Steady shear data on gelatinized 8% and 6.0% corn STDs were obtained with the Carri-Med CSL-100 rheometer using steady shear rates ($\dot{\gamma}$) in the same range as the frequencies (ω) in dynamic tests. These data were obtained at 25C on the 8% corn STD, and at 25C and 75C on the 6.0% corn STD. A modified Cox-Merz rule was used to correlate the apparent viscosity-shear rate and complex viscosity-oscillatory frequency data using log-log plots (Rao and Cooley 1992):

$$\eta^*(\omega)=C[\eta_a(\dot{\gamma})]^{\alpha}\big|_{\omega=\dot{\gamma}} \tag{3}$$

where, $\dot{\gamma}$ is complex viscosity (Pa s), η_a is apparent viscosity (Pa s), ω is frequency (rad s^{-1}), $\dot{\gamma}$ is shear rate (s^{-1}), C is a constant, and α is shift factor.

RESULTS AND DISCUSSION

A heated starch paste may be considered to be a composite material consisting of a macromolecular solution, sometimes containing either only leached amylose or both amylose and amylopectin, and granules which may be in various states from swollen granules to disrupted granules depending on factors such as temperature, water content or mechanical treatment (Eliasson 1986; Ellis *et al.* 1989). Its rheological properties depend on three factors (Eliasson 1986): (1) starch granules (dispersed phase): concentration, granule size and size distribution, shape of granules, swelling pattern of the starch, granule rigidity and deformability, (2) amylose/amylopectin matrix (continuous phase): viscoelasticity of the phase, amount and type of amylose/amylopectin which has leached from the granules, entanglements, and (3) interactions between the components: surface of starch granule, granule-amylose/amylopectin interactions, granule-amylose/amylopectin-granule interactions, granule-granule contact. Therefore, any change in the viscoelastic behavior of a starch-based medium may be interpreted as the result of changes in at least one factor of this list. In addition, the pasting conditions can strongly influence the rheological behavior of any corn STD (Doublier 1987). In this regard, we have controlled accurately the heating rate of the dispersion.

Model of Dolan *et al.* 1989

For the η^* versus time data obtained at 0.2 Hz (1.26 rad s^{-1}) on the 8% corn STD, magnitudes of E_g, k and α in Eq. 1 and 2, over the range of heating rates studied were: 240 kJ/mol, 1.95 E+30, and 0.668, respectively. These values are in reasonable agreement with those of Dolan *et al.* (1989): 210 kJ/mol, 0.846 E+26, and 0.494, respectively, considering that η^* instead of η_a and starch from a different source were used. The value of R^2 was relatively high ($R^2=0.96$) but the experimental data showed deviations from the predicted curve for time-temperature history values 5.0×10^{-31} to 1.25×10^{-30} K s (Fig. 1). Equations 1 and 2 with the calculated constants can be used to obtain apparent viscosity from any temperature versus time history during gelatinization, provided that the initial and maximum viscosities are known. However, because of granule sedimentation, the former is difficult to determine experimentally. Better fit may be obtained by expressing E_g as a function of temperature and the Arrhenius relationship to be temperature-time history dependent (Dolan *et al.* 1989; Dolan and Steffe 1990).

Viscosity-temperature Profiles

Influence of Heating Rates. Typical η^* versus time data at heating rates 1.6 to 6.0C min^{-1} are shown in Fig. 2. The curves shown are representative of

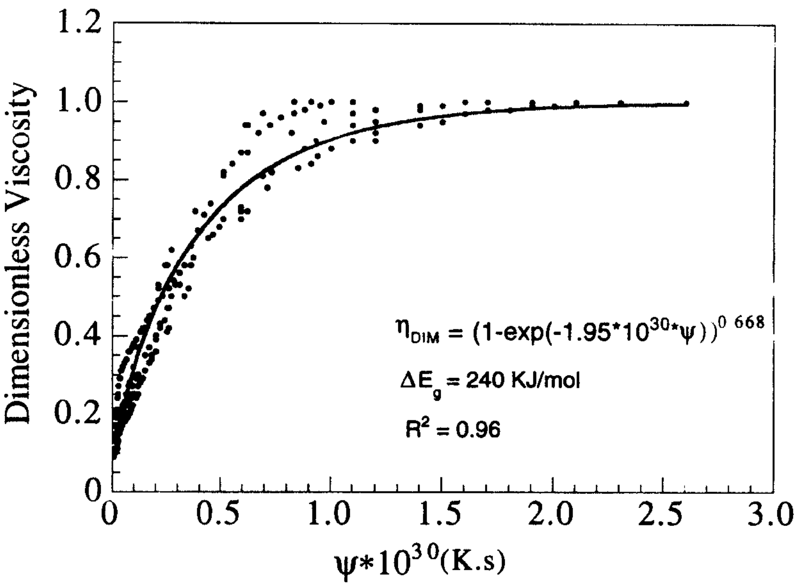

FIG. 1. DIMENSIONLESS VISCOSITY VERSUS TEMPERATURE-TIME HISTORY CURVE OF 8% CORN STARCH DISPERSION AT DIFFERENT HEATING RATES BASED ON DOLAN *ET AL.* (1989)

at least three runs for each heating rate. To analyze the effects of time and temperature on viscosity, η^* data obtained at a fixed frequency (1.26 rad s^{-1}) but at different heating rates were plotted against temperature (Fig. 3) instead of time and most data collapsed to a single curve. The largest difference in the magnitudes of complex viscosity between the highest (6.0C min^{-1}) and the lowest (1.6C min^{-1}) heating rates was about 25%. Although heating time plays an important role in determining the extent of starch gelatinization in experiments performed under isothermal heating conditions, the complex viscosity of a corn STD over the range of heating rates: 1.6C min^{-1} to 6.0C min^{-1} can be described as a simple function of temperature. Similar results were reported by Duisburg (1981) with a Brabender Viscograph at four different heating rates: a standard rate of 1.5C min^{-1}, and 2.0, 2.5, and 3.0C min^{-1}.

At low heating rates (e.g., 2.1C min^{-1}), a two stage increase in η^* with temperature can be seen (Fig. 3): the first peak in viscosity, usually located between 55 and 75C is due to a limited swelling of the starch granules (Doublier 1981; Kokini *et al.* 1992; Champenois *et al.* 1998) that leads to an increase in the granule-granule interactions due to their larger dimensions. The slight

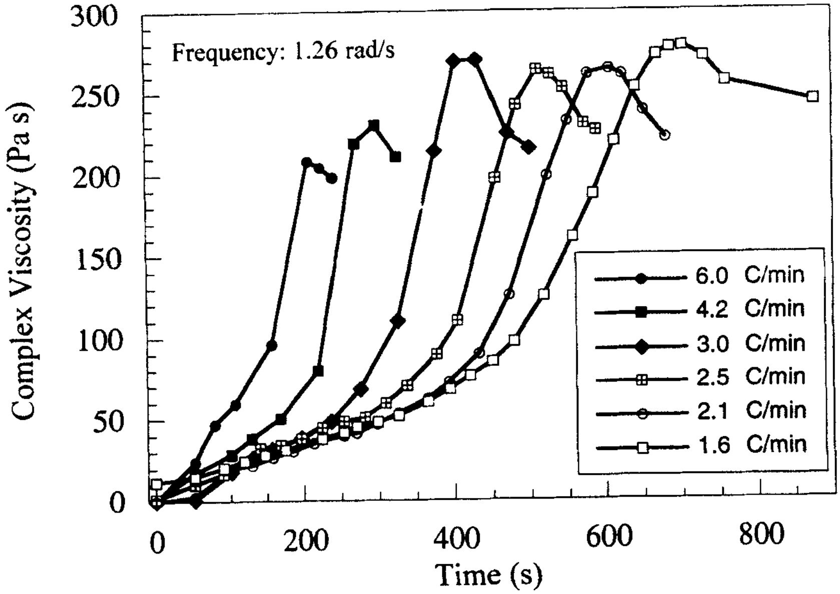

FIG. 2. COMPLEX VISCOSITY VERSUS HEATING TIME CURVE OF 8% CORN STARCH DISPERSION AT A FREQUENCY OF 1.26 RAD S^{-1} AND SEVERAL DIFFERENT HEATING RATES

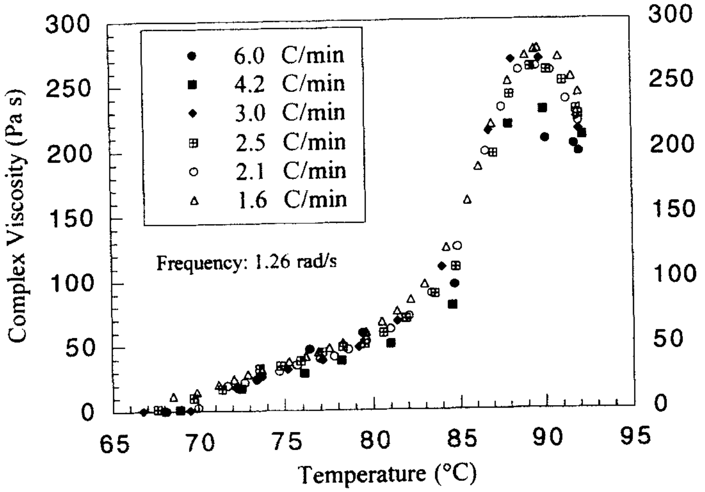

FIG. 3. COMPLEX VISCOSITY VERSUS HEATING TEMPERATURE CURVE OF 8% CORN STARCH DISPERSION AT A FREQUENCY OF 1.26 RAD S^{-1} AND DIFFERENT HEATING RATES

decrease that occurred after the first increase, was found to be concomitant with end of the DSC endothermic peak due to melting of crystallites (Eliasson 1986). Because of continued swelling of the starch granules, the viscosity increased until about 90C leading to the formation of a transient network of granules touching each other and sometimes resulting in granule disruption (Blanshard 1979) and amylose leaching (Eliasson 1986). Similar viscosity versus temperature behavior has been noted for a number of cereal starches (Launay *et al.* 1986; Doublier 1987).

After the peak viscosity was reached, increasing the temperature further resulted in decrease in viscosity (Eliasson 1986; Doublier 1987; Kokini *et al.* 1992; Reddy 1992) and has been attributed to rupture of the granules. Two distinct types of curves were obtained after the peak (Fig. 4) viscosity was reached depending on whether: (1) the temperature was maintained at 92C for about 2.5 min or at 95C for about 5.5 min, or (2) the temperature was increased up to 95C continuously after the peak viscosity temperature. The former was characterized by a slower rate of decrease in η^* after the temperature was held constant and the latter by a rapid rate of decrease in η^* up to the final temperature (95C) suggesting that the network structure formed during gelatinization was drastically weakened by increase in the temperature.

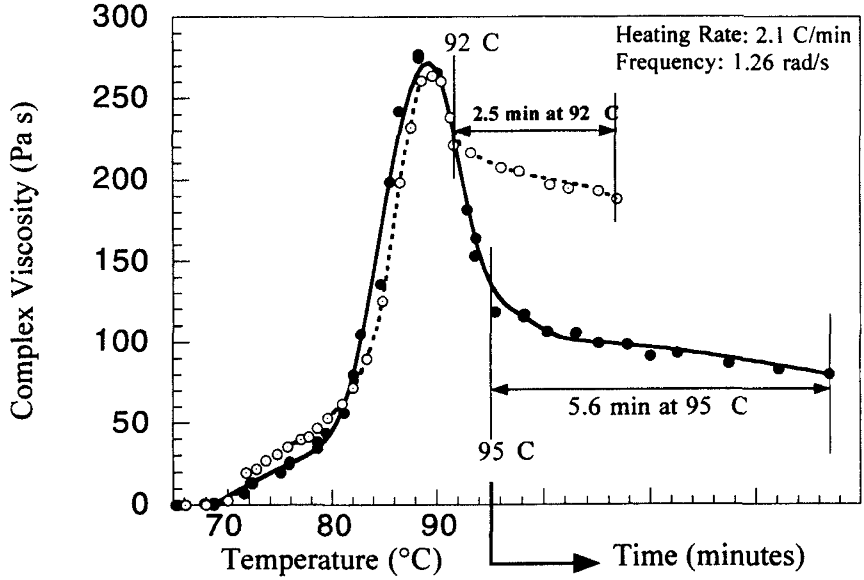

FIG. 4. COMPLEX VISCOSITY (η^*) OF 8% CORN STARCH DISPERSION AT A FREQUENCY OF 1.26 RAD S^{-1} AND HEATING RATE OF 2.1C MIN^{-1}

After the peak viscosity temperature was reached, dispersions were held at 92C and 95C.

It is theoretically possible that at very high heating rates, starch granule swelling would be limited by moisture diffusion. Another limitation would be to obtain reliable rheological data at a high heating rate because the temperature range over which viscosity of the corn STD is heated would be covered in a short time period.

Functional Viscosity Model

Influence of Frequency. At a fixed heating rate of 2.1C $\min^{-1}$ and over the range of frequencies employed from 1.26 to 47.12 rad s^{-1}, the profiles of η^* at a specific frequency versus temperature were similar (Fig. 5a), so that by choosing an arbitrary reference frequency (ω_r), all the η^* versus temperature curves at the different frequencies could be reduced to a single curve. Several different frequencies were suitable for use as ω_r. The resulting master curve of reduced complex viscosity $\eta_R^* = \eta^*\left(\frac{\omega}{\omega_r}\right)$ versus temperature obtained using $\omega_r = 6.28$ rad s^{-1} (1.0 Hz) at the heating rate of 2.1C $\min^{-1}$ is shown in Fig. 5b. Because logarithmic coordinates were used for the frequency scale in time-temperature superposition studies (Lopes da Silva *et al.* 1994), it was fortuitous that a linear frequency ratio was satisfactory over the frequency range: 1.26 to 47.12 rad s^{-1}. Indeed, when data from a limited number of experiments conducted at high ω values: 62.83 rad s^{-1} (10 Hz) and 78.54 rad s^{-1} (12.5 Hz) at the heating rate of 2.1C $\min^{-1}$ were considered, scaling of frequency could be achieved (Fig. 5b) by a more general relationship:

$$\eta_R^* = \eta^*\left(\frac{\omega}{\omega_r}\right)^{\beta} \qquad (4)$$

where, $\left(\frac{\omega}{\omega_r}\right)^{\beta}$ is the frequency shift factor and the magnitude of the exponent, β, needs to be determined from experimental data at ω and ω_r. For complex viscosity data at 62.83 rad s^{-1} (10 Hz) and 78.54 rad s^{-1} (12.5 Hz), with $\omega_r = 6.28$ rad s^{-1}, values of β were 0.913 and 0.922, respectively. It is noteworthy that both increasing and decreasing segments of the viscosity versus temperature data at all the frequencies were reduced to a single curve. Complex viscosity data on 8% corn STDs from experiments conducted at different heating rates and frequencies were also superposed using 6.28 rad s^{-1} (1.0 Hz) as ω_r (Fig. 6). Because data from Fig. 5b are also included (shown with a plus sign), Fig. 6 is the master curve of reduced complex viscosity η^*_R data obtained on the 8% corn STD at different heating rates and frequencies.

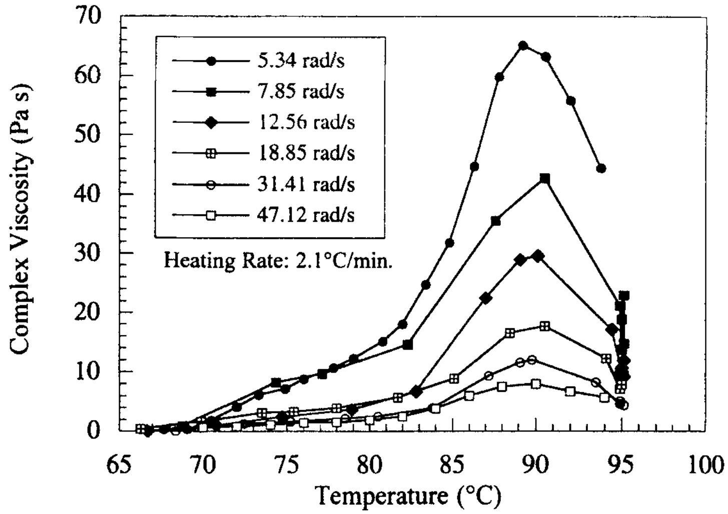

FIG. 5A. COMPLEX VISCOSITY OF 8% CORN STARCH DISPERSION AT DIFFERENT FREQUENCIES AND 2.1C MIN^{-1} HEATING RATE AS A FUNCTION OF TEMPERATURE DURING GELATINIZATION

Note that the viscosity profiles at different frequencies are similar.

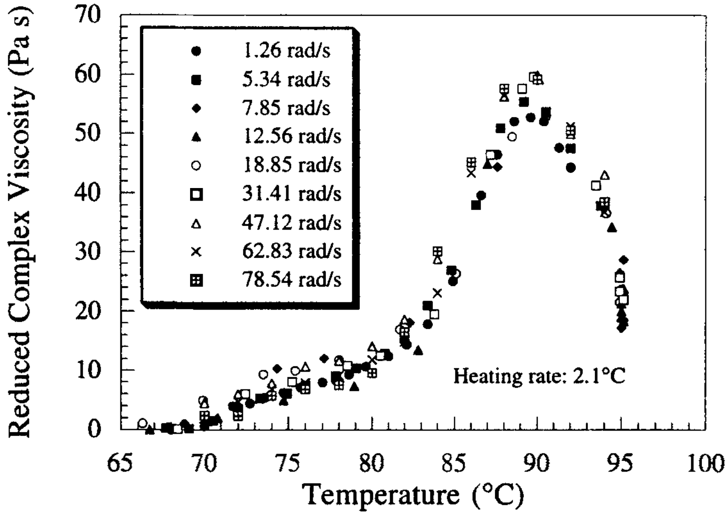

FIG. 5B. MASTER CURVE OF REDUCED COMPLEX VISCOSITY OF 8% CORN STARCH DISPERSION AT SEVERAL OSCILLATORY FREQUENCIES AND 2.1C MIN^{-1} HEATING RATE AS A FUNCTION OF TEMPERATURE DURING GELATINIZATION

The data in Fig. 5a were reduced to single curve using the frequency shift factor $\left(\frac{\omega}{\omega_r}\right)^{\beta}$ with

$\omega_r = 6.28$ rad s^{-1}.

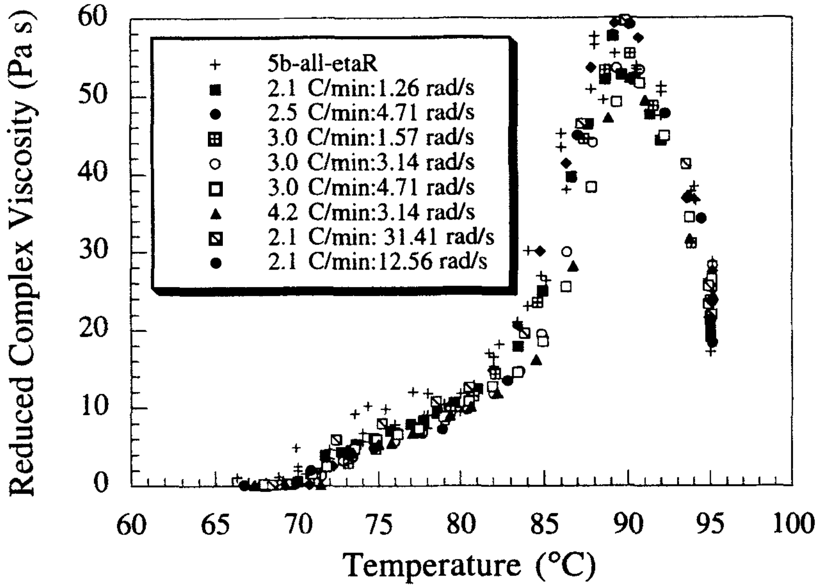

FIG. 6. MASTER CURVE OF REDUCED COMPLEX VISCOSITY OF 8% CORN STARCH DISPERSION AT SEVERAL OSCILLATORY FREQUENCIES AND HEATING RATES

All data were superposed using the frequency shift factor $\left(\frac{\omega}{\omega_r}\right)^{\beta}$ with $\omega_r = 6.28$ rad s^{-1}.

The reduced complex viscosity versus temperature master curves could not be described satisfactorily by any one equation. For accurate description of similar data on 3.5% corn STDs for computer simulation, Yang (1997) found that three different equations were necessary over different ranges of temperature. The superposition of the η^* versus temperature data suggests that the molecular mechanisms involved were similar at the different heating rates and oscillatory frequencies used, and depended mainly on the temperature history of the corn STD. It also suggests that the studied corn STDs exhibited thermorheologically simple behavior (Plazek 1996), i.e., that all the molecular mechanisms involved had the same temperature dependence.

Complex Viscosity Data of 6% and 3.5% Corn STDs

The above modeling procedure was found to be applicable to the η^* data obtained on 6% corn STDs at a heating rate of 3.0C min^{-1} and at dynamic frequencies: 3.14, 12.57 and 47.12 rad s^{-1} (Fig. 7). As expected, magnitudes of the complex viscosities of 6.0% corn STDs were lower than those of 8% corn STD. However, the gelatinization initiation and peak viscosity temperatures were close to those of the 8% corn STD. The η^* versus temperature data profile of the 6% corn STD was similar to that of the 8% corn STD: a continuous increase in η^* with temperature up to the peak followed by a drastic decrease. The same modeling procedure was applied to 3.5% corn STDs and the results used to numerically simulate heat transfer in a can containing a 3.5% corn STD (Yang 1997).

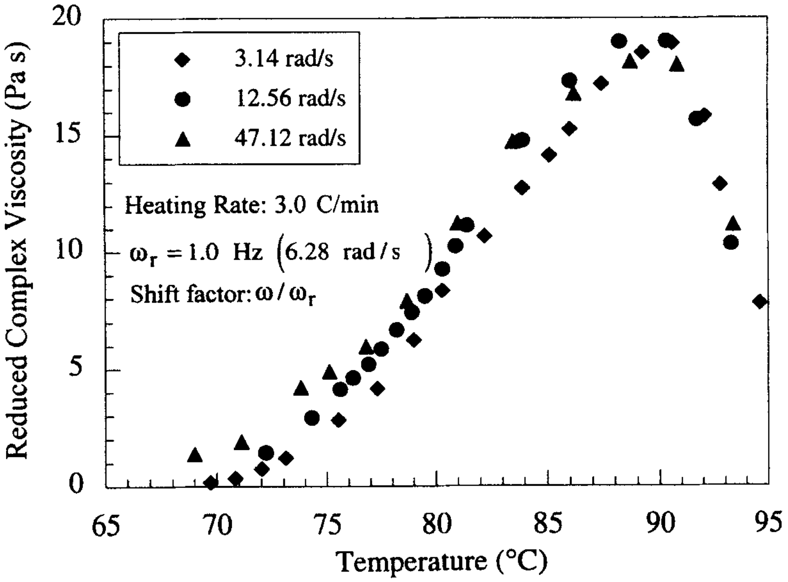

FIG. 7. MASTER CURVE OF REDUCED COMPLEX VISCOSITY OF 6% CORN STARCH DISPERSION AT SEVERAL OSCILLATORY FREQUENCIES

All data were superposed using the frequency shift factor $\left(\frac{\omega}{\omega_r}\right)^{\beta}$ with $\omega_r = 6.28$ rad s^{-1}.

Figure 8 shows the values of complex viscosity at six temperatures plotted as a function of concentration. Best fits were obtained when power relationships were used between viscosity data at 90C, 88C, and 86C and concentration, with R^2 of 0.99, 0.99, and 0.96, respectively:

$$\eta^* = A_c(C_s)^{B_c} \tag{5}$$

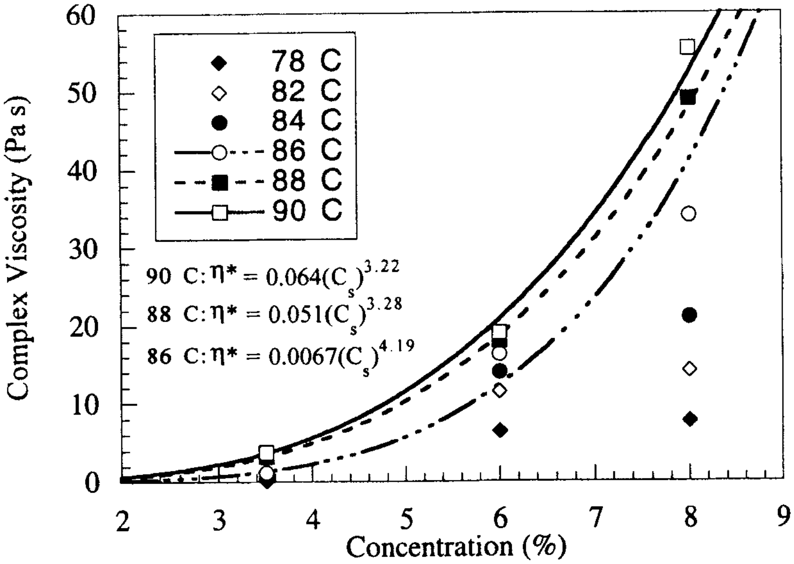

FIG. 8. COMPLEX VISCOSITY-CONCENTRATION CURVES OF CORN STARCH DISPERSION AT SPECIFIC TEMPERATURES DURING GELATINIZATION

where, A_c and B_c are constants, and C_s is starch concentration (w/w, %). Because of the transition from liquid-like to elastic-like behavior, the power model could not describe the effect of concentration at temperatures lower than 86C. For data at the temperatures: 90C, 88C, and 86C, the constant A_c was: 0.064, 0.051, and 0.0067; and the power coefficient was: 3.22, 3.28, and 4.19, respectively.

Relationship Between η^* and η_a

For the 8.0% corn STDs at 25C, the constant C and shift factor α in the modified Cox-Merz rule (Eq. 3) were found to be 5.72 and 0.97, respectively. For the 6% corn STDs, the parameters C and α were 3.68 and 0.98 at 25C, and 3.89 and 1.03 at 75C, respectively, suggesting that temperature does not affect considerably the parameters C and α. Figure 9 illustrates the applicability of Eq. 3 (Rao and Cooley 1992) to complex and apparent viscosities of the 6% corn STD.

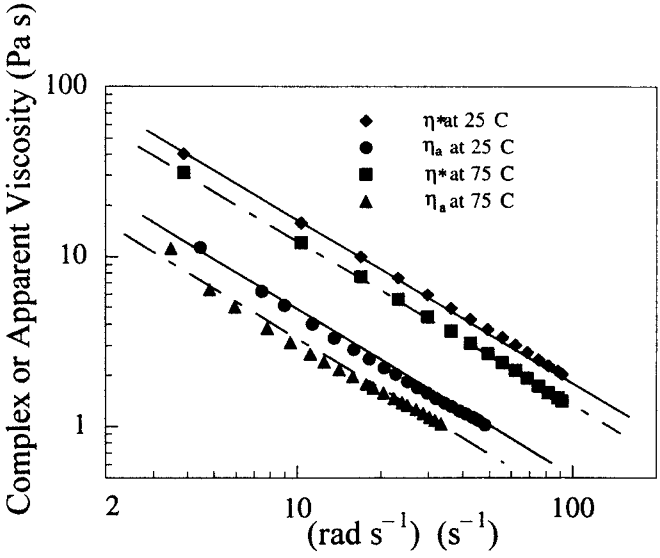

FIG. 9. COMPLEX VISCOSITY-FREQUENCY AND APPARENT VISCOSITY-SHEAR RATE DATA OF 6% CORN STARCH DISPERSION THAT FOLLOWED MODIFIED COX-MERZ RULE

CONCLUSIONS

Using temperature (60–95C) as the independent variable and shift factors based on a reference frequency, a master curve was developed for reduced η^* of a 8% corn STD. Similar reduced η^* versus temperature master curves were also developed for 6% and 3.5% corn STDs. The master curves at each starch concentration suggest that the studied corn STDs exhibited thermorheologically simple behavior, i.e., that over the studied range of heating rates and dynamic frequencies, all the molecular mechanisms involved had the same temperature dependence. Whether corn STDs at other concentrations and other starch dispersions also exhibit such a behavior needs to be studied in the future. The effect of concentration could be described by a power relationship when the elastic behavior was predominant during gelatinization. Relationships between η^* and η_a based on a modified Cox-Merz rule were derived that can be used to estimate values of the latter.

NOTATION

A_c	constant in Eq. 5 (Pa s)
B_c	constant in Eq. 5 (–)

C	constant in Eq. 3
C_s	starch concentration, w/w, %
E_g	activation energy of gelatinization (J mole^{-1})
G'	storage modulus (Pa)
G''	loss modulus (Pa)
k	reaction transmission coefficient, $(\text{K s})^{-1}$
R	gas constant (8.314 J/mol K); radius of can (m)
STD	starch dispersion
T	temperature of fluid (°C)

Greek Letters

α	shift factor in Eq. 3
β	exponent of frequency shift factor
η_a	apparent viscosity (Pa s)
$\eta_{\dim}$	dimensionless apparent viscosity
η^*	complex viscosity (Pa s) $=(G^*/\omega)$, where $G^* = \sqrt{(G')^2+(G'')^2}$
η^*_R	reduced complex viscosity $= \eta^*\left(\frac{\omega}{\omega_r}\right)^{\beta}$
η_{ug}	ungelatinized apparent viscosity (Pa s)
η_∞	highest magnitude of η_a during gelatinization (Pa s)
ψ	Integral of time-temperature history
$\dot{\gamma}$	shear rate (s^{-1})
ω	frequency (rad s^{-1})

ACKNOWLEDGMENTS

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