Rheological Measurement of Concentrated Pulp Fiber Suspensions in Oscillatory Shear Using a Novel Device

Di Yu,[a] Jing-song Zeng,[a,b,*] Ke-fu Chen,[a] Yu-cheng Feng,[a] and Xu Yang [a,c]

This paper presents a novel device based on an oscillating torsion resonator with continuously varying frequency capability to characterize the rheological properties of pulp fiber suspensions in a concentrated regime. Fiber suspensions made from unbleached wheat straw pulp at concentrations ranging from 5 wt% to 15 wt% were used. Pulp suspensions exhibit shear-thinning behavior up to a limited frequency value, after which Newtonian behavior prevails. Effects of frequency, fiber concentration, and beating process on suspension viscoelastic properties are discussed. The suspensions at different concentrations are structured in a similar way, leading to a weak gel-like structure. The storage modulus (*G*') of the suspension can be determined by a two-region (shear increasing – shear decreasing) profile, while loss modulus (*G*") keeps increasing for the whole frequency range investigated. At the same frequency, *G*' and *G*" increase nonlinearly with fiber concentration. The beating process brings a decrease in both *G*' and *G*". The power-law model is used for data fitting.

Keywords: Rheological measurements; Pulp fiber suspensions; Torsion resonator; Continuously varying frequency

Contact information: a: State Key Laboratory of Pulp and Paper Engineering, South China University of Technology, Guangzhou, 510640 China; b: School of Light Industry and Food Sciences, South China University of Technology, Guangzhou, 510640 China; c: Hangzhou Project & Research Institute of Electromechanical in Light Industry, Hangzhou, 310004 China;
** Corresponding author: zjs789zjs@126.com*

INTRODUCTION

The rheological properties of pulp fiber suspensions play a key role in various operations of papermaking. To achieve viable commercial papermaking processes, the raw material must typically be reacted and processed in suspension form. Rheological measurements of pulp fiber suspensions are important in both industrial production and scientific research. Many studies of pulp fiber suspensions have been carried out over the years, and the early work has been well described in the literature (Petrie 1999; Kerekes 2006; Cui and Grace 2007; Derakhshandeh *et al.* 2011). However, pulp fibers are slender, with a relatively large mean length, a wide distribution in length, and a high aspect ratio. In shear flow, fibers aggregate into local mass concentrations called flocs, which typically contain a few fibers. Having a higher concentration than the average concentration of suspension, flocs are also higher in strength than the suspension average (Kerekes 1983). Consequently, pulp fiber suspensions are heterogeneous in both mass and strength. This is an important factor in pulp fiber suspension rheology that presents unique challenges when subjected to rheological measurements.

Pulp fiber suspensions exhibit viscous as well as elastic behavior and are therefore considered viscoelastic. Taking both viscosity measurements and viscoelasticity

measurements in pulp fiber suspensions is not simple. Studies have been devoted to characterizing the rheological properties of pulp fiber suspensions using various strategies (Chase *et al.* 1989; Chen and Chen 1997; Swerin 1998; Daisuke *et al.* 2002; Djalili-Moghaddam and Toll 2006; Lasseuguette *et al.* 2008; Stickel *et al.* 2009; Agoda-Tandjawa *et al.* 2010; Derakhshandeh *et al.* 2010a,b; Chaussy *et al.* 2011; Derakhshandeh *et al.* 2012).

A noteworthy approach to measuring the rheological properties of pulp fiber suspensions is by oscillatory shear. This approach works well, especially for evaluations of viscoelasticity. Viscoelastic properties of pulp suspensions were measured using oscillatory strain in a parallel-plate rheometer in the consistency range of 3 to 8% in terms of storage and loss moduli (Swerin *et al.* 1992). Using the same approach, the elastic modulus was shown to be independent of the applied frequency (Damani *et al.* 1993). The viscoelasticity of two fiber suspensions with different fiber sizes in the presence of flocculants was examined. All measurements were performed in the oscillatory mode using a modified rheometer (Swerin 1998). The viscoelasticity of bio-mass slurries having average fibre lengths of 0.1 mm and an aspect ratio from 1 to 20 was measured using both parallel-plate and vaned geometries (Stickel *et al.* 2009). Despite excellent improvement obtained due to these efforts, the developed techniques obtained the rheological properties of complex fluids only at limited frequencies. The data were collected in a narrow frequency range (*i.e.,* 0.1 Hz to 10 Hz) or at several given frequencies, lacking the continuously varying frequency capability.

The purpose of this work was to characterize the rheological properties of pulp fiber suspensions by using a novel device based on an oscillating torsional resonator. The modified rheometer exhibits a continuously-varying-frequency capability unlike the traditional torsional resonator exhibiting only one oscillation resonance frequency, and it performed well in recent studies (Wang *et al.* 2008, 2010a,b; Xiong and Zhang 2010). In this work, the complex viscosity and viscoelasticity of concentrated pulp suspensions over a fiber mass concentration range of 5%, 10%, and 15% were analyzed and the effect of concentration and the beating process on the viscoelastic properties of wheat straw pulp suspensions was illustrated.

Theoretical Analysis

According to previous theoretical descriptions (Schrag and Johnson 1971; Blom and Mellema 1984; Wang *et al.* 2008), motion with one torsional oscillation mode of a forced oscillating-cup resonator is described by Eq. 1,

$$I\ddot{\theta} + (Z + D)\dot{\theta} + K\theta = M \quad (1)$$

where θ is the angular displacement; M is the driving torque exerted on the pendulum; I is the equal effective inertial momentum of the resonator; $Z = R + iX$ is the torsion impedance due to the surrounding liquid; R is the damping of the torsion pendulum; and K is the torsion-spring constant. If the driving torque is provided by $M = M_0 e^{i\omega t}$ (where $M_0 = |M|$ and ω is radian frequency), the angular displacement θ is $\theta = \theta_0 e^{i(\omega t - \varphi)}$ (where $\theta_0 = |\theta|$ and φ is phase angle, and angular displacement lags M). The response equations are provided (Wang *et al.* 2008, 2010) as in Eq. 2:

$$\theta_0 = \frac{M_0/I}{\sqrt{\left(\omega_0^2-\omega^2-\omega X/I\right)^2+\omega^2(R+D)^2/I^2}} \quad (2)$$

and in Eq. 3,

$$\tan\varphi = \frac{\omega(R+D)/I}{\omega_0^2-\omega^2-\omega X/I} \quad (3)$$

where $\omega_0 = \sqrt{K/I}$. From Eqs. 2 and 3, Eqs. 4 and 5 are obtained:

$$R = \frac{M_0}{\omega\theta_0}\frac{1}{\sqrt{1+1/\tan^2\varphi}} - D \quad (4)$$

$$X = \frac{M_0}{\omega\theta_0}\frac{1}{\tan\varphi\sqrt{1+1/\tan^2\varphi}} + \frac{1}{\omega}(\omega_0^2-\omega^2) \quad (5)$$

As with the analysis in previous studies (Glover *et al.* 1968; Blom and Mellema 1984; Wang *et al.* 2008), if an oscillating-cup has a wall far thinner than two spacing between the resonator and the sample holder, the relation between *Z* and the characteristic plane wave shear impedance (Z_{pl}) will approximately satisfy the criteria of Wang *et al.* (2008), as shown in Eq. 6,

$$Z \approx A_0 Z_{pl} \quad (6)$$

where $Z_{pl} = R_{pl} + iX_{pl}$ is defined as the ratio of the force per unit area to the linear velocity of the surface being considered; $A_0 = 4\pi a^3 l$ is the apparatus constant, at which a is the radius of the oscillating cup; and l is the length of the cup inserted into the liquid. From Eqs. 4 through 6 after measurements in both air and liquid, the components of the characteristic plane wave shear impedance is determined by Eqs. 7 and 8,

$$R_{pl} = E_4\left[\frac{1}{\omega\theta_{0l}\sqrt{(1/tan\,\varphi_l)^2+1}} - \frac{1}{\omega\theta_{0air}\sqrt{(1/tan\,\varphi_{air})^2+1}}\right] \quad (7)$$

$$X_{pl} = A_4\frac{\omega_0^2-\omega^2}{\omega} - E_5\frac{1}{\omega\theta_{0l}\,tan\,\varphi_l\sqrt{(1/tan\,\varphi_l)^2+1}} \quad (8)$$

where φ_{air} is phase angle; φ_l , θ_{0air} is angular displacement; θ_{0l} and ω are the continuously varying frequency in air and in liquid, respectively; and $A_4 = I/4\pi a^3 l$, $E_4 = E_5 = M_0/4\pi a^3 l$ are the apparatus constants.

Equations 7 and 8 provide values of R_{pl} and X_{pl} at every excited frequency (ω) can be calculated from the corresponding phase angle and angular displacement data (φ_{air}, φ_l, θ_{0air}, and θ_{0l} values) after determining values of apparatus constants (A_4, E_4, E_5) by calibration. The values φ_{air}, φ_l, θ_{0air} and θ_{0l} are measured both in liquid and in vacuum. Finally, viscoelastic properties of samples can be obtained from the relationship between the components of the complex shear modulus $G^* = G' + iG''$ and both R_{pl} and X_{pl}, as provided by Ferry (1980) in Eqs. 9a and 9b:

$$G'(\omega) = \frac{\left(R_{pl}(\omega)\right)^2-\left(X_{pl}(\omega)\right)^2}{\rho} \quad (9a)$$

$$G^{"}(\omega) = \frac{2\left(R_{pl}(\omega)\right)\left(X_{pl}(\omega)\right)}{\rho} \quad (9b)$$

as well as the Newtonian fluid viscosity (Ferry 1980) in Eq. 10:

$$\eta(\omega) = \frac{G^{"}(\omega)}{\omega} = \frac{2\left(R_{pl}(\omega)\right)\left(X_{pl}(\omega)\right)}{\omega\rho} \quad (10)$$

Torsion resonators with one vibrating mode often were employed for discrete frequency measurement. Through detecting the resonance curve data in both air and liquid, the torsion resonator will have the continuously varying frequency capability to measure frequency-dependent viscoelastic properties of complex liquids by using Eqs. 7 through 10.

EXPERIMENTAL

Materials

Instrument

The torsion resonator apparatus (Fig. 1) was developed from the resonant absorption mechanical spectrometer (Zhang 2003), with the effective inertia two-thin-walled cups joined by a cylindrically shaped torsion spring fixed to the base plate and suspended by a negligible effect silk thread. Apparatus constants in this study are provided in Table 1. Methods to measure and calculate the values of the apparatus constants were discussed in a previous paper (Wang *et al.* 2008, 2010b).

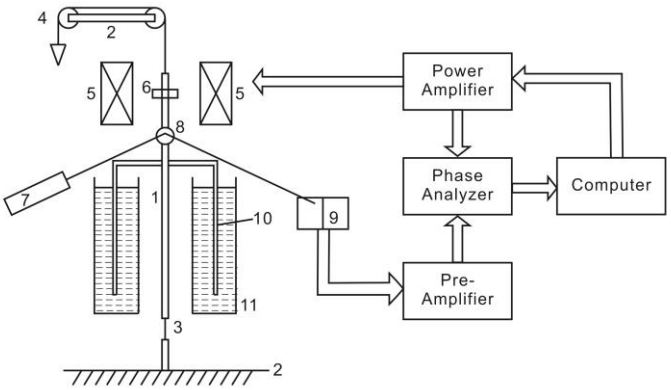

Fig. 1. Schematic diagram of the torsion resonator apparatus: (1) rotating grip and pendulum rod, (2) base, (3) torsion spring rod, (4) pulley and counterweight, (5) pair of driving coil, (6) magnet, (7) lamp unit with blade slit, (8) reflect mirror, (9) differential photocell, (10) rotational cylinder, and (11) double-layer container

The fibers used were from unbleached wheat straw kraft pulps. A laboratory PFI refiner was employed to beat the raw pulp, and the Shopper Riegler degree (SR°) was measured according to the standard ISO 5267-1:1999 (2010). The pulp was beaten to

38°SR, and the fiber fines were removed by 100 mesh (~ 150 μm) vacuum filtration. The fiber morphology was characterized with a fiber analyzer (Kajaani FS300, Kajaani, Finland). The average fiber length was 0.35 mm, the average fiber width was 15.12 μm, the coarseness was 0.105 mg/m, and the fiber curl was 13%.

Table 1. Apparatus Constants

a (cm)	l (cm)	I (10^{-5} kg m^2)	M_0 (10^{-6} N m)	K (N m)	f_0 (Hz)	Δf_{air} (Hz)	Q factor in air
1.5	4.5	2.96	1.96	0.5883	71.5	0.102	701
A_4 (kg/m^2)	E_4 (N/m^3)	E_5 (N/m^3)					
2.6	2.2	2.1					
Note: I is the equal effective momentum of inertia of the resonator; M_0 is the amplitude of the driving torque; K is the spring constant of the resonator; f_0 and Δf_{air} are the resonance frequency and bandwidth of the resonator in air, respectively							

Sample preparation

The raw pulp consisted of 73.8% water by mass. Samples were made into fiber mass concentrations (w/w%) from 5 to 15%, as shown in Fig. 2. The 15% sample was chosen for further beating treatment. Sample suspensions were well dispersed before each rheological measurement to eliminate thixotropy.

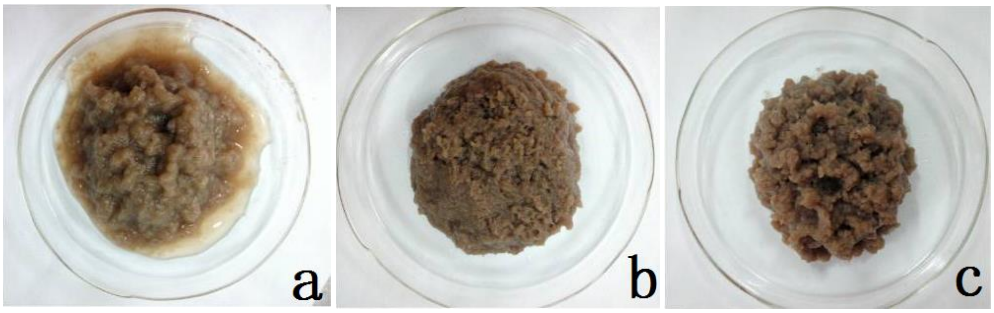

Fig. 2. Images of samples at various fiber concentrations (w/w%): (a) 5%, (b) 10%, and (c) 15%

RESULTS AND DISCUSSION

Effect of Frequency on Rheological Properties

Figure 3 shows curves of complex viscosity (η^*) *versus* frequency under oscillatory shear with continuously varying frequency for different fiber concentrations. At all three concentrations, the suspensions noticeably showed non-Newtonian flows. Similar behaviors have been observed on other fiber suspensions, such as cotton cellulose fiber suspension (Daisuke *et al.* 2002). In the lower frequency region (1 to 50 Hz), shear-thinning behavior was observed.

Beyond a limited frequency level (75 ± 5 Hz), Newtonian behavior was observed. This shear thinning-plateau viscosity profile has also been found in rheological characterization of softwood and hardwood bleached kraft pulp (Chen *et al.* 2003). The viscosity of the suspension increased with fiber consistency.

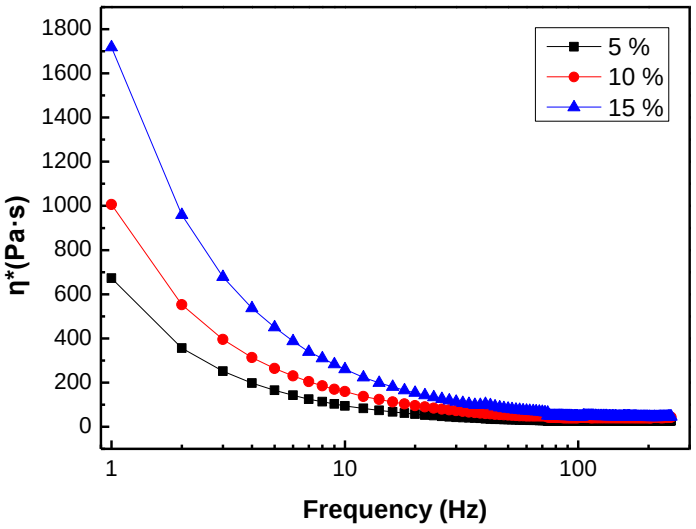

Fig. 3. Effect of frequency on the complex viscosity at different fiber concentrations

Effects of frequency on storage modulus (*G*') and loss modulus (*G*") are shown in Fig. 4. The storage modulus of suspension under oscillatory shear with a continuously varying frequency for different consistencies can be described by a two-region profile (*i.e.*, shear increasing - shear decreasing), while the loss modulus keeps increasing for the whole frequency range investigated. The storage modulus *G*' increases much more rapidly with concentration, and is greater than the loss modulus *G*". The storage modulus plays a dominant role in the rheological behavior of the suspension. As shown in Fig. 5, the *G*'/*G*" ratio was on the order of less than 10 for all the concentrations investigated. This means that at concentration of 5 w/w%, 10 w/w%, and 15 w/w%, the suspensions are structured similarly, leading to a weak gel-like structure. The overlapping of *G*' and *G*" emerged in the 5 w/w% sample in our tests.

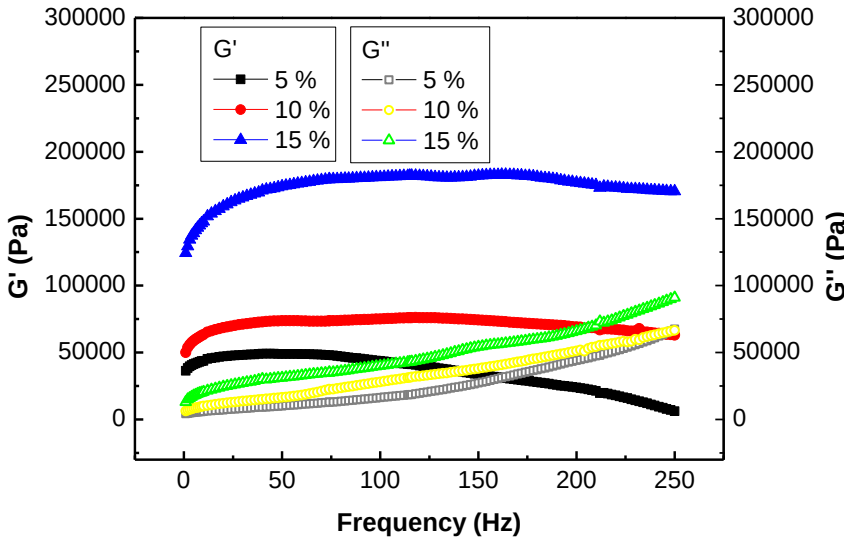

Fig. 4. Effect of frequency on the storage and loss moduli at different fiber concentrations

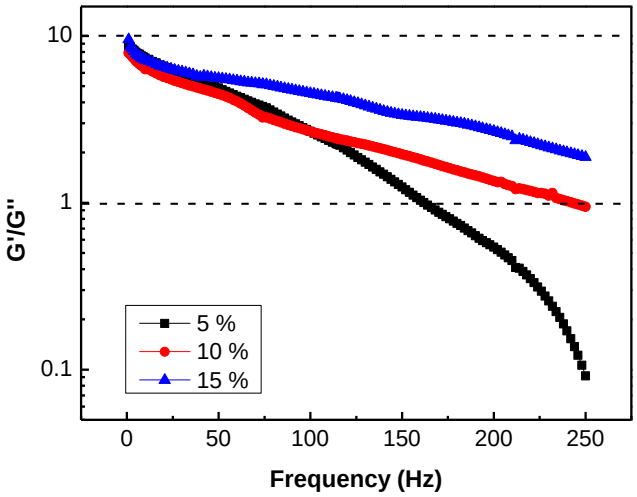

Fig. 5. Effect of frequency on the ratio of the storage modulus to the loss modulus at different fiber concentrations

The power-law model was used for data fitting, as can be seen in Tables 2. The results in Table 2 show the exponent value of the fitting curve at different concentrations, which seem to be independent of concentration. To the contrary, the constant *K* increases with concentration.

Table 2. Fitting of η* - Frequency at Different Concentrations

Concentration (w/w%)	*K*	*n*	r^2
5	626.56 ± 9.96	-0.75 ± 0.010	0.97
10	941.81 ± 13.36	-0.72 ± 0.008	0.97
15	1672.79 ± 11.98	-0.78 ± 0.005	0.99
Power-law model, $\eta^*=K\omega^n$ Mean values ± standard deviation η^*, complex viscosity (Pa·s); ω, frequency (Hz); *K, n* constants; r^2 determination coefficient			

Effect of Pulp Consistency on Rheological Properties

Rheological results as shown in Figs. 3 and 4 have been obtained from previous measurements for concentrated pulp fiber suspensions at concentrations ranging from 5 to 15 w/w%. Four frequencies were chosen, 1 Hz and 10 Hz in the shear - thinning regime, and 100 Hz and 200 Hz in the Newtonian regime. The complex viscosity (η^*), storage modulus (G'), and the loss modulus (G'') increase with the concentration, as shown in Figs. 6, 7, and 8. The power-law model was used for data fitting, as seen in Tables 3.

Table 3. Fitting of η* - Concentration at Different Frequencies

Frequency (Hz)	*K*	*n*	r^2
1	125.86 ±17.68	1.15 ± 0.04	0.91
10	17.48 ± 6.74	1.09 ± 0.05	0.97
100	12.65 ± 2.00	0.49 ± 0.06	0.97
200	13.78 ± 2.07	0.45 ± 0.02	0.97
Power-law model, $\eta^*=KC^n$ Mean values ± standard deviation η^*, complex viscosity (Pa·s); *C*, concentration (w/w%); *K, n* constants; r^2 determination coefficient			

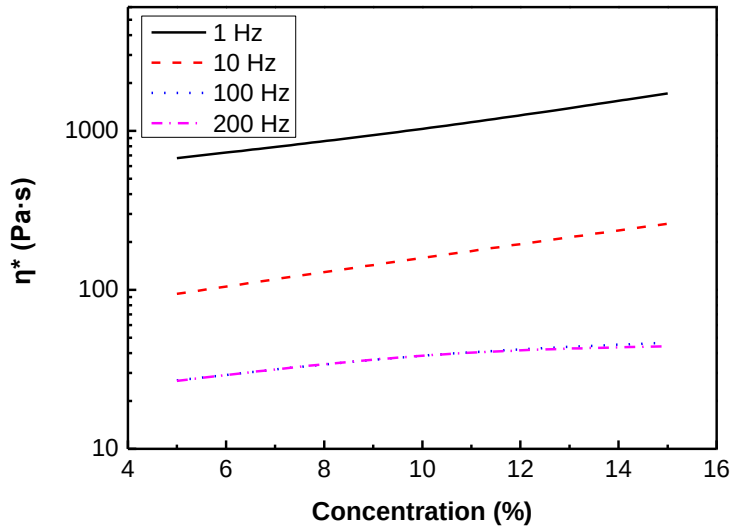

Fig. 6. Effect of fiber concentration on the complex viscosity at different frequencies

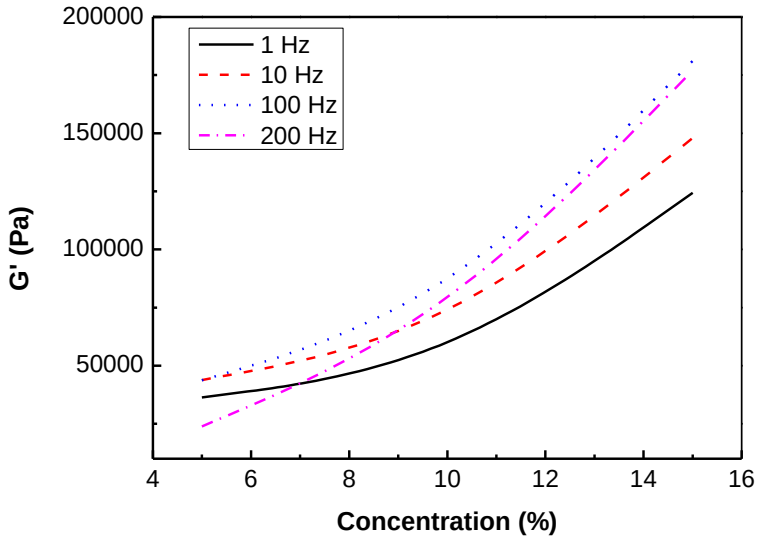

Fig. 7. Effect of fiber concentration on the storage modulus at different frequencies

Much attention was given by earlier researchers to the concentration dependence of *G*'. From this experimental data reported previously attempts were made to describe the concentration dependence of *G*' on a theoretical basis, starting with the fact that a change in slope occurred as the concentration increased. The exponent value from micro-fibrillar cellulose gels appears to depend essentially on the fiber mass fraction (Pääkkö *et al.* 2007; Hill 2008). In addition, a study by Daisuke *et al.* (2002) showed that in the relationship $G' = KC^n$, which was the same power-law model used in this study. The exponent value remained constant over a wide range of fiber length and diameters, contrary of the *K* factor, which changed according to the types of fibers.

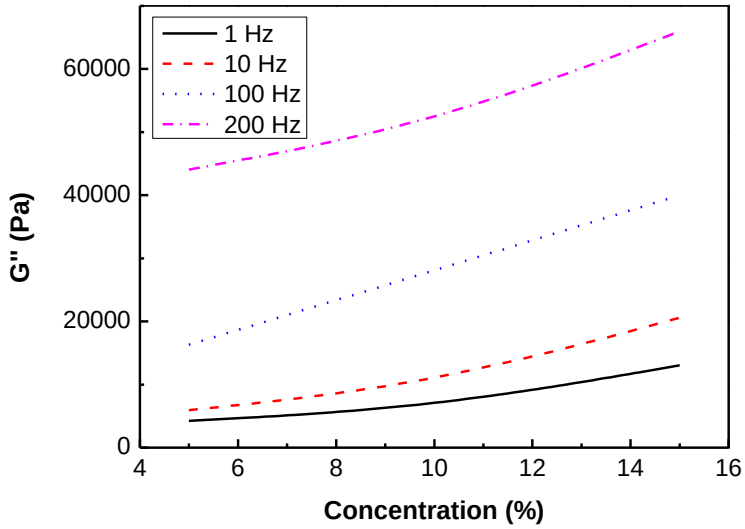

Fig. 8. Effect of fiber concentration on the loss modulus at different frequencies

It was concluded that the parameter *K* reflects the individual fiber characteristics, and that *n* reflects the structural property of the entire suspension. From these previous observations and the results in this study, we may state that the exponent value obtained in the present concentrated pulp fiber suspension reflects the network structure of the system.

Effect of Beating Process on Viscoelastic Properties

The beating process is performed on cellulose fibers in pulp suspensions through a refiner. The properties of paper depend on fiber morphology, which is strongly altered in beating process to become hydrated, fibrillated, and shortened.

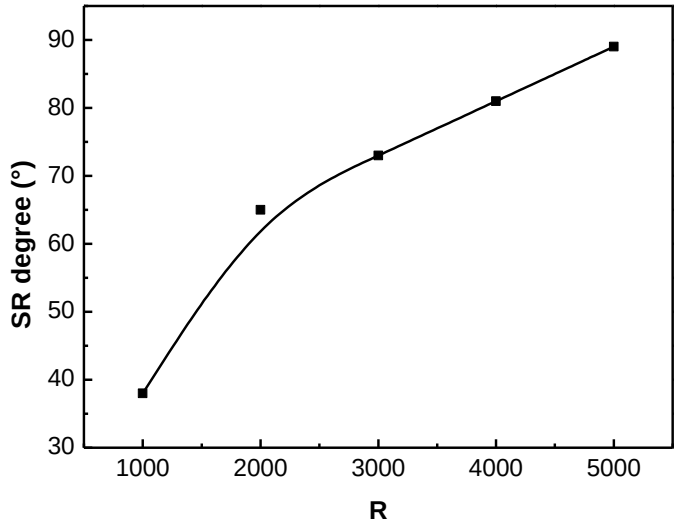

Fig. 9. Effect of number of revolution on SR degree

All these transformations on fibers will lead to an increase of their external fibrillation and flexibility, and consequently to an increase of the mechanical properties. This makes the beating operation an important part in papermaking. After the beating operation, the filtration resistance of the pulp suspension increases, and it is commonly assessed by the Shopper-Riegler degree (SR degree).

Figure 9 shows the beating process curve in this study, and the SR degree increases with the number of beater bar revolutions. The beating number is the working rounds of the lab PFI refiner beater bars. Frequency dependence of the storage modulus (*G*') and loss modulus (*G*") is shown in Fig. 10.

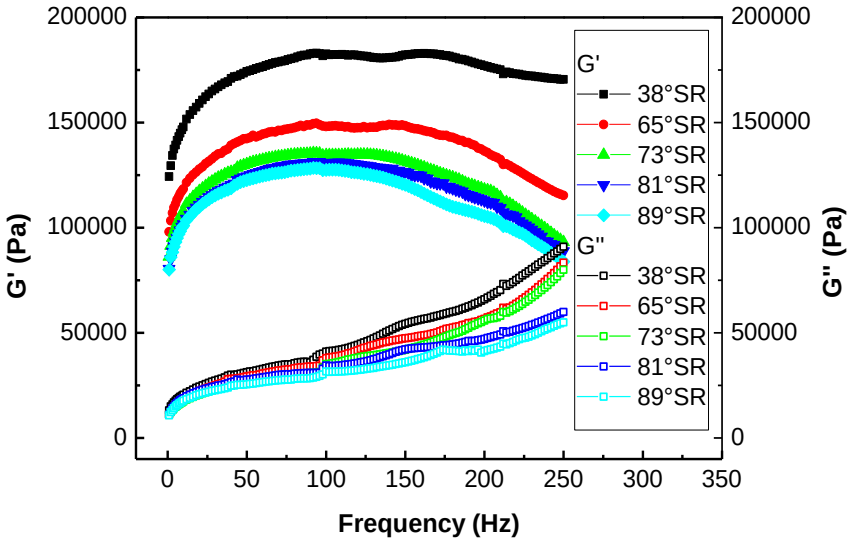

Fig. 10. Effect of frequency on storage modulus at different SR degrees and constant fiber concentration 15% w/w

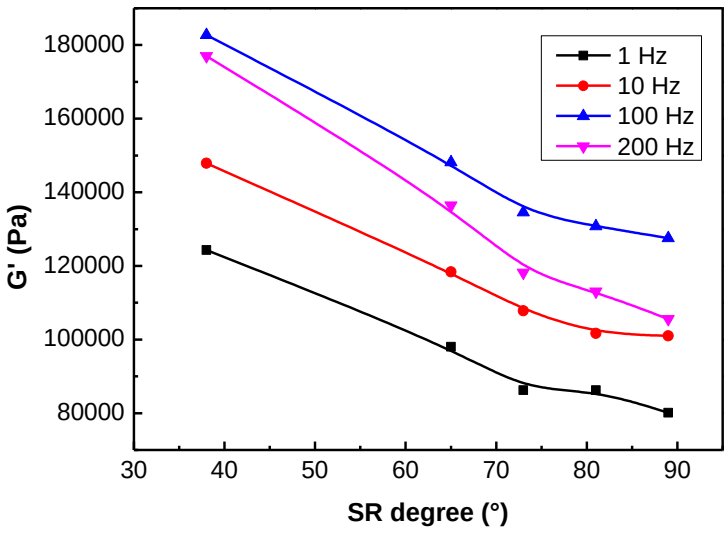

Fig. 11. Effect of SR degree on storage modulus at different frequencies and constant fiber concentration 15% w/w

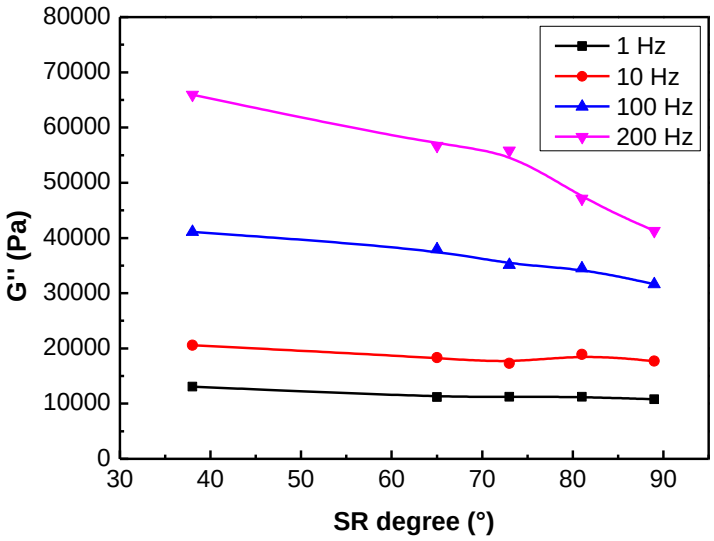

Fig. 12. Effect of SR degree on loss modulus at different frequencies and constant fiber concentration 15% w/w

The storage modulus of the suspension under oscillatory shear with a continuously varying frequency for different SR degrees can also be determined by a two-region profile while the loss modulus keeps increasing for the entire frequency range investigated (as discussed above). Both *G*' and *G*" decrease at the same time with an increase in SR degree for the whole frequency range investigated. Storage and loss modulus as a function of SR degree at different frequencies are shown in Figs. 11 and 12. These results indicate that "cutting" may play a key role in the beating process of this experiment, as the fibers are shortened with an increase in the beating level.

CONCLUSIONS

1. The pulp fiber suspensions exhibit shear-thinning behavior at lower frequency and Newtonian behavior beyond a limited level of frequency (75 ± 5 Hz). The storage modulus (*G*') of the suspension exhibits a shear-increasing then shear-decreasing profile, while the loss modulus (*G*") keeps increasing for the entire frequency range investigated. The *G*'/*G*" ratios of the suspensions are of the order less than 10, leading to a weak gel-like viscoelastic behavior.
2. As fiber concentration increases, *G*' and *G*" increase nonlinearly. The power-law model was used for fitting of experiment curves. The exponent value obtained in the *G*' curve fitting seems to be independent of concentration, which may reflect the network structure of the suspension system.
3. Both *G*' and *G*" simultaneously decrease with an increase in the SR degree for the entire frequency range investigated.

ACKNOWLEDGMENTS

This work was supported by a grant (No. 2010CB732205) from the Major State Basic Research Development Program of China (973 Program).

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Article submitted: June 23, 2014; Peer review completed: September 14, 2014; Revised version received and accepted: October 23, 2014; Published: November 14, 2014.