

The Effect of Consistency and Freeness on the Yield Stress of Chemical Pulp Fibre Suspensions

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To study the influence of mechanical treatments on the yield stress of chemical pulp suspensions, a traditional rheometer, coupled with local velocity measurements (ultrasonic Doppler velocimetry), was used to measure the yield stress of two types of commercial chemical pulp suspensions with different freeness values at mass concentrations (consistencies) ranging from 0.5 to 1.5%. Over the range of consistencies tested, the yield stress was found to depend on the consistency through a power law relationship for all tested samples. Moreover, the results showed that as the freeness decreased, the yield stress of hardwood suspensions increased to a maximum value then decreased. This variation in yield stress was also observed in softwood suspensions with mass concentrations above 1%. However, when the consistency was lower than 0.75%, the yield stress of softwood suspensions increased with decreasing freeness. This behaviour can be understood based on the underlying fibre properties of fibrillation, curl, and stiffness, suggesting that fibre morphology plays a significant role on the yield stress of pulp suspensions over the concentration range studied.

Keywords: Yield stress; Rheology; Pulp fibre suspensions; Freeness; PFI mill

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INTRODUCTION

The pulp and paper industry comprises a significant part of the global economy, and paper products play an important role in a number of aspects of society, including packaging, hygiene, and communication (Bousfield 2008). Most unit operations in this industry deal with the flow of various pulp fibre suspensions. To achieve optimal functionality of pulp and paper manufacturing and operations, knowledge of pulp suspension rheology is of great importance.

The characteristic rheology of a pulp suspension is dependent on many factors, including pulp concentration, fibre properties, fibre-fibre interaction, the presence of other components in the suspensions, and external factors such as temperature, shear forces, and shear history (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. Increasing the consistency (mass concentration based on filterable solids) of the fibre suspension increases the number of fibre-fibre interactions at a typical moment in time, forming a network structure throughout the suspension. The degree of interaction between 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 one fibre as it rotates about its centre. It can be described as follows,

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

where L is fibre length (m), d is fibre diameter (m), δ is fibre coarseness (kg/m), and $C_{v,m}$ is volume/mass concentration (%) of the suspension.

In pioneering work, Mason (1950) identified $N = 1$ as a “critical concentration” at which collisions first occur among fibres in the suspension. Kerekes and Schell (1992) showed that at $N = 60$ the number of contact points per fibre is approximately three, which is enough for a coherent fibre network to be established. In subsequent work, Martinez *et al.* (2003) identified another critical crowding number, N_{gel} , $N_{gel} \approx 16$, calling this a “gel crowding number”. Fibre suspensions below this value 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 cause motion throughout the suspensions, the shear forces imposed must exceed this network strength. This network strength is known as the yield stress of the pulp suspension (Derakhshandeh *et al.* 2010).

The yield stress is one of the most important rheological properties of pulp suspensions, both to designing equipment and to effective operations within the pulp and paper industry (Moller and Elmqvist 1980; Ein-Mozaffari *et al.* 2005; Olson *et al.* 2009). Applications of yield stress have been presented in relevant fields such as pulp pipe flow (Moller and Elmqvist 1980), paper sheet forming (Martinez *et al.* 2003), pulp mixing operations (Ein-Mozaffari *et al.* 2005) as well as pulp fluidization (Bennington and Kerekes 1996). Experimental studies of the yield stress on fibre suspensions have been done before (Gullichsen and Harkonen 1981; Kerekes *et al.* 1985; Bennington *et al.* 1990). Despite differences among some of the results of these studies, they all found the yield stress to be dependent on mass concentration according to the formula,

$$\tau_y = aC_m^b \quad (2)$$

where τ_y is the yield stress (Pa), a and b are constants specific to the fibre type, and C_m is the mass concentration of the pulp suspension (%). Values of a and b as measured in previous studies were summarised by Kerekes *et al.* (1985). In a subsequent work, Bennington *et al.* (1990) determined the yield stresses of commercial wood pulp suspensions using vaned rotors in housings with baffles and reported ranges of a and b from 1.18 to 24.5 and 1.25 to 3.02, respectively. In order to study the influence of fibre properties on the yield stress of pulp suspensions, Dalpke and Kerekes (2005) measured the yield stress for a range of pulps of differing species pulped by differing methods, and found fibre length to be very important with longer fibres exhibiting greater yield stress. The same results were reported by Ventura *et al.* (2007), who tried to establish a function to correlate yield stress with fibre length, concentration, and temperature.

As the consistency increases, the air content in the pulp fibre suspensions increases and gas congregates around the rotor of the rheometer, impeding momentum transfer into the suspension. Bennington *et al.* (1995) and Pettersson and Rasmuson (2004) studied the effects of air content on the yield stress of pulp suspensions at high mass concentrations and found that the yield stress was dependent on the fibre concentration and air content as follows,

$$\tau_y = aC_m^b(1 - \varphi)^c \quad (3)$$

where φ is the volume fraction of air in the suspension (%) and a , b , and c are parameters related to the fibre properties.

The complexities described above complicate measurement of pulp suspension rheology, and the yield stress determinations have been divided into two categories based on the measuring technique used: quasi-static measurements, where the yield stress has been determined in conventional rheometer-like devices in a near-static state; and dynamic surface strength measurements, where the yield stress is inferred from the disruption of flocs or fibres in flowing suspensions (Bennington *et al.* 1995; Derakhshandeh *et al.* 2010; Bonn *et al.* 2015). For quasi-static measurements, fibres and flocs are large compared with the geometry dimensions used in rheological devices and therefore fibre rotation is limited. Another behavior of fibre suspensions in the flow field is fibres migrating from the solid boundaries and creating a depletion layer that complicates rheological measurements (Nguyen and Boger 1985; Swerin *et al.* 1992). Due to these complications, various measuring devices have been employed to overcome issues of gap size and wall slip to measure the yield stress of pulp suspensions. One of the most common approaches is by using vaned-geometry devices having large measurement gaps, which can alleviate wall slip and fibre clogging issues (Gullichsen and Harkonen 1981; Bennington *et al.* 1990; Cullen *et al.* 2003). However, assumptions should be made when using vaned-geometry devices, *i.e.*, one must specify that the yield surface is cylindrical and defined by the outer diameter of the blades, with a constant shear distribution on the shearing surface (Ein-Mozaffari *et al.* 2005). Another approach to minimize depletion layer is to make use of rough surfaces in the rheometer-like devices. Thus, Swerin *et al.* (1992) and Damani *et al.* (1993) measured the yield stress of pulp suspensions in their parallel-plate rheometer having roughened walls of 105 to 205 μm . More recently, Derakhshandeh *et al.* (2010) measured the yield stress of pulp suspensions using a conventional rheometer coupled with local velocity measurements (ultrasonic Doppler velocimetry). The measured yield stress was in agreement with the results obtained using the linear shear stress ramp method (the variation of instantaneous viscosity as a function of shear stress can be obtained; the yield stress is the value at which the instantaneous viscosity exhibits a maximum), thereby verifying this simpler technique as reliable.

Before papermaking, pulp fibres are usually mechanically treated, *via* mechanical refining, to gradually change the fibre properties. The purpose of these treatments is to modify the fibre properties to obtain desirable paper properties and good paper machine runnability (Miles and Karnis 1991; Kerekes 2005). Mechanical treatment may damage the structure of the cell wall and change geometric properties, which include: fibre cutting and shorting, internal delamination, external fibrillation, fibre curl, and straightening. Following this modification, fibres enlarge their surface area (due to increased fibrillation and swelling ability) and the fibre flexibility to enhance the bonding (Page 1989). The refining also changes the pulp drainability, usually due to the production of fines. The freeness value, which characterizes the change in the drainage rate of pulps, is widely used to represent the change in fibre properties during beating and refining. It has been shown to be related to the surface conditions and swelling of the fibres and can suitably control fibre properties by being able to select the most appropriate level of refining energy necessary for the required grade of paper.

Few reports on the influence of mechanical treatment on the yield stress of chemical pulp suspensions at low mass concentration have been published. The aim of this work was to use ultrasonic Doppler velocimetry (UDV), coupled with a conventional rheometer, to measure the yield stress of chemical pulp suspensions with different freeness values at mass concentrations ranging from 0.5 to 1.5%. The results obtained may be valuable with respect to the application of chemical pulp suspensions in relevant fields of industry.

EXPERIMENTAL

Materials

Two commercial, dried, bleached kraft pulps, both hardwood and softwood, were obtained. The softwood pulp, containing 100% spruce pine fir, was obtained from Canfor Pulp Product, Inc. (Prince George, BC, Canada). The hardwood pulp was produced from eucalyptus and was obtained from CMPC, Inc. (Santa Fe, Chile). Both raw materials were swollen in distilled water for 24 h and were dispersed using a blender (TMI, Montreal, QC) for 10 min to achieve a homogeneous suspension. The pulps were refined in a PFI mill at 10% mass concentration according to TAPPI standard T481. Samples were taken after 0, 4000, 8000, and 12,000 revolutions. Because air bubbles could easily be trapped in concentrated suspensions and induce unwanted variance into the results, pulp suspensions diluted with deionized water at low concentrations of 0.5, 0.75, 1, 1.25, and 1.5 wt.% were prepared for further rheological measurements.

Characterisation

Changes in fibre morphology during the mechanical treatment of the pulp were studied using a light microscope (Microflex HFX-II, Nikon; Japan) at 400 times magnification. A variety of fibre properties, such as the fibre length, percent fines, and curl index, were measured using a Fibre Quality Analyzer (OpTest Equipment Inc., Hawkesbury, Canada). The freeness of the samples was measured using TAPPI standard T227 om-99 (1999).

Methods

A Haake RV12 rheometer (ThermoFisher Scientific Inc., Waltham, MA), coupled with a pulsed ultrasonic Doppler velocimetry apparatus (Model DOP3000, Switzerland), was used to measure the yield stress of the suspensions. In using UDV to obtain the local velocity profile across the large gap, the vane creates a sheared (yielded) zone within which the material flows. However, when the shear stress falls below the yield stress value, the fluid becomes stationary (un-yielded zone) and the velocity is approximately zero between R_1 and R_2 (Fig. 1), where R_1 is the vane radius (m) and R_2 is the cup radius (m).

For a vane of height h (m) and radius R_1 (m) rotating at a constant angular velocity, the measured torque, M (N·m), is related to the shear stress at the vane surface, τ_T (Pa), according to the formula:

$$\tau_T = \frac{M}{2\pi R_1^2 h} \quad (4)$$

With knowledge of the yield radius R_y (m) (obtained using UDV measurements) and the steady-state shear stress at the vane surface τ_T (obtained using the torque reading

from the rheometer), the yield stress τ_y can be calculated from Eq.5:

$$\tau_y = \tau_T \left(\frac{R_1}{R_y} \right)^2 \quad (5)$$

Further details about UDV measurements are described by Derakhshandeh *et al.* (2010).

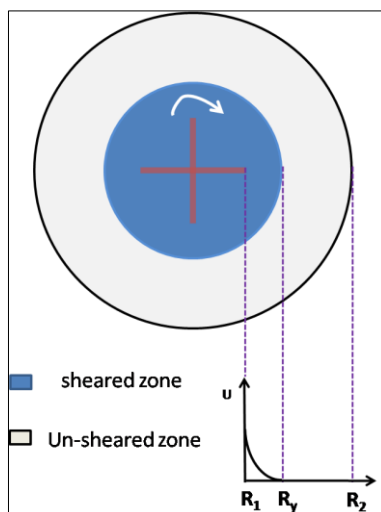


Fig. 1. Schematic of the vane in a large cup geometry and its characteristic dimensions (Derakhshandeh *et al.* 2010).

In the present work, a four-bladed vane 38.5 mm in height and 25 mm in diameter was placed in a transparent, cylindrical cup with an internal diameter of 100 mm and height of 64 mm, resulting in a gap size of 37.5 mm. All fibre suspensions were pre-sheared at 512 rpm for 5 min and allowed a relaxation period of 5 min. The optimum relaxation time was examined by checking the dependency of viscosity on the shear rate, which ensures that the pre-shearing conditions do not influence our results. Following pre-shearing and relaxation, the torque and angular velocity data were recorded after 3 min at each applied shear rate. Within the measurements above, the velocity profiles of hardwood and softwood suspensions were recorded using UDV measurements at 64 and 128 rpm, respectively, to calculate the yield stress. All measurements were conducted at 26 °C. For pulp suspensions of high consistency the mean velocity decreases from a maximum at the tip of the vane and suddenly drops to zero at the yielding radius, resulting in a discontinuous jump of the velocity during the transition from the yielded to the un-yielded region, which leads to a shear banding phenomenon (Derakhshandeh *et al.* 2010; Divoux *et al.* 2010). Since the phenomenon of shear banding is more dominant at high mass concentrations, the effect of shear banding on the discontinuous velocity profile becomes negligible in our study.

RESULTS AND DISCUSSION

Mechanical Fibrillation

The fibrillation methods used yielded samples with different properties, as summarised in Table 1. The fibre curl index is defined as the ratio of the actual fibre length

to the distance between the two fibre ends minus one. It indicates the continuous curvature of the fibres greater than 0.5 mm in length and within the selected range limits. As shown in Table 1, the freeness of the pulps decreased with increasing refiner revolutions. Both the fibre length and fines content of the samples did not change significantly during the refining process. This is because the PFI mill refines at low intensity, limiting fibre cutting and causing fibrillation (Kerekes 2005). Figure 2 shows macroscopic images of hardwood and softwood cellulose fibres exposed to mechanical treatments in a PFI mill before and after 4000, 8000, and 12,000 revolutions. The images show that the fibres became more fibrillated with increasing revolutions. Furthermore, hardwood cellulose fibres were not fibrillated to the same extent as softwood fibres during refining. Table 1 also shows that softwood samples had a larger curl index than hardwood samples and that the curl index gradually changed with refining actions, as indicated in Fig. 3.

Table 1. Properties of Pulp Samples Refined to Various Degrees

| Samples | Hardwood | | | | Softwood | | | |
|---------------------------|----------|-------|-------|-------|----------|-------|-------|-------|
| | H1 | H2 | H3 | H4 | S1 | S2 | S3 | S4 |
| Refiner Revolutions | 0 | 4000 | 8000 | 12000 | 0 | 4000 | 8000 | 12000 |
| Freeness (mL) | 420 | 330 | 280 | 220 | 670 | 580 | 480 | 430 |
| Average Fibre Length (mm) | 0.723 | 0.742 | 0.737 | 0.749 | 2.264 | 2.234 | 2.320 | 2.335 |
| Average Fibre Width (um) | 17.2 | 18.0 | 18.1 | 17.6 | 28.2 | 28.8 | 28.8 | 28.6 |
| Mean Curl Index (%) | 14.2 | 11.5 | 14.0 | 9.1 | 21.4 | 23.8 | 24.1 | 19.5 |
| Fines Content (%) | 14.36 | 13.68 | 14.15 | 13.74 | 31.8 | 34.29 | 32.32 | 29.54 |

Yield Stress

Yield stress was measured for hardwood and softwood fibre suspensions at mass concentrations ranging from 0.5 to 1.5%. Measurements were performed four times to check the reproducibility and to minimize experimental errors. Figures 4 and 5 present the resulting curves for hardwood and softwood fibre suspensions, respectively. Figures 4 and 5 show that the yield stress of 0.5 to 1.5 wt.%, non-refined, bleached hardwood kraft pulp ranged from 0.13 to 3.71 Pa and that of softwood pulp ranged from 0.85 to 8.14 Pa. Moreover, the yield stresses for both hardwood and softwood pulp suspensions with mechanical treatment were higher than without refining. Compared to hardwood pulp suspensions, the yield stress of softwood pulp suspensions was much higher at a certain concentration, indicating fibre-fibre interaction and that the bonding ability of softwood fibres is much stronger mainly because of the higher average fibre length, which facilitates the creation of more contact points within fibres.

Figure 4a illustrates that over the range of concentrations tested, the yield stress of hardwood fibre suspensions increased non-linearly with consistency. This was also true for softwood fibre suspensions as shown in Fig. 5a. Furthermore, the shapes of the graphs for hardwood and softwood fibre suspensions in Figs. 4a and 5a were similar, and the yield stress was found to be a power law function of the consistency, as shown in Eq. 2. To determine the values of a and b , non-linear regression was used to analyse the experimental data points and the results are summarised in Table 2. The values of a and b ranged from 1.26 to 5.29 and 1.83 to 3.42, respectively, within the ranges obtained by Bennington *et al.* (1990). Moreover, the correlation coefficients (R^2) were acceptable for all rheological parameters, indicating that mechanical treatment of the pulp fibres had little influence on the relationship between yield stress and mass concentration. However, the yield stress values of 1.5% pulp suspensions do not fall on the fitted lines within the

error bars. The reason for this is that wall slip and shear banding can affect the pulp flow at low shear rate, the yield stress may then present a slight deviation from actual values, as classically observed in emulsions, microgels or colloidal gels (Meeker *et al.* 2004; Bonn *et al.* 2015). Given this, with increasing concentration, the mechanical treatments on chemical pulp fibres had little effect on the mechanics of fibre network formation.

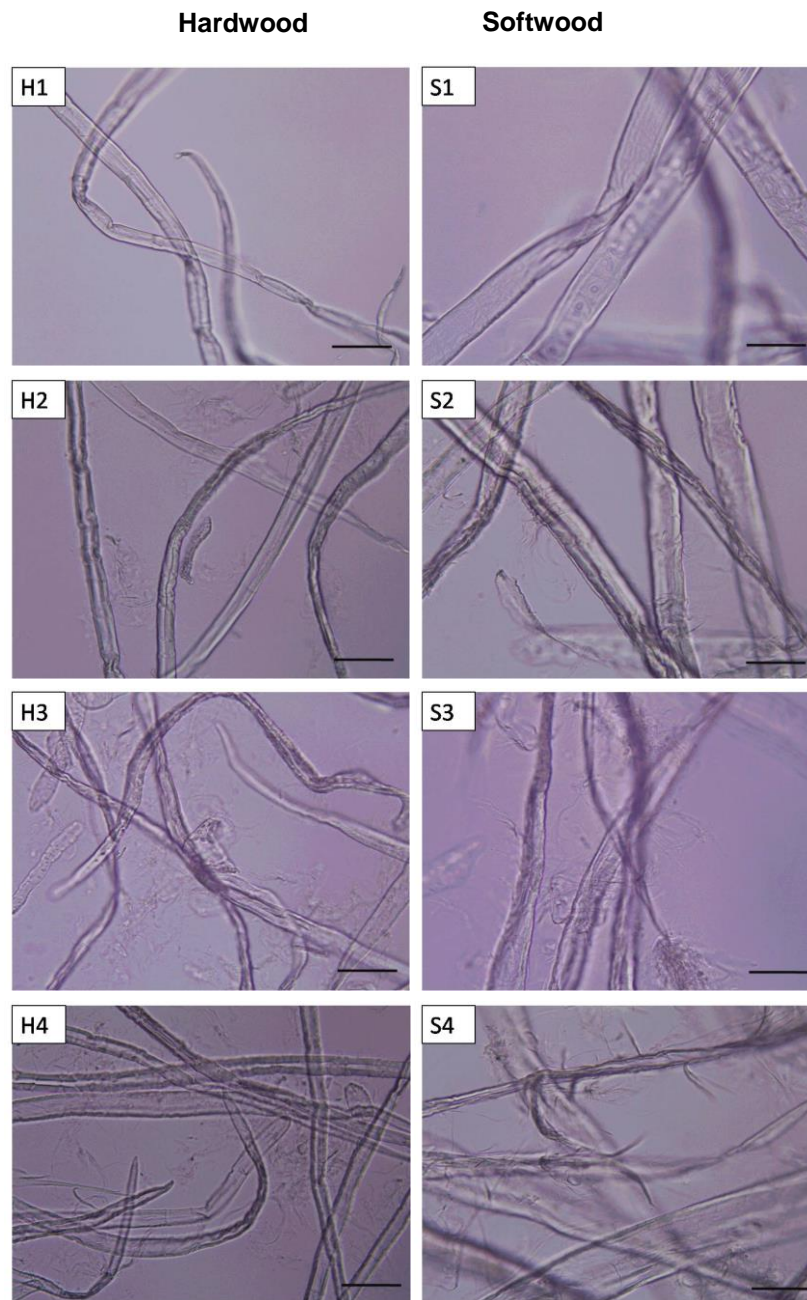


Fig. 2. Effects of mechanical refining on hardwood and softwood cellulose fibres through the PFI mill. Hardwood H1, H2, H3, and H4 at 0, 4000, 8000, and 12,000 revolutions, respectively; Softwood S1, S2, S3, and S4 at 0, 4000, 8000, and 12,000 revolutions, respectively. Scale bar 50 μ m

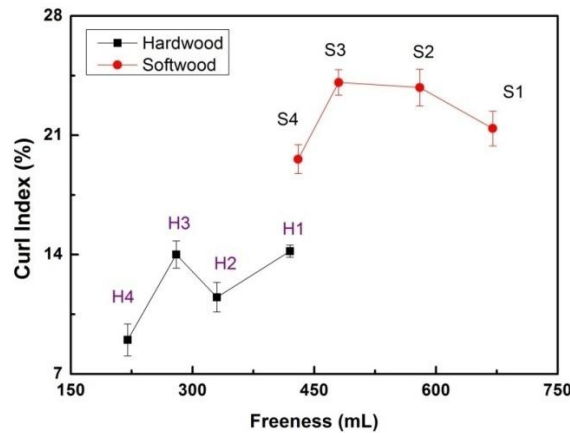


Fig. 3. Curl index versus freeness value for hardwood and softwood fibre suspensions. Data provided as the average \pm standard deviation

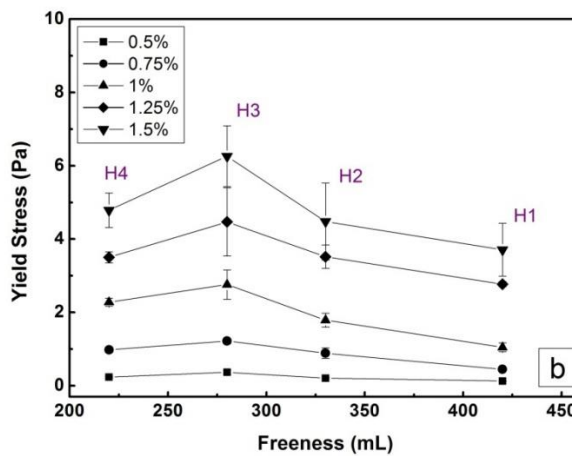
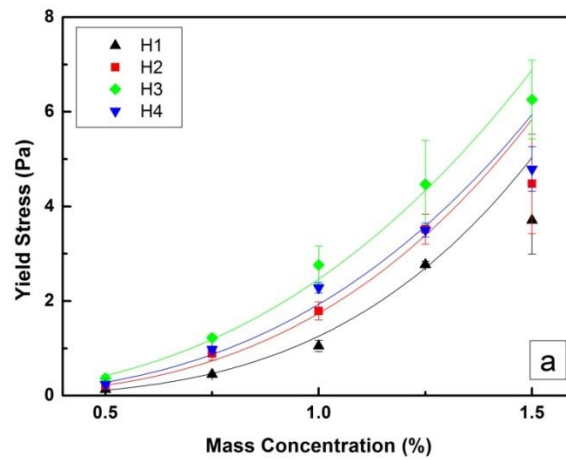


Fig. 4. Yield stress values of hardwood fibre suspensions as a function of (a) mass concentration and (b) freeness obtained using ultrasonic Doppler velocimetry at 26 °C at a constant rotational velocity of 64 rpm. The solid lines in figure (a) are power-law fits $\tau_y = aC_m^b$ with constants listed in Table 2. Data provided as the average \pm standard deviation

As shown in Fig. 4b, the yield stress of hardwood suspensions increased to a maximum value then decreased, with decreasing freeness values over the range of concentrations tested. This variation in yield stress was observed in softwood suspensions with mass concentrations above 1%, as shown in Fig. 5b. However, when the concentration was lower than 0.75%, the yield stress of softwood suspensions increased with decreasing freeness. Because the fibre length and fines content varied only slightly during the refining process, both fibre fibrillation and curl are important since they influenced the results.

As shown in Fig. 4b, for samples H1 and H3, with a freeness of 420 and 280 mL, respectively, the curl indices were nearly the same, as illustrated in Fig. 3. However, the yield stress of H3 was much higher than that of H1. This is because H3 fibres were more fibrillated and quite rough with fibrils sticking out along the fibre surface, as shown in Fig. 2, which enlarged the coefficient of friction and contact area between fibres, allowing them to form networks with higher strength.

The same results were also shown in Fig. 5b when comparing samples S2 and S3. For sample H4, when compared to sample H3, the decrease in yield stress was likely due to the decrease of the curvature of H4 fibres, as illustrated in Fig. 3, which may not facilitate the creation of fibre hooking and inhibits fibre-fibre bond creation. In addition, increased refining process might decrease the stiffness of H4 fibres, and thus able to decrease the strength of fibre network (Huber *et al.* 2008).

Although the fibres are also more fibrillated in H4 and the decreased curl could increase the contact points between the fibres since they are in a more expended form, the decrease in curl index and fibre stiffness played a more important role in reducing the yield stress. This explanation is also suitable for samples S3 and S4 with concentrations above 1%. However, for samples H1 and H2 and S3 and S4 with concentrations below 0.75%, the influence of fibre fibrillation, fibre stiffness and fibre curl on the yield stress were reversed.

According to the results above, as the freeness decreased, the variation in yield stress was largely dependent on fibre morphology and mass concentration, suggesting that freeness alone did not account for the yield stress over the concentration range studied.

Table 2. Summary of Fitted Constants to the Power Law Model for All Tests

| Samples | Hardwood | | | | Softwood | | | |
|----------------|----------|-------|-------|-------|----------|-------|-------|-------|
| | H1 | H2 | H3 | H4 | S1 | S2 | S3 | S4 |
| Freeness (mL) | 420 | 330 | 280 | 220 | 670 | 580 | 480 | 430 |
| a (Pa) | 1.26 | 1.74 | 2.46 | 1.93 | 3.03 | 3.07 | 5.04 | 5.29 |
| b | 3.42 | 2.99 | 2.53 | 2.77 | 1.83 | 2.01 | 2.18 | 1.85 |
| R ² | 0.992 | 0.979 | 0.983 | 0.967 | 0.977 | 0.997 | 0.905 | 0.967 |

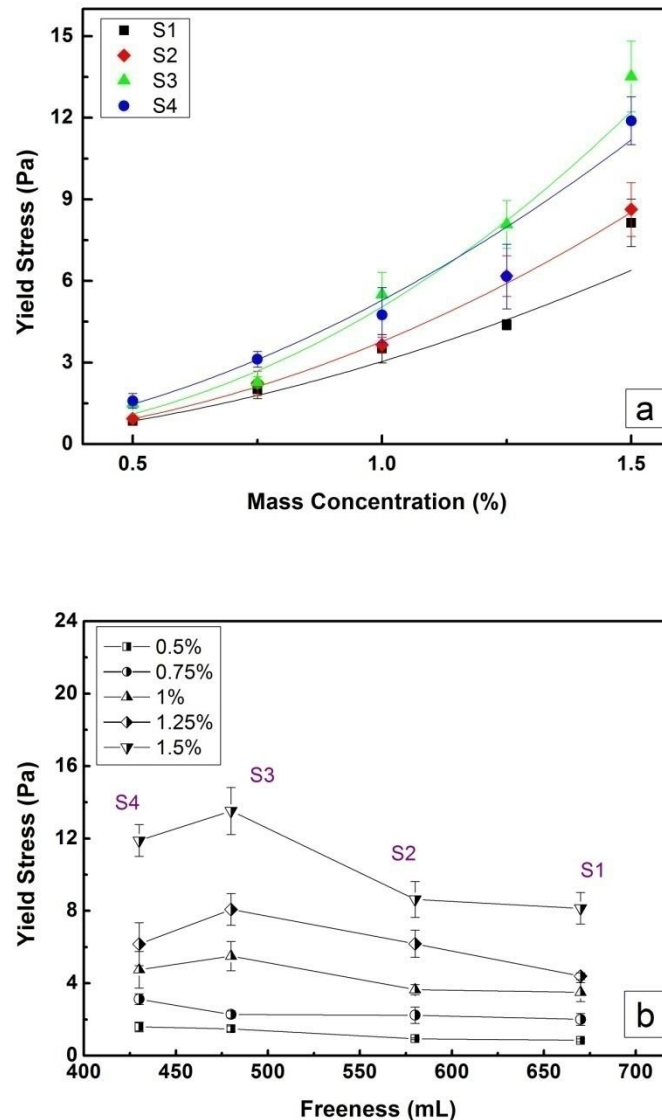


Fig. 5. The yield stress values of softwood fibre suspensions as a function of (a) mass concentration and (b) freeness obtained using ultrasonic Doppler velocimetry at 26 °C at a constant rotational velocity of 128 rpm. The solid lines in figure (a) are power-law fits $\tau_y = aC_m^b$ with constants listed in Table 2. Data provided as the average \pm standard deviation.

CONCLUSIONS

1. Under steady-state shear conditions over the range of consistencies tested, the yield stress of all suspensions increased non-linearly with increasing consistency.
2. The yield stress was found to depend on the consistency *via* a power law relationship.
3. The yield stress of fibre suspensions is strongly affected by mechanical treatments. For hardwood fibre suspensions, as the freeness decreases, the yield stress increased to a maximum value then decreased, this variation in yield stress was also observed

in softwood suspensions with mass concentrations above 1%. However, when the concentration was lower than 0.75%, the yield stress of softwood suspensions increased with decreasing freeness. The increase of yield stress is mainly due to the increased fibrillation, curl, and surface area of fibres, which enlarged the coefficient of friction and bonding ability of fibres. The subsequent decrease of yield stress with further refining is mainly due to the decrease in curl index and fibre stiffness, which may not facilitate the creation of fibre hooking and inhibits fibre-fibre bond creation. The results show that the variation of yield stress is largely dependent on the fibre morphology over the concentration range studied.

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