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# Influence of sucrose on the rheology and granule size of cross-linked waxy maize starch dispersions heated at two temperatures

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## Abstract

Cross-linked waxy maize (CWM) starch dispersions (STDs) of concentration 50 g $kg^{-1}$ were heated in sucrose solutions containing 0–600 g $kg^{-1}$ (g sucrose/kg dispersion) at 85 °C at low shear and in intermittently agitated cans at 110 °C. The STDs heated in 0–300 g $kg^{-1}$ sucrose exhibited antithixotropic behavior, while those heated in 400–600 g $kg^{-1}$ sucrose exhibited thixotropic behavior. The mean starch granule diameter of the starch dispersions did not show strong dependence on sucrose concentration. The dispersions, especially those with high sucrose concentrations and heated at 110 °C, exhibited $G'$ versus frequency ($\omega$) profiles of gels. The STDs exhibited first normal stress differences that increased in magnitude with the concentration of sucrose. Values of the first normal stress coefficient of canned dispersions calculated from dynamic rheological data plotted against $\omega$ and experimental values plotted against shear rate of some of the STDs overlapped.

*Keywords:* Cross-linked waxy maize starch; Sucrose; Rheology; Heating temperature; Viscoelastic behavior

## 1. Introduction

Starch occurs in the form of a partially crystalline, birefringent granule which consists of two chemically identical components: the linear α-(1 → 4) glycosidically linked amylose and the dendroidically branched amylopectin with α-(1 → 6) glycosidic bonds forming the branching points (Aberle, Burchard, Vorwerg, & Radosta, 1994). During continuous heating, an unmodified aqueous starch paste loses viscosity relatively easily producing weak bodied, stringy, and cohesive pastes. Cross-linking treatment is intended to add chemical bonds at random locations in a granule which stabilize the granules and strengthen the relatively tender starch. Cross-linked waxy maize (CWM) starch pastes are more viscous and heavy bodied, and are less likely to breakdown with extended cooking times, increased acid content or severe agitation (Langan, 1986).

Rheological properties reflect structure of a material. One may view concentrated gelatinized starch dispersions (STDs) as microgel systems (Evans & Lips, 1992; Ketz, Prud'homme, & Graessley, 1988) whose flow and viscoelastic behavior are strongly influenced by the physical state of the granules (Evans & Lips, 1992; Okechukwu & Rao, 1995). The net force between the granules is attractive and complex, and more important than Brownian diffusion and hydrodynamic interaction. Therefore, heated starch dispersions are often non-Newtonian fluids that may also exhibit yield stress at low shear rates (Christianson & Bagley, 1984; Evans & Haisman, 1979; Giboreau, Cuvelier, & Launay, 1994).

Since granules in gelatinized STDs have a size distribution, rheological behavior of the dispersions is affected by their size, size distribution and shape, as well as interaction between the granules, continuous phase viscosity, and rate and time of deformation (Rao, Okechukwu, Da Silva, & Oliveira, 1997). The flow behavior of heated STDs can be described by flow models whose parameters can be used to examine the behavior of different STDs. When yield stress is exhibited by an STD, the Herschel–Bulkley (HB) model can be used to describe the shear rate–shear stress data:

$$\sigma - \sigma_0 = K\dot{\gamma}^n \tag{1}$$

where $\dot{\gamma}$ is shear rate ($s^{-}1$), $\sigma$ is shear stress (Pa), $n$ is the flow behavior index, $K$ is the consistency index, and $\sigma_0$ is yield stress. The HB model reduces to the power law model when

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the yield stress of a sample is negligible:

$$\sigma = K\dot{\gamma}^n \tag{2}$$

Heated CWM STDs exhibited the somewhat rare time dependent shear-thickening behavior (antithixotropic) (Chamberlain, Rao, & Cohen, 1999; Tattiyakul & Rao, 2000). Thus, it was of interest to determine if heating an STD in the presence of sucrose has an effect on such behavior.

Addition of sucrose affected the rheological behavior of the dispersions of several starches: wheat (Al-Malah, Azzam, & Abu-Jdayil, 2000; Cheer & Lelievre, 1983), acorn (Aee, Hyang Hie, & Nishinari, 1998), potato (Evans & Haisman, 1982), corn (Evageliou, Richardson, & Morris, 2000), potato and corn blends (Sikora, Mazurkiewicz, Tomasik, & Pielichowski, 1999), and sago (Ahmad & Williams, 1999). Ahmad and Williams explained the influence of the sugars on the structure of the dispersions in terms of inhibition of chain organization. The sugar molecules with equatorial hydroxyl groups (e.g. ribose) prevent chain reordering, making the whole structure weaker, while those with axial hydroxyl groups (e.g. fructose) have the opposite effect. However, CWM starch allows for studying the role of the starch granule in heated STDs without the influence of leached amylose.

Hoseney (1998) noted that in DSC endotherms of starch, addition of sucrose and other sugars to wheat starch increased the gelatinization initiation temperature. Therefore, the specific effects of sucrose on a STD may depend on variables, such as the type of starch and sucrose concentration, and on the heating temperature.

Small amplitude oscillatory (dynamic) tests have been used to characterize the viscoelastic behavior of STDs in several studies (Da Silva, Oliveira, & Rao, 1997; Evans & Lips, 1992). Another measure of viscoelastic behavior is the first normal stress difference; but few studies have reported encountering first normal stress differences of either STDs or of fluid foods (Rao, 1999). The first normal stress difference is known to be responsible for the climbing fluid film phenomenon often called the 'Weissenberg effect', and extrudate swell and normal force pump. The first normal stress coefficient $\Psi_1(\dot{\gamma})$ is an important material function which can be evaluated from the first normal stress difference, $\sigma_{11} - \sigma_{22}$:

$$\sigma_{11} - \sigma_{22} = \Psi_1(\dot{\gamma})(\dot{\gamma})^2 \tag{3}$$

Interrelationships between the parameters obtained using different rheological tests are useful to estimate one set from another. It was shown previously (Da Silva et al., 1997; Rao et al., 1997) that the Cox–Merz rule, which states that the apparent and complex viscosities are equal, is not followed by CWM STDs. However, Bird, Hassager, and Abdel-Khalik (1974) developed relationships for predicting $\Psi_1(\omega)$ from dynamic rheological data based on the co-rotational Goddard–Miller model. The relationships take the form (Bird, Armstrong, & Hassager, 1977, p. 343; Ferry, 1980, pp. 76–78):

$$\Psi_1(\omega) = \Psi_1^d + \Psi_1' \cos 2\omega t - \Psi_1'' \sin 2\omega t \tag{4}$$

$$\Psi_1^d = \eta''/\omega \tag{5}$$

$$\Psi_1'(\omega) = [-\eta''(\omega) + \eta''(2\omega)]/\omega \tag{6}$$

$$\Psi_1''(\omega) = [\eta'(\omega) - \eta'(2\omega)]/\omega \tag{7}$$

where $\Psi_1(\omega)$, rigorously defined as $[(\sigma_{11} - \sigma_{22})/(\dot{\gamma}_{21}^0)^2]$, is the sum of a constant term $\Psi_1^d$ and two oscillating terms, accounted by $\Psi_1'$ and $\Psi_1''$; $\dot{\gamma}_{21}^0$ is the strain rate amplitude. Eqs. (5)–(7) suggest that oscillatory shear stress data are related to oscillatory primary normal stress difference data (Ferry, 1980). The prediction of $\Psi_1(\omega)$ from dynamic rheological data is also useful because it may be possible to extend values of $\Psi_1$ to lower shear rates accessible only via oscillatory tests.

Our objectives were to investigate the effects of two heating temperatures: 85 and 110 °C, and sucrose concentrations ranging from 0 to 600 g kg$^{-1}$ on the granule size distribution, and the rheology: flow, dynamic and first normal stress difference of 50 g kg$^{-1}$ CWM STDs.

## 2. Materials

CWM (Purity W®, National Starch and Chemical Co., Bridgewater, NJ) STDs, 50 g kg$^{-1}$, which were heated in aqueous sucrose solutions with concentrations: 0, 100, 200, 300, 400, 500, and 600 g kg$^{-1}$ of STD were studied. The dispersions were heated either at 85 °C for 30 min in a Rotavapor (Büchi, Switzerland) flask rotating at 8 rpm or at 110 °C in sealed cans (size #300: 76 mm × 113 mm) for 10 min under intermittent agitation in a retort (Steritort®, FMC, Madera, CA). The STDs heated in the Rotavapor are denoted as RV and those heated in cans at 110 °C as CN.

### *2.1. Rotavapor samples*

To achieve instantaneously the desired temperature of a dispersion, the following energy balance equation (Okechukwu & Rao, 1995) was used:

$$m_C C_{pC}(T_C - T_o) + m_H C_{pH}(T_H - T_o) = (m_C + m_H) C_{pp}(T_p - T_o) \tag{8}$$

where $m_C$ is a small mass of starch, sucrose and water at a chosen temperature $T_C$ that is below-gelatinization onset, $T_o$ is the reference temperature, $C_{pC}$ is the heat capacity of the mass, $m_H$ is the mass of hot water at $T_H$, $C_{pH}$ is the heat capacity of hot water, $C_{pp}$ is the heat capacity of the paste, and $T_p$ is the equilibrium (desired pasting) temperature that is reached instantaneously. If the heat capacities $C_{pC}$, $C_{pH}$, and $C_{pp}$ are nearly equal and all the temperatures are in a

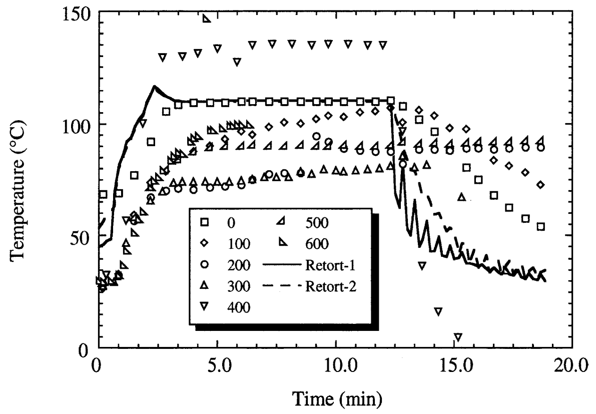


Fig. 1. Temperatures at the centers of canned starch dispersions during heating in an agitated retort (Steritort®). Also, Retort 1 and Retort 2 are temperatures at the retort rotating reel circumference and hub, respectively.

narrow range, a simpler equation results:

$$m_C T_C + m_H T_H = (m_C + m_H) T_p \tag{9}$$

By choosing $T_C$ and $T_H$ carefully, one can make the total mass ($m_C + m_H$) reach the desired final temperature $T_p$, and avoid any risk of gelatinization before the pasting time. An Excel spreadsheet was used to calculate the temperature $T_C$. Since $T_H$ could be as high as 98 °C, variation of density of water with temperature was taken into account.

The temperature of the sample in the Rotavapor was monitored with a type-T thermocouple located in the sample. Heating of the sample was followed by rapid cooling to 20 °C using tap water at about 11–15 °C. The total cooling time varied with the viscosity of the cooked dispersion, but for all of them the temperature fell below the gelatinization temperature in a couple of minutes.

### 2.2. Canned samples

Weighed amounts of starch and water were transferred to a 2000 ml Erlenmeyer flask and, under magnetic stirring, the required amount of sucrose was added slowly to the dispersion. The mixture of starch, sucrose, and water was allowed to hydrate overnight. No. 300 cans (76 mm × 113 mm, headspace 5 mm) were filled with the dispersions and sealed. The cans were subjected to intermittent axial rotation in a Steritort® that prevented settling of the starch granules. Needle-type thermocouples (Type CNS, Ecklund, Cape Coral, FL) were placed at the centers of the cans and the temperatures were recorded as a function of time during heating on a computer using a data logging system (CalSoft, TechniCal, New Orleans, LA). The canned STDs were heated for 10 min after the retort reached 110 °C, then cooled down to room temperature in the retort using water in mains.

## 3. Methods of characterization

### 3.1. Granule size distribution

The granule size distribution of the gelatinized starch dispersions was determined at room temperature of about 23 °C using a laser diffraction particle size analyzer (Coulter LS130, Coulter Corporation) as described earlier (Okechukwu & Rao, 1996).

### 3.2. Rheological behavior

Shear rate versus shear stress, normal force as a function of shear rate, and dynamic rheological data ($G'$, $G''$, tan $\delta$ as a function of frequency) were obtained on the STDs at 20 °C. A cone (either 6 or 4 cm dia, 2°)-plate geometry of an AR 1000 N rheometer (TA Instruments, New Castle, DE) was used to obtain the rheological data. Dynamic rheological data on each STD were obtained in the linear viscoelastic range, in the range of frequency from 0.01 to 10 Hz. The shear rate versus shear stress and normal force data were obtained immediately after the frequency sweep data. They were obtained first as the shear rates were increased continuously up to 300 $s^{-1}$ followed by shear rates in a decreasing mode from 300 to 0 $s^{-1}$in 6 min; the former are referred to as 'up' curves and the latter as 'down' curves. Using fresh samples, the series of frequency and shear rate sweeps were repeated at least three times to ensure that the data were reproducible. Data from a set representative of the replicates were analyzed and reported here. A few creep tests were carried out in order to determine the zero shear viscosity, but no such plateaus were found at shear rates as low as $10^{-4}$ $s^{-1}$.

Parameters of the HB model (Eq. (1)) and, when necessary, of the power law model (Eq. (2)) were determined using the non-linear regression software in KaleidaGraph (Synergy Software) on the shear rate–shear stress data.

## 4. Results and discussion

### 4.1. Temperature of STD in the Rotavapor and in the cans

The temperature versus time records of canned STDs indicated that the temperature at the center of each sample did not reach a steady state instantaneously due to thermal lag (Fig. 1). Eventhough the same retort temperature (110 °C) was used, the STDs with different sucrose concentrations attained a different steady state temperatures. The latter result is to be expected in view of the link between heat transfer and fluid flow that is affected by viscosity. Tattiyakul (2001) showed that heating rates in a 3.5% corn STD and a 5% CWM STD were different because of their different viscosities. Nevertheless, the STDs were subjected to temperatures

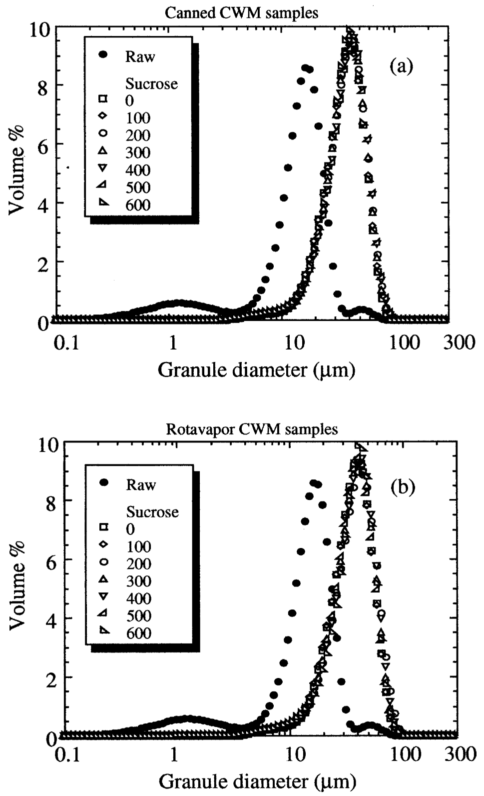


Fig. 2. (a) and (b) Granule size distribution of 50 g kg$^{-1}$ starch dispersions with 0–600 g kg$^{-1}$ sucrose heated at 85 °C in Rotavapor® and in cans at a retort temperature 110 °C.

higher than 85 °C. In addition, the thermocouple in the 400 g kg$^{-1}$ STD malfunctioned and registered temperatures much above that of the retort, and that in the 600 g kg$^{-1}$ STD failed after about 10 min.

In contrast, all the STDs heated in the Rotavapor were brought to 85 °C rapidly as described earlier and held at 85 °C for 30 min. The variation in the temperature of each sample was ± 1 °C during heating in the Rotavapor

Table 1
Average diameter and standard deviation of cross-linked waxy maize starch dispersions heated in sucrose solutions

| Sucrose (g kg$^{-1}$) | Rotavapor (85 °C) | | Canned (110 °C) | |
|---|---|---|---|---|
| | Diameter (μm) | Standard deviation (μm) | Diameter (μm) | Standard deviation (μm) |
| Raw[a] | 15.82 | 0.22 | 15.82 | 0.22 |
| 0 | 38.48 | 0.49 | 39.26 | 0.34 |
| 100 | 38.32 | 0.41 | 39.46 | 0.33 |
| 200 | 41.24 | 1.66 | 40.61 | 0.28 |
| 300 | 40.09 | 0.32 | 40.93 | 0.32 |
| 400 | 40.51 | 0.52 | 41.95 | 0.21 |
| 500 | 39.39 | 0.14 | 41.74 | 0.11 |
| 600 | 39.79 | 0.25 | 39.26 | 0.05 |

[a] Raw samples are dispersions hydrated in distilled water.

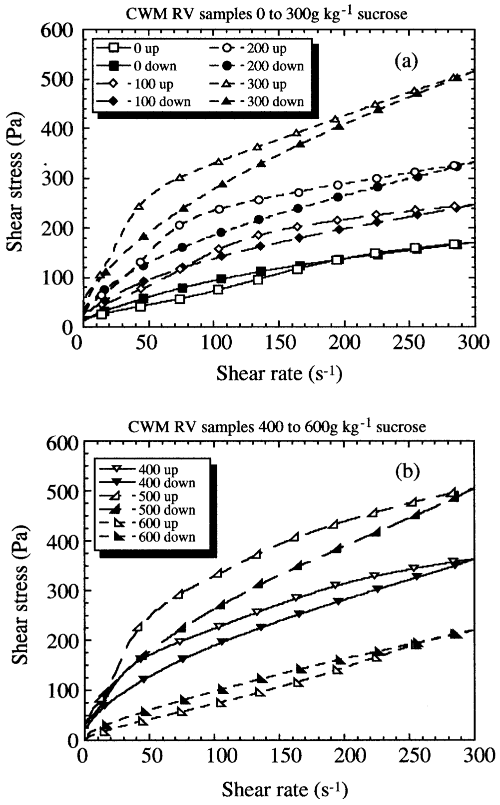


Fig. 3. (a) and (b) Flow behavior of 50 g kg$^{-1}$ starch dispersions with 0–300 g kg$^{-1}$ and 400–600 g kg$^{-1}$ sucrose heated at 85 °C in Rotavapor®.

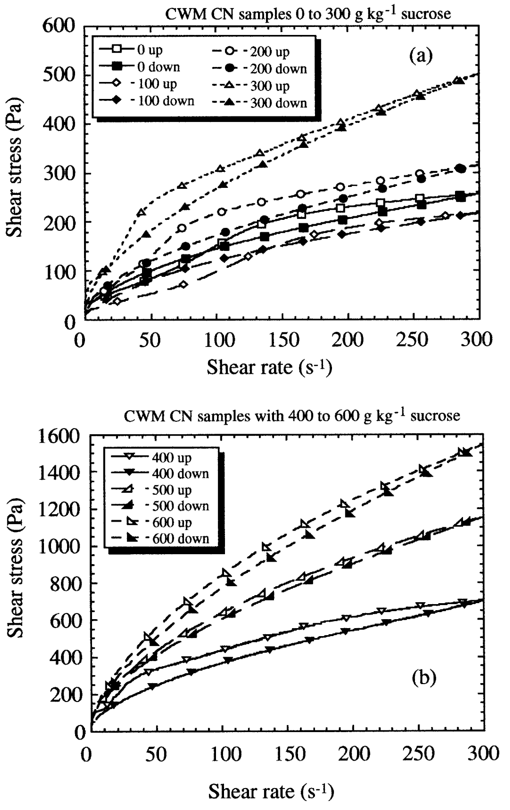


Fig. 4. (a) and (b) Flow behavior of 50 g kg$^{-1}$ starch dispersions with 0–300 g kg$^{-1}$ and 400–600 g kg$^{-1}$ sucrose heated in cans at a retort temperature 110 °C.

Table 2
Herschel–Bulkley model parameters of cross-linked waxy maize starch dispersions heated in sucrose solutions: $\sigma_0$ is yield stress, $K$ is consistency coefficient, and $n$ is the flow behavior index

| Sucrose (g kg$^{-1}$ | Rotavapor (85 °C) | | | Canned (110 °C) | | |
|---|---|---|---|---|---|---|
| | $\sigma_0$ (Pa) | $K$ (Pa s$^n$) | $n$ (–) | $\sigma_0$ (Pa) | $K$ (Pa s$^n$) | $n$ (–) |
| 0 | 0 | 7.1 | 0.76 | 19.9 | 7.6 | 0.61 |
| 100 | 2.8 | 12.0 | 0.54 | 8.3 | 7.2 | 0.59 |
| 200 | 14.5 | 12.6 | 0.57 | 14.2 | 11.1 | 0.58 |
| 300 | 20.2 | 16.3 | 0.60 | 61.7 | 9.3 | 0.70 |
| 400 | 34.7 | 18.0 | 0.64 | 73.3 | 8.7 | 0.74 |
| 500 | 42.5 | 16.2 | 0.65 | 145.7 | 11.4 | 0.78 |
| 600 | 7.2 | 6.4 | 0.74 | 196.5 | 13.8 | 0.83 |

### 4.2. Granule size distribution

The granule size distributions of the CN and RV STDs did not change much due to changes in sucrose concentration (Fig. 2a and b). Eventhough the heated granules enlarged in comparison to the unheated granules, the presence of sucrose did not result in a large change in diameters of the starch granules. The mean granule diameter and the standard deviation of both the CN and RV STDs are summarized in Table 1.

### 4.3. Shear rate versus shear stress behavior

At sucrose concentrations 0–300 g kg$^{-1}$ both the RV and the CN dispersions exhibited antithixotropic behavior in that segments of the down curves reached values of shear stress higher than those of the up curves (Figs. 3a and 4a) at the same values of shear rate. The higher stress values translate in to higher apparent viscosities. The antithixotropic behavior occurred over a narrow range of shear stress values: 100–140 Pa in agreement with the observation of Tattiyakul and Rao (2000) that such behavior occurred when the shear stress values are <150–200 Pa.

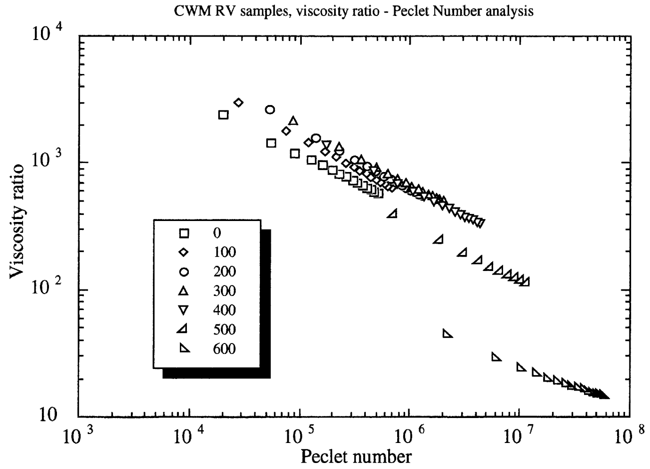


Fig. 5. Viscosity ratio/apparent viscosity of starch dispersion heated at 85 °C in Rotavapor®/viscosity of the sucrose solution as a function of Peclet number.

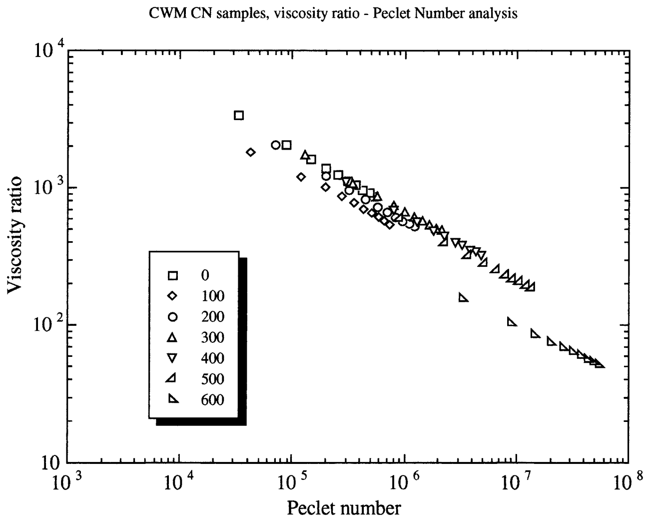


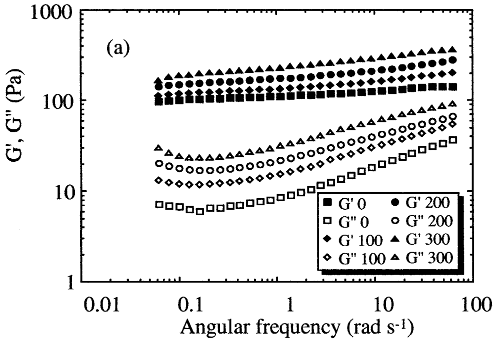
Fig. 6. Viscosity ratio/apparent viscosity of starch dispersion heated in cans at a retort temperature 110 °C/viscosity of the sucrose solution as a function of Peclet number.

In contrast, with the exception of the RV STD with 600 g kg$^{-1}$, the STDs with higher sucrose concentrations exhibited thixotropic behavior (Figs. 3b and 4b). Previous work attributed antithixotropic behavior in retorted CWM starch dispersion without sucrose to shear-induced clusters (Chamberlain et al., 1999). Therefore, it seems that in CN dispersions with sucrose concentrations 400, 500, and 600 g kg$^{-1}$ and RV dispersions with 400 and 500 g kg$^{-1}$, shear-induced clusters were not formed. Further, as previously reported by Tattiyakul and Rao (2000), the antithixotropic behavior was observed when values of the shear stress were less than 120–150 Pa.

From the values of the HB and power law parameters of the down curves, shown in Table 2, it can be seen that the consistency coefficient and the yield stress of CN samples increased with increase in sucrose concentration. But, those of the RV samples increased up to sucrose 400 g kg$^{-1}$, but decreased with further increase in sucrose to 500 and 600 g kg$^{-1}$. The apparent viscosities at 10 s$^{-1}$ of the CN and RV samples were: 5.1 and 4.1, 3.6 and 4.4, 5.6 and 6.1, 10.8 and 8.5, 12.1 and 11.3, 21.4 and 11.5, and 29.0 and 4.2 Pa s, at sucrose content 0, 100, 200, 300, 400, 500 and 600 g kg$^{-1}$, respectively. Since the magnitudes of the flow behavior index were less than 1.0, all the STDs were shear-thinning fluids.

The higher yield stresses of the CN samples, especially those with sucrose 300–600 g kg$^{-1}$, may be attributed to the higher heating temperature, about 110 °C compared to 85 °C. Since the higher heating temperature did not result in increase in the mean granule diameter of the STDs and, hence, volume fraction of the granules, it is likely that it contributed to changes in granule morphology that increased the binding energy of the network of the water–sucrose–starch granule microstructures (Hiemenz & Rajagopalan, 1997).

Mechanical spectra of CWM RV samples 0 to 300 g kg-1 sucrose



Mechanical spectra of CWM RV samples, 400 to 600 g kg-1 sucrose

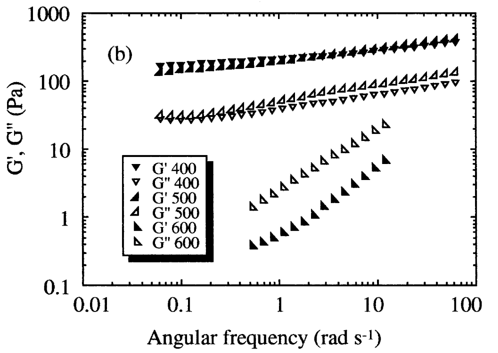


Fig. 7. (a) and (b) Mechanical spectra of starch dispersions with 0–300 and 400–600 g kg$^{-1}$ sucrose heated at 85 °C in Rotavapor®. Note the gel-like behavior of dispersions except for STD with 600 g kg$^{-1}$ sucrose.

The apparent viscosity of a STD depends on the volume fraction of the starch granules and the viscosity of the sucrose solution in which the starch granules were heated. The influence of viscosity of the sucrose solutions, starch granule radius, and shear rate, can be examined in terms of the viscosity ratio, defined as the apparent viscosity of the STD divided by the viscosity of the sugar solution, and the Peclet number:

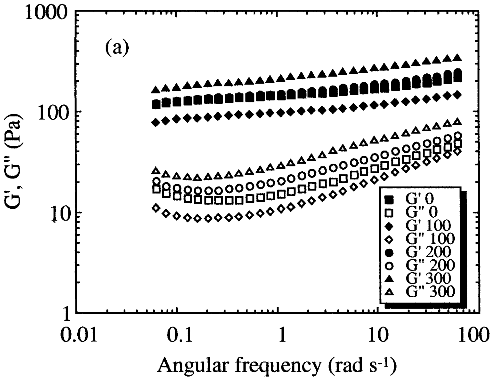
$$Pe = \frac{\eta r^3 \dot{\gamma}}{kT} \qquad (10)$$

Table 3
Power-law parameters of complex viscosity ($\eta^*$) versus frequency ($\omega$) data: $K^*$ is consistency coefficient, $n^*$ is flow behavior index

| Sucrose (g kg$^{-1}$) | Rotavapor (85 °C) | | | Canned (110 °C) | | |
|---|---|---|---|---|---|---|
| | $K^*$ (Pa s$^{n^*}$) | $n^*$ | $R^2$ | $K^*$ (Pa s$^{n^*}$) | $n^*$ | $R^2$ |
| 0 | 122.3 | 0.06 | 1.00 | 146.6 | 0.08 | 1.00 |
| 100 | 137.8 | 0.08 | 1.00 | 98.8 | 0.09 | 1.00 |
| 200 | 178.6 | 0.10 | 1.00 | 154.4 | 0.10 | 1.00 |
| 300 | 207.5 | 0.10 | 1.00 | 219.5 | 0.11 | 1.00 |
| 400 | 218.5 | 0.12 | 1.00 | 235.0 | 0.12 | 1.00 |
| 500 | 168.6 | 0.17 | 1.00 | 276.3 | 0.14 | 1.00 |
| 600[a] | 2.0 | 0.95 | 0.98 | 258.7 | 0.17 | 1.00 |
| 600[b] | 2.9 | 0.80 | 0.99 | NA[c] | | |

[a] RV samples in the range of 1–10 rad s$^{-1}$.
[b] RV samples in the range of 10–100 rad s$^{-1}$.
[c] NA stands for not applicable.

Mechanical spectra of CN samples 0 to 300 g kg-1 sucrose



Mechanical spectra of CN samples with 400 to 600 g kg-1 sucrose

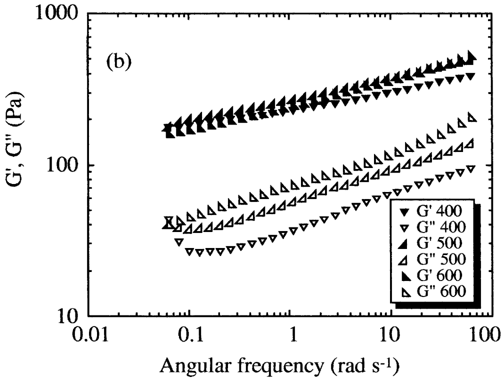


Fig. 8. (a) and (b) Mechanical spectra of starch dispersions with 0–300 and 400–600 g kg$^{-1}$ sucrose heated in cans at a retort temperature 110 °C. All the dispersions exhibited gel-like behavior.

where $\eta$ is the viscosity of the sucrose solution, $r$ is the mean radius of the starch granule, $k$ is the Boltzmann constant, and $T$ is the absolute temperature. The viscosities of the sucrose solutions were calculated by interpolation of data in the NBS Circular C440 (Bates, 1942). The Peclet number is the ratio of shear-induced convective (hydrodynamic) effect to diffusion of particles by Brownian diffusion (Hiemenz & Rajagopalan, 1997). Such plots for the RV and CN samples are shown in Figs. 5 and 6, respectively. When $Pe \gg 1$, the hydrodynamic effects are dominant and often the flow behavior of a dispersion is shear-thinning, a result confirmed here (Table 2). Whereas the viscosity ratio versus Pe plots for the CN samples are relatively close to each other, those of the RV STDs, especially those with sucrose concentrations 500 and 600 g kg$^{-1}$, follow separate curves.

### 4.4. Dynamic rheological behavior

Magnitudes of $G'$ and $G''$ as a function of $\omega$ of the RV and CN STDs are shown on double logarithmic coordinates in Figs. 7(a,b) and 8(a,b), respectively. While the log $G'$ versus log $\omega$ values can be described by straight lines, the profiles of log $G''$ versus log $\omega$ are concave down; the $G'$ and $G''$ profiles are indicators of typical gel-like behavior. In general, at a fixed frequency, $G'$ and $G''$ of CN STDs were

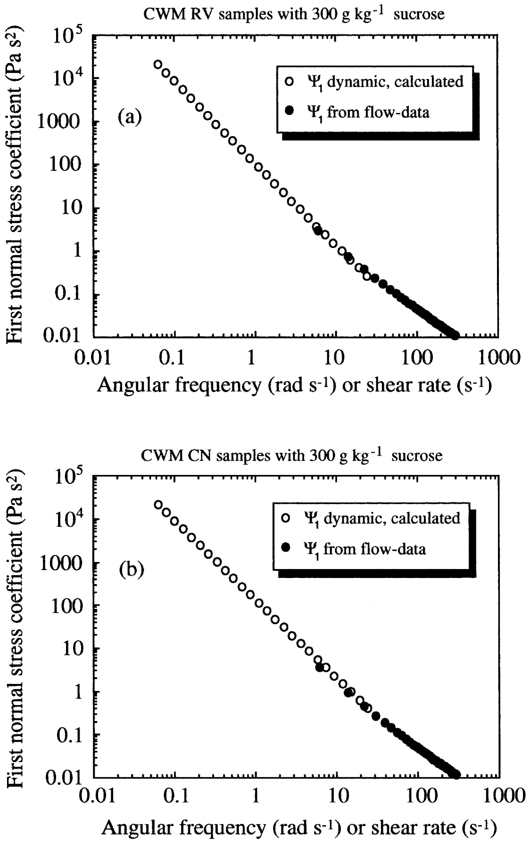


Fig. 9. (a) and (b) Experimental, $\Psi_1(\dot{\gamma})$, and calculated, $\Psi_1(\omega)$, first normal stress coefficients for starch dispersions with 300 g $kg^{-1}$ sucrose heated at 85 °C in Rotavapor® and in cans at a retort temperature 110 °C.

higher than those of the RV STDs. In addition, as with shear rate versus shear stress data, where as $G'$ and $G''$ of the CN samples increased with sugar concentration, those of the RV samples increased up to sucrose 500 g $kg^{-1}$ and decreased with further increase in sucrose to 600 g $kg^{-1}$. In addition, a power law type equation was fit to the values of log $\eta^*$ versus log $\omega$ data:

$$\eta^* = K^* \omega^{n^*-1} \tag{11}$$

From the values of $K^*$ and $n^*$ in Table 3, it can be seen that the flow behavior index increased at high sugar concentrations, indicating that the dispersions were becoming less shear-thinning. In particular, the RV samples with 600 g $kg^{-1}$ exhibited nearly Newtonian behavior.

### *4.5. First normal stress differences*

From the experimental first normal stress difference data, values of the first normal stress coefficient $\Psi_1(\dot{\gamma})$ were calculated using Eq. (3). Plots of $\Psi_1(\dot{\gamma})$ as a function of shear rate of RV and CN STDs with 300 g $kg^{-1}$ sucrose are shown in Fig. 9a and b, respectively. In addition, values of $\Psi_1(\omega)$ that were calculated from the dynamic rheological data using Eq. (4) are shown in the figures and they are in good agreement with experimental values.

For all the CN dispersions, values of $\Psi_1(\omega)$ predicted by Eq. (4) were close to the experimental values of $\Psi_1(\dot{\gamma})$ obtained under steady flow. For the RV samples, experimental values of $\Psi_1(\dot{\gamma})$ of dispersions with 300 and 600 g $kg^{-1}$ sucrose obtained under steady flow were close to those predicted using Eq. (4). At other sucrose concentrations, the predicted and experimental values were parallel to each other.

## 5. Conclusions

Sucrose content (100–600 g $kg^{-1}$) did not affect significantly the granule size distribution of CWM starch dispersions (STDs). The heated STDs without sucrose and those with sucrose 100–300 g $kg^{-1}$ exhibited antithixotropic behavior. One consequence of this characteristic is that apparent viscosities of the STDs increase with time due to shearing. The temperature at which the STDs were heated played an important role. The canned STDs heated in a retort at 110 °C exhibited higher magnitudes of flow and dynamic rheological properties than those heated at 85 °C in a Rotavapor; in particular, yield stresses of canned STDs with sucrose 300–600 g $kg^{-1}$ were much higher. It is likely that the higher heating temperature contributed to changes in granule morphology that increased the binding energy of the network of the water–sucrose–starch granule microstructures. The apparent viscosities, and $G'$ and $G''$ of the canned samples increased with sugar concentration, while those of the Rotavapor samples increased up to sucrose 400 g $kg^{-1}$ and decreased with further increase in sucrose to 500 and 600 g $kg^{-1}$. The STDs exhibited normal stresses as a function of shear rate and values of the first normal stress coefficient $\Psi_1(\dot{\gamma})$ of some of the STDs were in good agreement with those calculated from dynamic rheological data. This observation is important because, when it is not possible to obtain first normal stress data, it may be possible to estimate them from dynamic rheological data; in turn such data are useful to understand die swell and rod climbing effect.

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