

Research Article

Shuxin Huang*

Viscoelastic characterization of corn starch paste: (II) The first normal stress difference of a cross-linked waxy corn starch paste

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Abstract: The power-law strain model used to characterize the first normal stress difference (N_1) was checked here second based on the experimental linear viscoelastic properties, steady shear viscosities, and N_1 s of two cross-linked waxy corn starch pastes reported in 2003. Parameter f in a structured integral viscoelastic constitutive equation was obtained using the viscosities in the dynamic and steady shear experiments, and both the original and power-law strain models were employed to describe the normal component in the strain matrix of the equation and the N_1 s of the two pastes. The results showed that the three types of viscoelastic properties of the paste can be described well by the structured equation including the power-law strain model. In contrast, the descriptions of N_1 s based on the original strain model have huge deviation from the experiments for both pastes, which are similar to the first characterization of N_1 of corn starch paste with sucrose in 2022.

keywords: cross-linked waxy corn starch, paste, viscoelastic property, structuralized integral constitutive equation, power-law strain model

1 Introduction

Corn starch or maize starch paste is widely used in the food, pharmaceutical, textile, and alcohol industries [1–3], and its viscoelastic properties influence the processing and quality of its products. As a result, research on the viscoelastic properties of corn starch pastes is usually related to

the technology of starch processing [1,4–7]. Such studies are also related to the theoretical development of the viscoelastic analysis of starch-based fluids [8,9].

Huang [9] reported a theoretical characterization of both the steady shear viscosity η and first normal stress difference N_1 for a cross-linked waxy corn starch paste mixed with sucrose using a structural viscoelastic model in 2022. This work was based on a literature search on the viscoelastic properties of corn starch pastes over the past 30 years. Table 1 lists two supplemental references found recently on the viscoelasticities of corn starch pastes, together with the original nine pieces of references in the study by Huang [9]. The two criteria for selecting the works in Table 1 were that: (1) Each study must contain viscoelastic data of pure corn starch paste, and (2) includes at least a frequency sweep, steady shear viscosity, and another viscoelastic property (e.g., the steady N_1 or transient viscoelastic property), under specific temperature and concentration conditions. The purpose of including the three types of viscoelastic data is to predict the third set of experimental data based on the theoretical characterization using the first two. The 11 references in Table 1 indicate the insufficient status of viscoelastic studies on corn starch pastes.

The original purpose of the study by Huang [9] was to describe the steady shear viscoelastic experiment of a corn starch paste in the study by Acquarone and Rao [12] using an integral viscoelastic model. The experiment by Acquarone and Rao [12] is the simplest, which does not include transient experiment; and the integral viscoelastic model has strong capability to describe the viscoelastic property of complex fluids in the experiences of the author of previous studies [21–27]. However, the initial trial of the N_1 characterization failed [9], and then, a power-law strain model was proposed to describe the normal component in the strain matrix and the calculation of N_1 was acceptable. The trial [9] indicated that the traditional Cauchy strain tensor and Finger strain tensor could be invalid for the theoretical characterization of N_1 of corn starch paste.

* **Corresponding author: Shuxin Huang**, Department of Engineering Mechanics, Shanghai Jiao Tong University, Shanghai, 200240, China; Key Laboratory of Hydrodynamics of the Ministry of Education, Shanghai Jiao Tong University, Shanghai, 200240, China; State Key Laboratory of Ocean Engineering, Shanghai Jiao Tong University, Shanghai, China, e-mail: huangshuxin@sjtu.edu.cn

Table 1: Viscoelastic properties of corn starch pastes in selected literature

No.	Material ¹	Experiment ²	Theory ³	Ref. ⁴
1	CWCS	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \eta-t$	Power law, HB	[10], 1997
2	CWCS	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \eta-t$	Power law	[11], 1997
3	CWCS, sucrose	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, N_1-\dot{\gamma}$	Power law	[12], 2003
4	WCS, guar, xanthan	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \text{multi } \eta-t$	Power law	[13], 2008
5	WCS, xanthan, sucrose	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \eta-t, \text{multi } \eta-t$	Power law	[14], 2009
6	NCS, WCS	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \eta-t, \text{multi } \eta-t$	Power law	[15], 2010
7	NCS, oil, water	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, J-t$	HB, Burgers	[16], 2018
8	NCS, HACs	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \gamma-t$	Power law	[17], 2018
9	NCS, cellulose, polydextrose	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, \text{multi } \eta-t$	HB	[18], 2018
10	CWCS, sucrose	$G'-\omega, G''-\omega, \eta-\dot{\gamma}, N_1-\dot{\gamma}$	Power law	[19], 2003
11	CWCS	$\eta^*-\omega, \eta-\dot{\gamma}, N_1-\dot{\gamma}$	Power law	[20], 2003

Note ¹The first C in CWCS is “crosslinked,” W is “waxy,” CS is “corn starch” or “maize starch,” N is “normal,” and HA is “high amylose.”

² G' , storage modulus; G'' , loss modulus; ω , angular frequency; η , shear viscosity; $\dot{\gamma}$, shear rate; t , time; N_1 , first normal stress difference; J , creep compliance; γ , shear strain; multi $\eta-t$ means the viscosity-time curve in multi-step rate experiment; η^* , the magnitude of complex viscosity, called simply complex viscosity here.

³HB is the Herschel–Bulkley model.

⁴[19,20] were added in the present work, and the others can be found in [9].

Column 4 in Table 1 indicates that no theoretical work has attempted to describe the N_1 of corn starch paste, although the literature in Table 1 covering the past 30 years contains three limitations. After almost 25 years of experience of using integral- and differential-type constitutive equations [21–27], it is believed that the N_1 characterization of the paste could be a problem. The study by Huang [9] is the only one that characterizes the N_1 of corn starch paste based on the typical viscoelastic theory, and therefore, it is proposed that such a theoretical characterization should be conducted.

Sucrose is present in corn starch paste [9,12]. Sugar can affect the pasting and retrogradation of starch pastes [4,14,28], and further viscoelastic experiments [4,12,14] have shown that sucrose can increase the viscosity of corn starch pastes. But this is not always true, and Hirashima et al. [4] and Acquarone and Rao [12] reported that sucrose can decrease the viscosity of pastes. Therefore, sucrose influences the viscoelastic properties of corn starch paste, and the power-law strain model in the study by Huang [9] still needs to be checked for pure corn starch paste. Viscoelastic characterization of pure corn starch paste is a basic requirement for both processing applications and the development of viscoelastic models of the paste.

The aim of the present work was to check whether the power law strain model [9] is suitable for the calculation of the steady N_1 of pure corn starch paste. Experimental data are from previous studies [19,20], which were found in the recent search of literature on the viscoelastic properties of corn starch pastes. Both groups of experimental data [19,20] also obey the power-law strain model but cannot be described by the linear strain model [9].

2 Materials and methods

2.1 Corn starch paste

Research works [19,20] are from the research group of Professor Rao, and in fact, there are five studies [10–12,19,20] that come from the group in Table 1. Almost half of the studies in Table 1 are related to the works of Professor Rao, which shows that Professor Rao once dedicated much time to extensively study the viscoelastic properties of corn starch paste. The corn starch used in previous studies [19,20] was a crosslinked waxy corn starch (CWCS) with a brand of Purity W[®] from the National Starch and Chemical Co. in Bridgewater, N.J., USA, which is the same as that employed by Acquarone and Rao [12]. Another CWCS with no. lot TBK 3886 also from the National Starch and Chemical Co. in Bridgewater, N.J., USA, was adopted in two earlier studies [10,11]. Therefore, this research group is familiar with the viscoelastic properties of CWCS paste.

The viscoelastic properties of pure corn starch paste including the steady N_1 were only reported in the studies [19,20], and therefore, these two groups of experimental data can be used to check the theoretical model in the study by Huang [9] for pure corn starch paste. Acquarone and Rao [12] did not report the N_1 for pure CWCS paste, and only introduced it for a 5 wt% CWCS paste with 30 wt% sucrose, which was theoretically analyzed by Huang [9].

The concentration of the pure CWCS paste in the study by Youn and Rao [19] was 5 wt%, which was heated in cans (Steritort[®], FMC, Madera, USA) at a retort temperature of

110°C for 10 min. A frequency sweep experiment was conducted in the linear viscoelastic region. Steady shear experimental data were obtained through a triangular shear loop experiment, half of the time of the loop was 3 min, and the maximum shear rate of the loop was 300 s^{-1} . The viscosity and N_1 in the down region of the shear rate were used as steady shear experimental data. More details of the experiment can be found in the study by Youn and Rao [19].

The concentration of the pure CWCS paste in Genovese and Rao [20] was also 5 wt%, which was heated at 80°C in a Rotavapor (Büchi, Switzerland) for 10 min. A frequency sweep was also conducted in the linear viscoelastic region. A steady shear experiment was also obtained through a triangular shear loop experiment, but half of the loop time was 10 min, and the maximum shear rate of the loop was 500 s^{-1} . The viscosity and N_1 in the down region of the shear rate were used as steady shear data. More details of the experiment can be found in the study by Genovese and Rao [20].

The viscoelastic properties of the two pure CWCS pastes [19,20] have some differences due to the change in the experimental conditions, although the same CWCS and concentration were employed. Figure 1(a) shows the difference in viscosities and N_1 s between the two experiments, and in Figure 1(b), the deviation between viscosities is in the range of approximately 10–30%, and the N_1 deviation is approximately 25–40%.

The definition of deviation is,

$$\text{deviation} = \frac{|x - x^*|}{x} \times 100\%, \quad (1)$$

where x is a group of data, and x^* is the other used for comparison. x and x^* used in the comparisons in Figure 1(b) are Genovese and Rao's data [20] and Youn and Rao's data [19], respectively.

2.2 Viscoelastic model

A structured integral viscoelastic constitutive equation is given in the study by Huang [9], which is written as

$$\tau = \int_{-\infty}^t m(t - t', f) \cdot \phi_1(I_1, I_2) \cdot [\delta - S_t(t')] \cdot dt', \quad (2)$$

where τ is the stress tensor, $m(t - t', f)$ is the time- and shear-rate-dependent memory function, t and t' are the present and past times, respectively, with unit of s, f reflects the effect of shear rate on the change in structure or relaxation spectrum, $\phi_1(I_1, I_2)$ is a strain-dependent function, I_1 and I_2 are the first invariants of C_t^{-1} and C_b , respectively, C_t^{-1} is the Finger strain tensor, C_t is the Cauchy strain tensor, δ is the unit tensor, and $S_t(t')$ is a changeable strain tensor. In simple shear flow [29,30], $S_t(t')$ is expressed as

$$S_t(t') = \begin{pmatrix} 1 + S_{11}(\gamma) & \gamma & 0 \\ \gamma & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix}, \quad (3)$$

where $S_{11}(\gamma)$ can be written as equation (4) or (5),

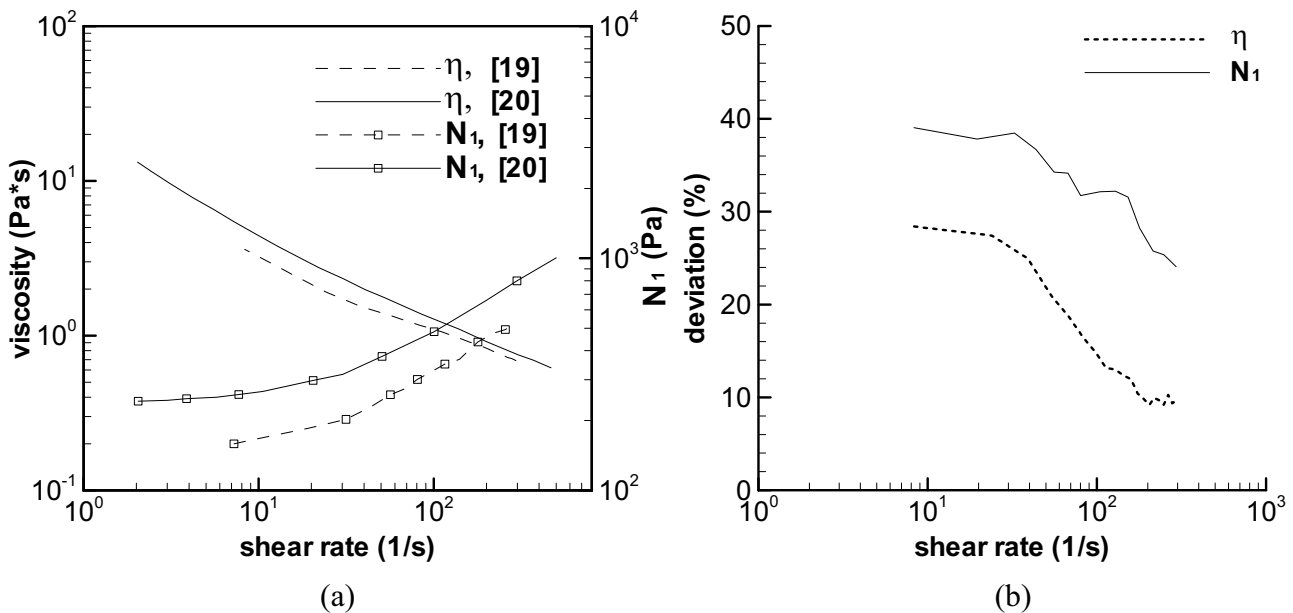


Figure 1: Deviations between the two experiments of Youn and Rao [19] and Genovese and Rao [20]. (a) Viscosities and N_1 s of the CWCS paste in the two experiments. (b) Deviations between the experimental data.

$$S_{11}(\gamma) = \gamma^2, \quad (4)$$

$$S_{11}(\gamma) = k\gamma^n, \quad (5)$$

where k and n are the parameters. If equation (4) is adopted, the original strain component without including one on the diagonal in the Finger strain tensor is recovered. Equation (5) is the power-law strain model, which can be used to describe the special strain component experienced in deformation.

The memory function m is given as

$$m = \sum_i \frac{g_i \cdot f(\dot{\gamma})}{\lambda_i} \cdot e^{\left(-\frac{t-t'}{\lambda_i}\right)}, \quad (6)$$

where λ_i and g_i are the relaxation times (s) and the relaxation modulus coefficients (Pa), respectively, at a low shear rate or at rest. The ϕ_1 -function [25] adopted is given as

$$\phi_1(\gamma) = \frac{1}{\sqrt{1 + \gamma^2}}. \quad (7)$$

Equation (2) without parameter f , incorporating equation (4), can be found in the work by Meister [29]. However, Meister [29] did not use equation (7). Equation (5) can be employed to examine whether the calculation of N_1 for the fluid obeys the traditional Cauchy and Finger strain tensors.

Equation (2) was solved numerically by the Laguerre–Gauss integration method, which was fulfilled via a Fortran code [31].

2.3 Characterization of viscoelastic property

The characterization procedure for the steady shear viscoelastic properties of the corn starch paste based on equation (2) has been given in the study [9], which is summarized here.

- (1) The relaxation spectra (λ_i , g_i), structural parameter f , and parameters (k , n) are used in equation (2).
- (2) Linear viscoelastic experimental data should be employed to obtain spectra (λ_i , g_i).
- (3) $f = f_1 \times f_2$, where f_1 equates to the complex viscosity divided by the viscosity calculated using equation (2) without the f -parameter, and f_2 equates to the experimental steady shear viscosity divided by the viscosity calculated using equation (2) with the f_1 -parameter.
- (4) A simplex method [31,32] is adopted to fit (k , n) based on the experimental steady shear N_1 .

3 Results and discussion

3.1 Linear viscoelastic property

$G'(\omega)$ and $G''(\omega)$ were given experimentally by Youn and Rao [19], and therefore, the following equations can be employed to obtain (λ_i , g_i),

$$G'(\omega) = \sum_i \frac{g_i \lambda_i^2 \omega^2}{1 + \lambda_i^2 \omega^2}, \quad (8)$$

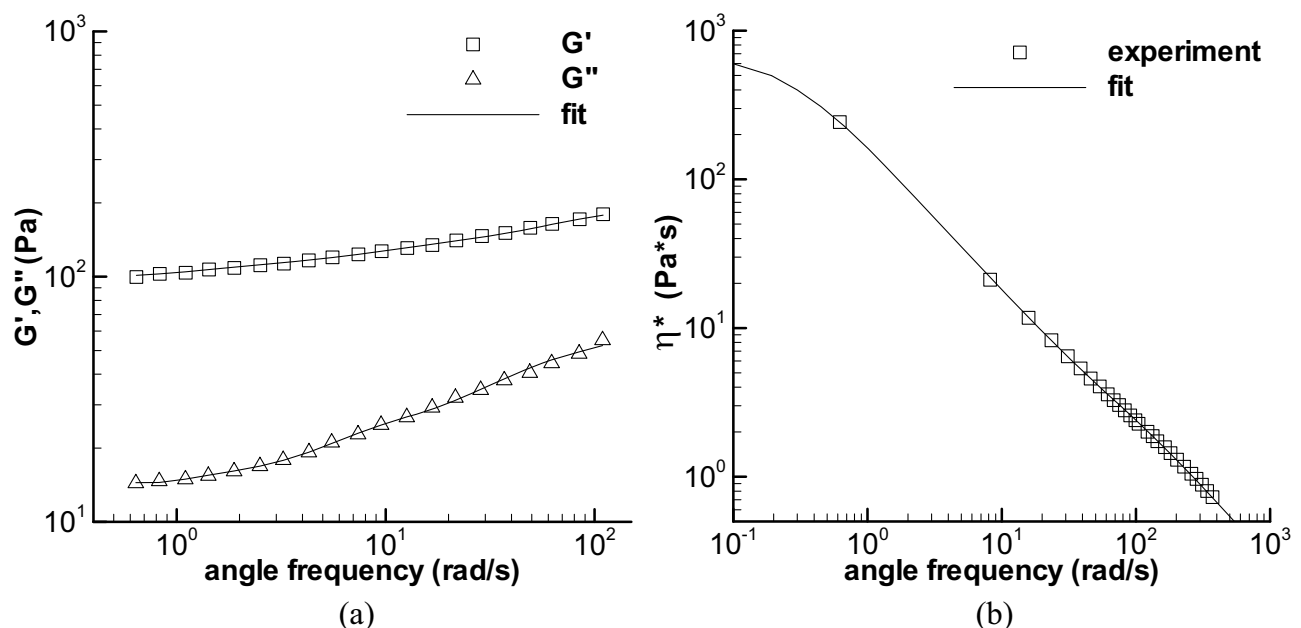


Figure 2: (a) Linear viscoelastic experiment of Youn and Rao [19] (symbol) and fit here (line) for the pure CWCS paste. (b) Linear viscoelastic experiment of Genovese and Rao [20] (symbol) and fit here (line).

Table 2: Relaxation spectra of the CWCS paste experimentally given by Youn and Rao [19]

i	λ_i (s)	g_i (Pa)
1	1.931×10^{-4}	1.257×10^3
2	1.511×10^{-2}	5.302×10^1
3	1.031×10^{-1}	2.460×10^1
4	6.600×10^{-1}	1.482×10^1
5	1.738×10^1	3.767×10^1
6	2.752×10^1	6.168×10^1

$$G''(\omega) = \sum_i \frac{g_i \lambda_i \omega}{1 + \lambda_i^2 \omega^2}. \quad (9)$$

The fitting procedures for equations (8) and (9) can be found in the study by Bird et al. [33], which was fulfilled with the solver function in Excel 2003 of Office software of Microsoft Co. (Redmond, Washington State, USA). The fitting results for the paste are shown in Figure 2(a), and the relaxation spectra (λ_i , g_i) are listed in Table 2.

Genovese and Rao [20] only gave the complex viscosity η^* of the CWCS paste, and therefore, the fitting equation [33] is

$$|\eta^*| = \sqrt{\eta'^2 + \eta''^2}, \quad (10.1)$$

$$\eta'(\omega) = \sum_i \frac{g_i \lambda_i}{1 + \lambda_i^2 \omega^2}, \quad (10.2)$$

$$\eta''(\omega) = \sum_i \frac{g_i \lambda_i^2 \omega}{1 + \lambda_i^2 \omega^2}. \quad (10.3)$$

The fitting results for the paste are shown in Figure 2(b), and the spectra (λ_i , g_i) are listed in Table 3.

3.2 Viscosity

Figure 3(a) and (b) shows the theoretical characterizations of the steady shear viscosities for the two CWCS pastes given by Youn and Rao [19] and Genovese and Rao [20], respectively, using equation (2). Both characterization procedures are identical, and the calculation of the viscosity of

Youn and Rao [19] is used as an example here. The coordinate of the complex viscosity is the angle frequency, which is substituted using the shear rate in Figure 3(a), and therefore, the complex viscosity in Figure 3(a) is a group of assumed steady shear viscosities. We can see that the calculated viscosity using equation (2) without the f -parameter, which is denoted by “no f ” in Figure 3(a), agrees well with the complex viscosity, which is the function of equation (7). This phenomenon can also be found in the studies by Huang [9,25].

There are some minor deviations between the “no f ” and complex viscosity in Figure 3(a) in the shear rate range of lower than about 0.4 s^{-1} , which can also be found in the studies by Huang [9,25]. There is no reasonable explanation for this phenomenon, and an assumption of the shear rate effect [9,25] is employed to eliminate the deviation using parameter f_1 . The corresponding calculation with f_1 is also shown in Figure 3(a), and the f_1 -parameter for the CWCS paste of Youn and Rao [19] is shown in Figure 3(c).

Figure 3(a) shows that the experimental steady shear viscosity has a large deviation from the calculated viscosity with the f_1 -parameter, which was eliminated artificially here using an assumed shear rate effect, namely, parameter f_2 . Then, the corresponding calculation with $f = f_1 \times f_2$ agrees with the experiment, which is denoted by “ f ” in Figure 3(a). Parameters f_2 and f for the paste of Youn and Rao [19] are also shown in Figure 3(c). Parameters f_1 and f were used through a linear interpolation method [9,25–27].

3.3 First normal stress difference

The calculations on N_1 using equations (4) and (5), incorporating (λ_i , g_i) and f , for the CWCS paste of Youn and Rao [19] are shown in Figure 4(a), and those for the paste of Genovese and Rao [20] are given in Figure 4(b). The “original” in Figure 4 means calculations using equation (4) and “power law” refers to those using equation (5). The “power law” calculations approach the N_1 experiments [19,20]. The explanation for this phenomenon [9] is that the elasticity of corn starch paste is weak, and the deformation of the paste in steady shear flow could not obey the traditional Cauchy and Finger strains. Thus, the calculations of equation (4) have large deviations from the experiments. Traditional strains are often suitable for characterizing N_1 s of highly elastic polymer solutions and melts [29,30]. The success of equation (5) for the N_1 calculations of corn starch pastes here and in the study by Huang [9] indicates the failure of the traditional strain in the N_1 characterization of certain viscoelastic fluids.

Table 3: Relaxation spectra of the CWCS paste experimentally given by Genovese and Rao [20]

i	λ_i (s)	g_i (Pa)
1	9.191×10^{-3}	1.009×10^2
2	2.076×10^0	8.759×10^1
3	5.464×10^0	7.056×10^1
4	1.121×10^1	1.352×10^1

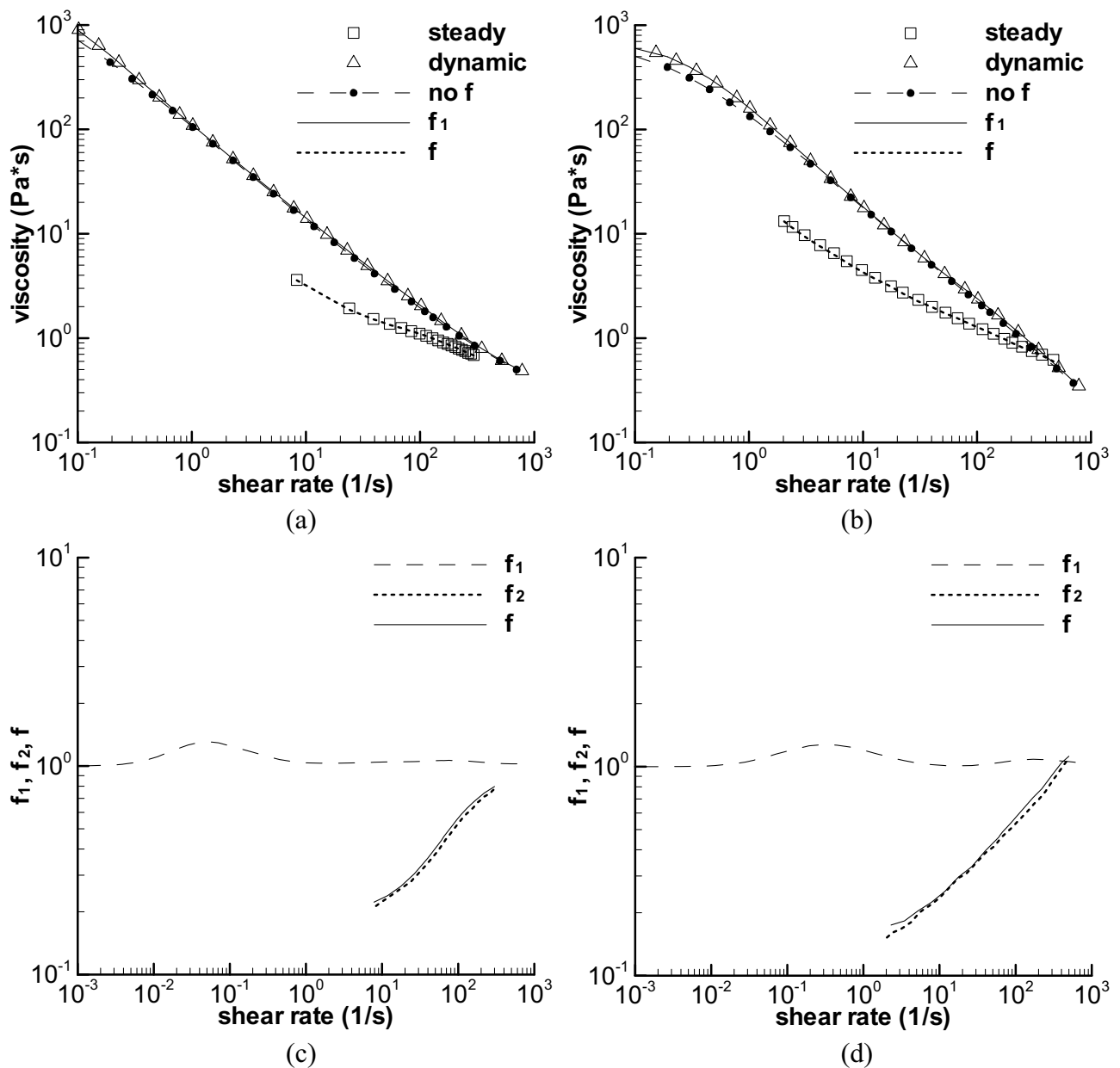


Figure 3: Characterization of steady shear viscosities of two CWCS pastes. (a) Calculated viscosities for the CWCS paste given by Youn and Rao [19], square is from the steady shear experiment by Youn and Rao [19], triangular is from the dynamic shear experiment, which is the complex viscosity calculated from the spectra in Table 2, and three lines are calculations here using equation (2). (b) Calculated viscosities for the CWCS paste given by Genovese and Rao [20], square is the experiment [20], triangular is the complex viscosity calculated by the spectra in Table 3, and the three lines are obtained by calculations using equation (2). (c) Parameters f_1 , f_2 , and f are used in the calculation for the paste by Youn and Rao [19]. (d) Parameters f_1 , f_2 , and f are used in the calculation for the paste by Genovese and Rao [20].

Figure 5 shows the deviations in the calculations of the power-law strain model from the experiments in previous studies [19,20]. The deviation between the power law calculation and the experiments by Youn and Rao [19] is lower than 15%, and that for the experiments by Genovese and Rao [20] is lower than 20%. The x in the deviation calculation

using equation (1) is the calculation of the power-law strain model, and x^* used is the corresponding experiment. The experimental errors of N_1 caused by different experimental conditions are approximately 25–40% in Figure 1(b), higher than the analysis errors in Figure 5, which are mostly below 10% at high shear rates. The present theoretical analysis

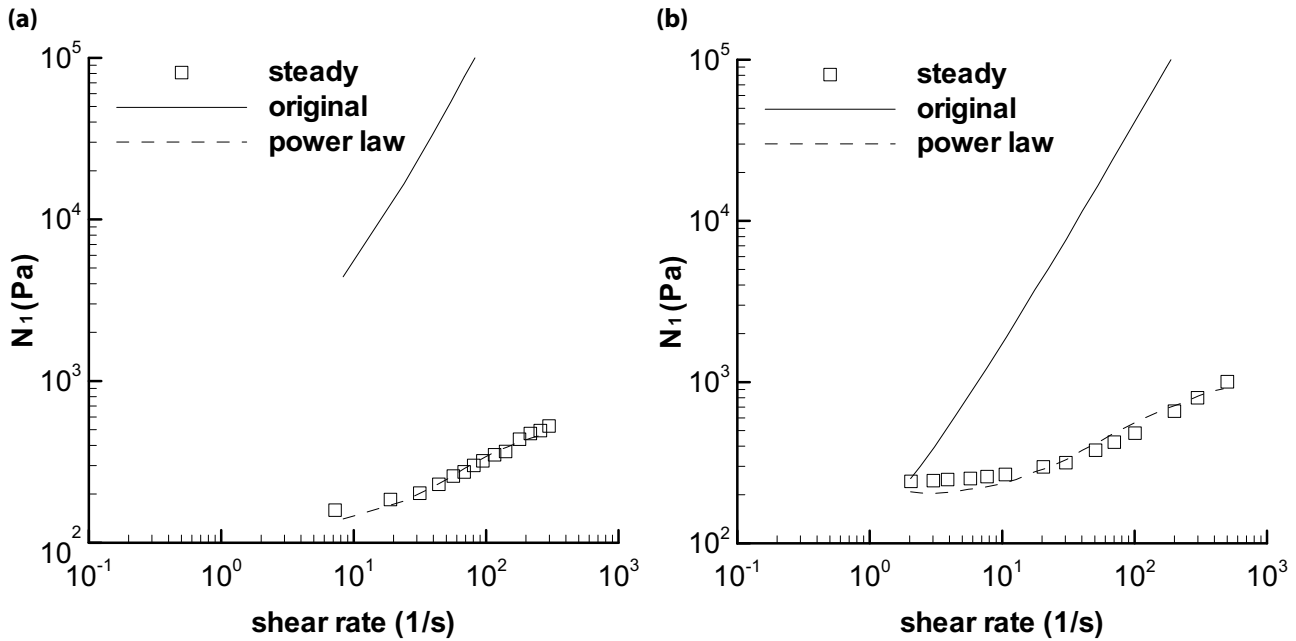


Figure 4: Characterizations of first normal stress differences for two CWCS pastes. (a) Calculated N_1 s for the CWCS paste given by Youn and Rao [19], square is the experiment by Youn and Rao [19], and two lines, solid, and dashed are the calculations here using equations (4) and (5), respectively. (b) Calculated N_1 s for the CWCS paste given by Genovese and Rao [20].

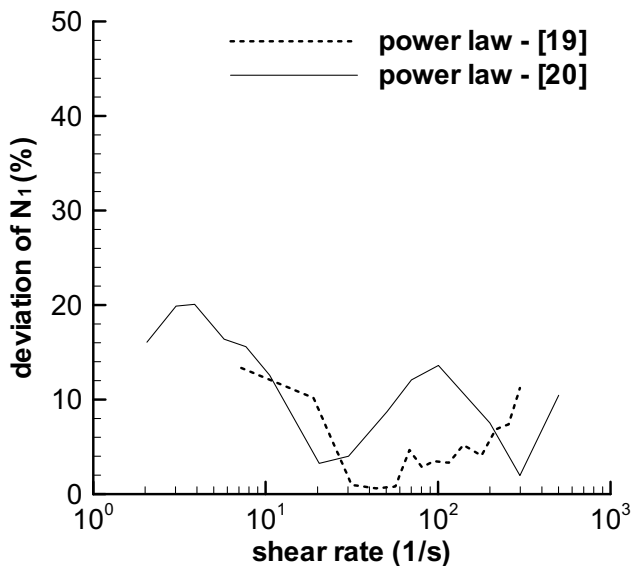


Figure 5: Deviations between the calculated N_1 s with the power-law strain model and the experiments in previous studies [19,20]. Dashed line is the deviation between the calculation here and the experiment by Youn and Rao [19], and the solid line is that for the experiment by Genovese and Rao [20].

shows some reasonable aspects, at least in terms of the good reproducibility of the experimental data.

The k and n obtained are listed in Table 4. In the study by Huang [9], the n values for the two CWCS pastes mixed with 300 g/kg sucrose approached 1.0, and therefore, a

Table 4: Parameters of power-law strain model

Experiment	k	n
Youn and Rao [19]	0.467	0
Genovese and Rao [20]	12.95	0.568

linear strain model was also proposed to express N_1 s. However, the n values in Table 4 for the two pure CWCS pastes do not approach 1.0, which could be caused by the sucrose. The $n = 0$ for the paste of Youn and Rao [19] indicates that the strain has no effect on the calculation of N_1 and cannot be considered in the calculation of N_1 for the paste. The reason is unclear for the $n = 0$ phenomenon.

At the end of the theoretical characterization of N_1 of the CWCS paste with sucrose [9], Huang proposed that the linear strain model should be checked for N_1 of other corn starch pastes or macromolecular fluids. The present work affirms that N_1 of the two pure CWCS pastes cannot be described by the linear strain model. Although pure CWCS also contains additional chemical bonds and can strengthen native starch [12,34], the linear strain model is not necessary for characterizing N_1 of such strengthened structures, and the power-law strain model works for pure CWCS pastes. Further examination of the application of the power-law strain model for corn starch pastes and certain macromolecular fluids is still needed, which could be the basis for improving the viscoelastic theory of fluids.

4 Conclusion

The steady shear viscoelastic properties of the two pure cross-linked waxy corn starch (CWCS) pastes, especially the first normal stress difference, were well characterized here using the integral viscoelastic equation with the power-law strain model. Experimental data for the two pastes were from the study by Youn and Rao [19] and Genovese and Rao [20]. The success of the power-law strain model in the calculation of N_1 for the two pastes, together with the study by Huang [9], indicates the failure of the traditional strain model in the characterization of N_1 for such fluids. Further examination of the power-law strain model is still needed.

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Ethical approval: The conducted research is not related to either human or animal use.

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