# Thixotropic Flow Behaviour in Chemical Pulp Fibre Suspensions

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This paper presents results on the thixotropic behaviour of hardwood and softwood pulp fibre suspensions. Three rheological tests including hysteresis-loops, creep tests, and step-wise experiments were used to investigate the thixotropic rheology. Both suspensions exhibited a plateau in their flow curves where a slight change of the applied shear stress led to a dramatic change in the corresponding shear rates. During creep experiments under controlled stress, they evolved either towards a rapid shear (liquid regime) or stoppage (solid regime), depending on the relative values of the imposed stress, leading to a viscosity bifurcation around a critical stress. The transient response of pulp to step changes in shear rate was marked by a characteristic time, which can be used to understand the rate of structural change for pulp suspensions. Moreover, the yielding and thixotropic behaviour of the pulp suspensions were highly dependent on shear history and the time of rest prior to the measurement.

*Keywords: Non-Newtonian fluids; Rheology; Time-dependence; Yielding; Pulp fibre suspensions*

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## INTRODUCTION

Suspensions of materials such as clay, colloidal gels, pastes, minerals, fibres, *etc.*, are rheologically complex, since they consist of fine particles dispersed in a liquid (Coussot 2005; Mewis and Wagner 2009). Various specific particle-particle and particle-medium interactions may lead to the formation of microstructures in a suspension at rest. The shear-induced changes in microstructures complicate the rheological properties of these suspensions, and different types of macroscopic flow behaviours such as shear thinning, yielding, viscoelasticity, and thixotropy can be detected (Møller *et al.* 2006; Lopez-Sanchez and Farr 2012). In terms of both practical and fundamental significance, thixotropy and yield stress are two of the most important rheological properties of suspensions (Coussot *et al.* 2002a; Denn and Bonn 2011; Derakhshandeh *et al.* 2012).

Thixotropy is the continuous decrease of viscosity over time when a sample is sheared from its rest, and the subsequent recovery of viscosity over time when the flow is discontinued (Barnes 1997; Mewis and Wagner 2009). From a physical point of view, this effect seems to result from shear/structure coupling *via* competition between a restructuring process (aging) and shear-induced destructuration (shear rejuvenation) (Coussot *et al.* 2002a; Ovarlez *et al.* 2013). Detailed reports of thixotropy can be found in existing reviews of the subject (see Bauer and Collins 1967). More recent and general reviews of the field are those by Barnes (1997) and Mewis and Wagner (2009).

Pulp fibre suspensions are heterogeneous mixtures, within which fibres are considered to be in a solid phase. The unique behaviour of the fibres in suspension gives rise to their complex rheology (Kerekes 2006; Derakhshandeh *et al.* 2011). Pulp suspensions typically consist of fibres 15 to 30 µm in diameter and 1 to 3 mm in length. Fibres mechanically entangle to form fibre flocs with an average size of 2 to 3 cm. Increased fibre consistency also increases the number of fibre-fibre interactions, forming a network structure throughout the suspension. The degree of interaction between neighbouring fibres in a flowing pulp suspension can be estimated using the crowding number, $N$, which is defined as the number of fibres in a volume swept out by the length of a single fibre (Kerekes and Schell 1992). It can be described as follows,

$$N = \frac{2}{3} C_v \left(\frac{L}{d}\right)^2 \approx \frac{5C_m L^2}{\delta} \tag{1}$$

where $L$ is fibre length (m), $d$ is fibre diameter (m), $\delta$ is fibre coarseness (kg/m), $C_v$ is volume consistency (%), and $C_m$ is mass consistency (%) of the suspension.

In previous studies, Mason (1950) identified $N = 1$ as a “critical concentration” at which collisions first occur among fibres in shear flow. Kerekes and Schell (1992) showed that at $N = 60$ the number of contact points per fibre is approximately three, which is enough to establish a coherent fibre network. In a subsequent study, Martinez *et al.* (2003) identified another critical crowding number, $N_{gel}$ (approximately 16), which is a “gel crowding number”. Below this value, fibre suspensions are essentially dilute, while above it they interact significantly but are not completely immobilized as they are at $N = 60$. The forces at fibre contact points create fibre flocs that give the network mechanical strength. To create motion in the suspension, the yield stress—which characterizes the network strength—must be overcome; it is achieved using shear forces larger than the yield stress (Divoux *et al.* 2011; Bonn *et al.* 2015). There have been extensive studies of yield stress on fibre suspensions, and various measuring techniques have been used to determine the yield stress of pulp suspensions (Wikström and Rasmuson 1998; Kerekes 2006; Derakhshandeh *et al.* 2010a). Among these studies, Martinez *et al.* (2003) investigated the impact of primary fibre variables on the yield stress of pulp suspensions and set up an equation as follows,

$$\tau_y = \alpha\left(N - N_{gel}\right)^{\beta} \tag{2}$$

where $\tau_y$ is the yield stress (Pa), $N_{gel}$ is the gel crowding number (*i.e.*,$N_{gel}$= 16), and $\alpha$ and $\beta$ are constants specific to the fibre type.

Besides the yield stress, pulp fibre suspensions also exhibit thixotropic behaviour. When studying the yielding and thixotropic behaviour of mechanical pulp fibre suspensions at a mass concentration of 6%, Derakhshandeh *et al.* (2012) found that concentrated mechanical pulp suspensions exhibited a plateau in their flow curves where a slight increase in the shear stress generated a jump in the corresponding shear rate. In addition, both shear history and the time of rest prior to the measurement played significant roles on the rheology of mechanical pulp suspensions. In practice, thixotropic properties of pulp suspensions are often not taken into account due to the lack of simple, systematic, and relevant procedures to characterize them.

Measuring thixotropic materials is very difficult. Phenomena such as wall slip, shear banding or other shear heterogeneities, sedimentation, shear fracture, and irreversible changes in the sample can all introduce errors (Meeker *et al*. 2004; Mewis and Wagner

2009). Typical ways of measuring thixotropy include hysteresis loops, start-up and creep experiments, and transient experiments created by changing the shear stress or shear rate in a stepwise manner (Barnes 1997). The hysteresis technique was introduced by Green and Weltmann (1943) and consists of linearly increasing and decreasing the shear rate (or sometimes shear stress) between zero and a maximum value. A serious limitation of the hysteresis loop is that shear rate and time effect are coupled in this experiment, even though it provides a relative measure of thixotropy. In a start-up experiment, the sample is suddenly subjected to a constant shear rate after it has been at rest, whereas in a creep experiment a constant shear stress is applied. The major drawbacks of the hysteresis loop method can be avoided when using stepwise changes in shear rate or shear stress; this approach is typically followed in thixotropic suspensions (Völtz *et al*. 2002).

Chemical pulp fibres are produced through the chemical extraction of lignin and hemicellulose, and this type of pulp is important for the international pulping industry. For paper producers, chemical pulp fibres are mainly used to meet high quality paper standards while still obtaining a desirable yield (Chauhan *et al.* 2011). However, there is little information on the thixotropic behaviour of chemical pulp suspensions. Thus, the aim of this paper was to study the thixotropy of chemical pulp fibre suspensions by using hysteresis loops, creep experiments, and step-wise experiments. The influence of rest time on the thixotropy of pulp suspensions was simultaneously evaluated. The obtained results provide some basic understanding of the microstructural behaviour of these suspensions.

**Table 1.** Fibre Properties of Hardwood and Softwood Pulps

| Type | Average Fibre Length (mm) | Average Fibre Width (μm) | Coarseness (mg·m$^{-1}$) | Fines Content (%) |
|---|---|---|---|---|
| Hardwood | 0.745 | 17.8 | 0.077 | 31.82 |
| Softwood | 2.399 | 27.5 | 0.223 | 14.36 |

## EXPERIMENTAL

### Materials

Two commercial bleached kraft pulps based on hardwood and softwood were obtained in a dried form. The softwood pulps, containing 100% spruce pine fir, were supplied by Canfor Pulp Products (Prince George, BC, Canada). The hardwood pulps were produced from eucalyptus and were obtained from CMPC (Santa Fe, Chile). The average fibre length, fibre width, coarseness, and fines content for both pulps were determined in-house using a Fibre Quality Analyzer (FQA) (OpTest Equipment Inc., Hawkesbury, Canada), as presented in Table 1. Both raw materials were swollen in distilled water for 24 h and then dispersed using a blender (PTI, Vorchdorf, Austria) for 5 min to achieve a consistent suspension. Because air bubbles become trapped in concentrated suspensions and induce unwanted variance into the results, hardwood and softwood suspensions at crowding numbers of 120 were prepared using deionized water for the rheological measurements, except in the tests to determine the effect of crowding number on the yield stress.

## Rheometry

A measure of the relative importance of shear forces compared to Brownian motion is given using the Peclet number (Eq. 3),

$$Pe = 6\pi\eta\dot{\gamma}R^3/K_BT \tag{3}$$

where $K_B$ is the Boltzmann constant ($m^2s^{-2}kgK^{-1}$), $R$ is the radius of the particles (m), $T$ is the absolute temperature (K), $\dot{\gamma}$ is the shear rate ($s^{-1}$), and $\eta$ is the medium viscosity (Pa·s). For suspensions with higher Peclet numbers, the contribution of the Brownian motions to structure build-up in pulp is negligible (Barnes 1997; Derakhshandeh *et al.* 2012). As a rule of thumb, the crossover between Brownian and non-Brownian particle systems is often taken to be between 1 and 5 μm (Moller *et al.* 2009). Smaller particles remain in suspension due to Brownian motion, whereas large systems are practically immobile. Pulp fibre suspensions are a good example of the latter case.

All rheological measurements were made using a controlled-stress rheometer (Brookfield Engineering Laboratories Inc., MA, USA). The main measuring system used rotating vane-in-cup geometry, which has an advantage over other geometries, in that wall slip and sedimentation effects can be avoided (Barnes and Nguyen 2001; Cullen *et al.* 2003). However, there are assumptions made when using vaned-geometry devices, *i.e.*, that the fluid between the blades rotates with them and acts as a rigid body without any secondary flow (Ein-Mozaffari *et al.* 2005; Derakhshandeh *et al.* 2010b). The vane had four blades with a diameter of 40 mm, a length of 80 mm, and a cup diameter of 100 mm.

Rheological experiments included hysteresis loops, transient experiments performed by changing the shear rate in a stepwise manner, and creep experiments. Before measurements, pulp fibre suspensions were pre-sheared at 200 $s^{-1}$ for 3 min, followed by relaxation for a given period of time to ensure reproducible initial conditions during subsequent tests (Cloitre *et al.* 2000; Erwin *et al.* 2010). All measurements were conducted at 26 °C.

# RESULTS AND DISCUSSION

## Hysteresis Loops

In the hysteresis experiment, the shear stress was increased and decreased linearly with ramp rates of 0.2 Pa/s and 0.1 Pa/s, respectively. Pulp suspensions and the instantaneous shear rate of hardwood and softwood were recorded. The qualitative responses of the chemical pulp suspensions at a crowding number of 120 during the stress ramp are illustrated in Fig. 1. Different regions were distinguished in the stress ramp. For low imposed stresses, a continuous increase of shear rate was observed. This result is possibly due to small deformations corresponding to small irreversible rearrangements. At a critical stress of 64 Pa, there was a dramatic increase in the shear rate (from about 0.2 $s^{-1}$ to 7 $s^{-1}$), generating an apparent plateau in the flow curve, which suggests that no significant flows are feasible below this critical shear stress. Hence, this critical stress is identifiable as a (time-dependent) static yield stress, which is a measure of the mechanical strength of the suspension structure (Vasu *et al.* 2013). The plateau in the flow curve is due to the shear banding effect, which has also been observed in colloidal gels, bentonite suspensions, and other soft glassy materials (Coussot *et al.* 2002b; Møller *et al.* 2006). Just after this plateau, there was an abrupt change in the slope of the stress-rate curve, with the stress and shear rate increasing together. Upon subsequently decreasing the imposed stress, the upward and downward flow curves overlapped, suggesting that a steady state branch, independent of the flow history, was present in the flow curve. Further decreasing the imposed stress led to the continuous decrease of the shear rate, but a dramatic decrease of shear rate (one order of magnitude) was not observed here. This effect was mainly due to the microstructure in the suspensions becoming destructured rather rapidly, while the rebuilding process occurred much more slowly during a down-stress ramp.

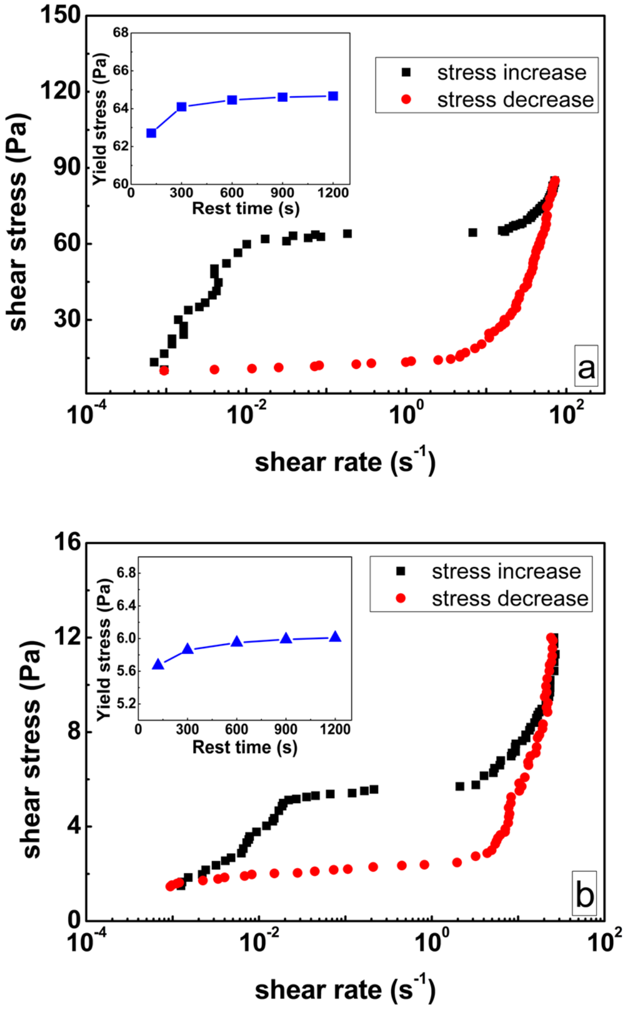

**Fig. 1.** Hysteresis loops for hardwood (a) and softwood (b) pulp suspensions at the same crowding number of 120. Samples were pre-sheared at 200$s^{-1}$ for 3 min followed by a 10-min rest. The inset shows the evolution of critical shear stress with the time of rest.

The area enclosed by the loop for softwood was much smaller than for hardwood under the same crowding number, meaning that the hardwood pulp suspensions exhibited a more severe thixotropic behaviour. The critical shear stress for softwood (approximately 5.9 Pa) was also much lower (Fig. 1), indicating that the interactions between hardwood fibres were stronger at a given crowding number. Moreover, the shapes of the hysteresis curves for hardwood and softwood were analogous, suggesting that the chemical pulp suspension systems exhibited strong mechanical similarities.

When the effect of rest time on the hysteresis experiments was investigated, samples were pre-sheared at 200 $s^{-1}$ for 3 min and then allowed to rest for 2, 5, 10, 15, or

20 min prior to a shear stress ramp. The critical shear stress values obtained from the hysteresis profiles were plotted against the time of rest (Fig. 1, insets). The static yield stress for both hardwood and softwood pulp suspensions increased non-linearly with increases in the time of rest, and only a slight increase in yield stress was observed when the rest time was above 300 s. Given this result, it can be argued that at rest these materials restructured themselves, leading to a more rigid "structure". However, after a given period of time, the microstructures in the suspensions evolved more slowly. The apparent increase in yield stress over the time of rest agrees with previous observations on the yield stress of thixotropic fluids (Alderman *et al.* 1991; Coussot *et al.* 2002a; Nguyen *et al.* 2006), and it explains some of the difficulties in measuring yield stress.

To study the influence of crowding number on the yielding properties of chemical pulp suspensions, the critical shear stress values of chemical pulp suspensions at five different crowding numbers were obtained from hysteresis experiments (Fig. 2). A higher crowding number resulted in higher yield stress. Compared with softwood pulp suspensions, hardwood pulp suspensions had much higher yield stress at each crowding number. This effect was attributed to the higher average fibre length of softwood, resulting in less fibres and fibre contact points under a given crowding number and consequently, decreased bonding in the fibre network. Equation 2 was used to correlate yield stress with crowding number (Table 2). The value of $\beta$ was about 2.06, which was different than the $\beta$ value of 2.3 reported by Martinez *et al.* (2003). This difference was mainly due to the different pulp species and experimental methods used.

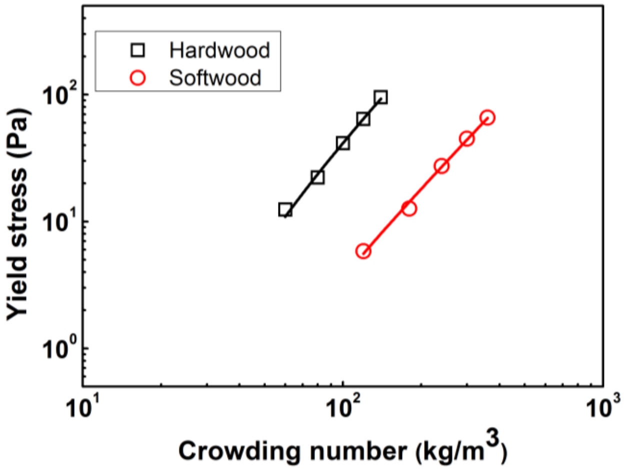

**Fig. 2.** Yield stress as a function of crowding number, as deduced from the hysteresis loops. Samples were pre-sheared at 200 $s^{-1}$ for 3 min followed by a10-min rest.

**Table 2.** Summary of Fitted Coefficients to Eq. 2 for All Tests

| Samples | $\tau_y= \alpha$ (N - $N_{gel})^{\beta}$ | | |
|---|---|---|---|
| | $\alpha$ | $\beta$ | $R^2$ |
| Hardwood | $4.42\times10^{-3}$ | 2.064 | 0.995 |
| Softwood | $3.95\times10^{-4}$ | 2.057 | 0.993 |

## Creep Tests

To study in greater detail the flow of the suspension over the observed plateau in the flow curves plotted in Fig. 1, creep tests were performed with the following procedure. The samples were pre-sheared at a high imposed shear rate of 200 s$^{-1}$ for 3 min, followed by a 10-min rest. Starting from identical initial conditions, different levels of shear stress were imposed on the samples, and the resulting shear rate was measured as a function of time (Fig. 3). In Fig 3, at small values of shear stress, the shear rate decayed to a fixed value which is attributed to the limitation of the rheometer to measure small shear rates. In this case, the minimum shear rate indicates the solid-like behaviour of the suspensions.

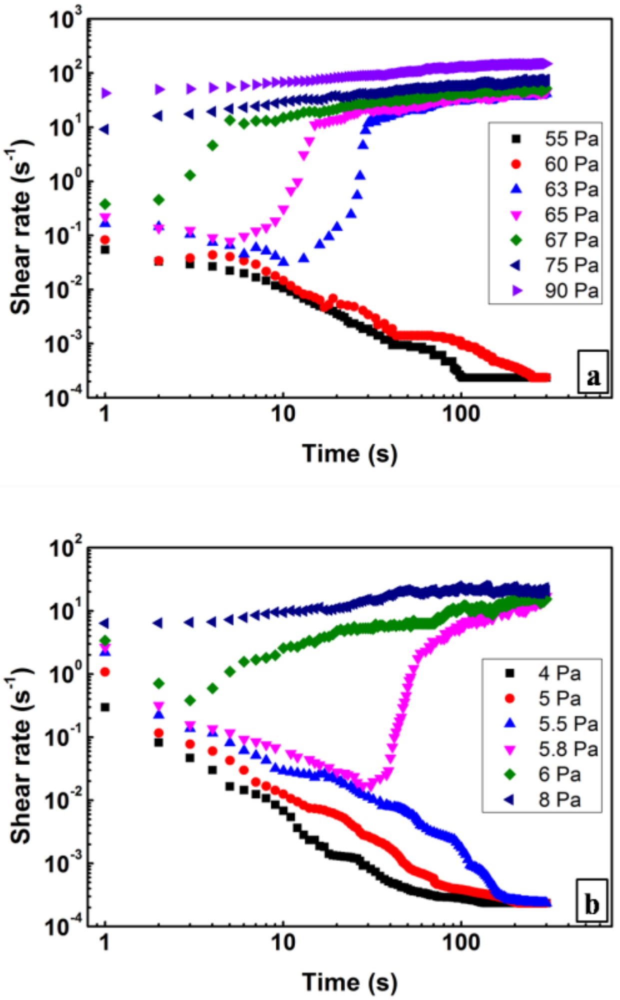

**Fig. 3.** Creep tests for hardwood (a) and softwood (b) pulp suspensions at the same crowding number of 120. Samples were pre-sheared at 200 s$^{-1}$ for 3 min followed by a 10-min rest.

Creep tests showed that both materials exhibited solid and liquid regimes. At low stress, the induced deformation tended to saturate (solid regime), whereas at high stress they tended to flow at a constant rate (liquid regime). For stress values smaller than the critical value ($\sigma_c \approx 63$ Pa), the resulting shear rate was so low that the build-up of a structure won over its destruction, and the material progressively stopped flowing. In contrast, for stress values above $\sigma_c$, destruction of the microstructure won out, and the flow accelerated over time until the shear rate reached a steady-state value. Notably, the transition between these two systems is discontinuous as a function of the imposed stress; this phenomenon is called viscosity bifurcation (Coussot *et al*. 2002a; Ovarlez *et al*. 2013). Moreover, it is significant that the critical stress values for both pulp suspensions obtained from creep measurements agreed reasonably well with the static yield stress values measured from hysteresis experiments.

In thixotropic samples, the time of rest prior to measurement is important, and the creep curves can become quite complex, especially around the yield stress (Uhlherr *et al*. 2005; Coussot *et al.* 2006; Mewis and Wagner 2009). Figure 4 illustrates the creep curves corresponding to various rest times for softwood pulp suspensions at a constant shear stress of 5.8 Pa. As the rest time increased, the strain levelled off initially, only to accelerate again after some time. In addition, the creep curves shifted downward, and the delay time increased, *i.e.*, the viscosity increased with rest time, presumably reflecting structure build-up. The same shift in the shear rate profiles was observed in hardwood pulp suspensions (data not shown).

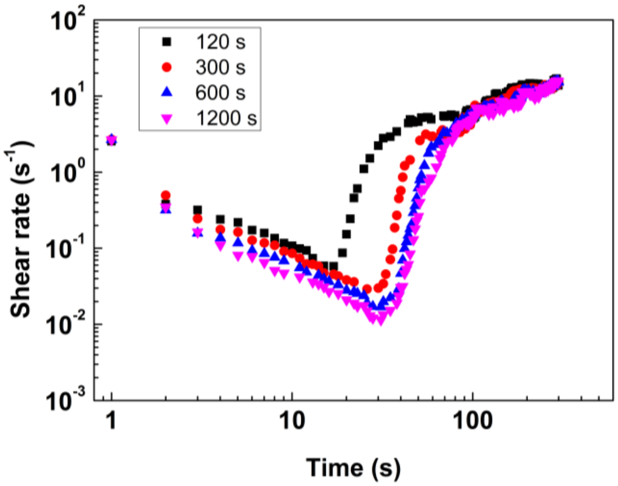

**Fig. 4.** Shear rate *vs*. time for softwood pulp suspensions at a crowding number of 120 and a constant stress of 5.8 Pa. The sample was pre-sheared at 200s$^{-1}$ for 3min followed by a 2-, 5-, 10-, or 20-min rest.

## Step-wise Experiments

Thixotropic behaviour is commonly studied by tracking the material responses resulting from step wise changes in shear rate or shear stress, as the coupled effects of time and shear history can be clearly separated in such experiments. Hardwood and softwood pulp suspensions at a crowding number of 120 were investigated with a sequence of shear rate steps that included an initial lower shear rate (60 s$^{-1}$) that was increased to an appropriate shear rate (80 s$^{-1}$, 100 s$^{-1}$, or 120 s$^{-1}$) and then reduced back to 60 s$^{-1}$. The resulting apparent viscosities ($\mu_{app}$) were measured as a function of time. An example of the shear rate steps together with the system response, *i.e.* the apparent viscosity of hardwood pulp suspension, are shown in Figs. 5(a) and (b), respectively.

The viscosity decreased in time at a given shear rate, and the suspension exhibited lower viscosity when subjected to a shear rate of 100 s$^{-1}$. From a structural point of view, the suspension became destructured under shear. However, when the applied shear rate was decreased from 100s$^{-1}$ to 60 s$^{-1}$, the apparent viscosity increased, indicating an increase in structure. However, the viscosity still decreased over time. This trend was the opposite of that observed by Derakhshandeh *et al.* (2012). Notably, there was a turning point in the viscosity curve when the suspension was sheared at a shear rate of 100 s$^{-1}$, mainly because of inertial effects induced by the measuring head of the rheometer (Barnes 1997).

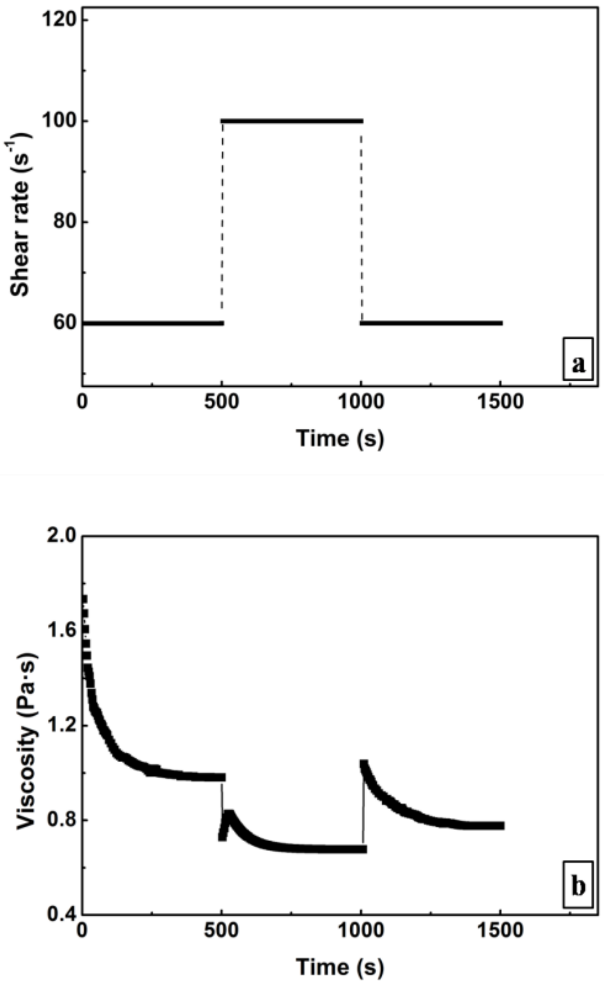

**Fig. 5.** Response of hardwood pulp fibre suspension at a crowding number of 120 subjected to a stepwise change in shear rate. (a) Stepwise changes in shear rate; (b) dynamic viscosity of the suspension. The sample was pre-sheared at 200 s$^{-1}$ for 3 min followed by a10-min rest.

A simple exponential model was used to quantify the thixotropic features (Eq. 4),

$$\mu_{app}(t) = a \cdot exp(-{}^{t}/_{\tau}) + \mu_{\infty} \quad (4)$$

where $\tau$ is the characteristic time (s) and $\mu_{\infty}$ is the viscosity after shearing for an infinite time (Pa·s). To derive the characteristic time $\tau$, a non-linear regression was used to analyze the experimental data points. The corresponding characteristic times ($\tau$) are summarized in Table 3 for samples at different initial and final flow conditions. For the first shear rate of $\dot{\gamma} = 60$ s$^{-1}$, the characteristic time constants were $\tau$=55.08 s and $\tau$=37.92 s for hardwood and softwood pulp suspensions, respectively. For the initial shear rate of $\dot{\gamma} = 60$ s$^{-1}$, the characteristic time ($\tau$) increased as the final shear rate increased. However, for the final shear rate of $\dot{\gamma} = 60$ s$^{-1}$, $\tau$ decreased with increases in the initial shear rate. After a stepwise change in shear rate, $\tau$ became larger when the samples were sheared at the same shear rate of 60 s$^{-1}$. This result indicates that the structure of pulp suspensions is governed by shear-induced collisions. Moreover, the characteristic time constants for hardwood suspensions were larger than those for softwood pulp suspensions at a given shear rate, which was due to the properties of the fibres themselves.

**Table 3.** Thixotropic Time Constants for Hardwood and Softwood Pulp Suspensions

| Samples | Initial Shear Rate, $\dot{\gamma}_i$=60 s$^{-1}$ | | Final Shear Rate, $\dot{\gamma}_e$= 60 s$^{-1}$ | |
|---|---|---|---|---|
| | Final Shear Rate, $\dot{\gamma}_e$ (s$^{-1}$) | Characteristic Time, $\tau$(s) | Initial Shear Rate, $\dot{\gamma}_i$(s$^{-1}$) | Characteristic Time, $\tau$ (s) |
| Hardwood | 80 | 22.98 | 80 | 159.82 |
| | 100 | 62.93 | 100 | 101.45 |
| | 120 | 108.19 | 120 | 75.21 |
| Softwood | 80 | 11.41 | 80 | 140.84 |
| | 100 | 52.78 | 100 | 87.09 |
| | 120 | 56.25 | 120 | 74.97 |

## CONCLUSIONS

1. Chemical pulp fibre suspensions are thixotropic materials that exhibit yield phenomena, and there is a remarkable similarity in the observed flow behaviour between softwood and hardwood pulp suspensions.
2. Pulp suspensions exhibited a plateau in their flow curves where a slight change in the shear stress generated a jump in the corresponding shear rate. A viscosity bifurcation was observed, where the suspensions stopped flowing to reach a saturated state below a critical stress and reached a steady state of flow at higher values.
3. The rest time prior to the experiment influenced the creep curves. As the rest time increased, the creep curves shifted downward, and the delay time increased.
4. A simple exponential model can be used to quantify the thixotropic response of pulp to stepwise changes in the shear rate and the characteristic time that reflects the changes in pulp suspension microstructure.

## ACKNOWLEDGMENTS

This research was supported by grants from the Jiangsu Provincial Graduate Education Innovation Project (KYLX15_0933) and the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD).

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Article submitted: October 14, 2015; Peer review completed: January 30, 2016; Revised version received and accepted: February 17, 2016; Published: February 22, 2016.
DOI: 10.15376/biores.11.2.3481-3493