Rheology of pulp suspensions of bleached sugarcane bagasse: Effect of consistency and temperature

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ABSTRACT: Rheological properties, such as yield stress and apparent viscosity, of pulp suspensions of bleached sugarcane bagasse were studied in a stress-shear rate controlled rheometer using concentric cylinders geometry. Results were statistically analyzed and presented as a function of the suspension consistency (0.5% ≤ C_m ≤ 4.0%) and temperature (20°C, 40°C, and 60°C). The yield stress was influenced by the consistency and temperature. The apparent viscosity was influenced only by the consistency. A power law model was fitted to the experimental results of yield stress. In flow tests, all the suspensions showed shear-thinning behavior, which was in agreement with the Carreau-Yasuda model.

Application: For optimum design and operation of process equipment, mills should consider the effect of rheological properties of pulp suspensions on energy consumption for transportation.

Pulp suspensions consist of wood or other lignocellulosic fibers, broken down by physical or chemical processes, dispersed in water [1]. These fibers are basically hollow tubes with typical mean length of 1–3 mm and diameter of 15–30 µm [2]. Many processes in the paper industry deal with the flow of pulp at different concentrations and temperatures. Understanding the changes in the flow properties of these suspensions due to changes in consistency and temperature are important for the optimal performance of the unit operations. One aspect that has received relatively little attention is the development of models to predict the rheological behavior of bleached sugarcane bagasse fibers suspensions. Cellulosic fiber suspensions exhibit complex rheological behavior due to the presence of flexible and thin fibers. These fibers form networks that tend to flocculate and help to transmit stresses, resulting in non-Newtonian behavior. Thus, fiber suspensions may show a variety of rheological phenomena, such as yield stress, shear thinning, shear thickening, thixotropy, extensional effects, and viscoelasticity [2,3].

Yield stress is one of the most important rheological properties for the design of process equipment in the paper industry. Bennington et al. [4] determined the yield stress for commercial wood pulp suspensions of low and medium consistency. They found good correlation using equations of the form $\tau_y = aC_v^b$, where $b \approx 3$, in agreement with the prediction of theoretical models. Later, Dalpke and Kerekes [5] determined that the yield stress of pulps of different species increased with increasing consistency and fiber length. They also determined that the empirical yield stress estimations were limited by the type of pulp. Because the reported values for the yield stress vary based on the measurement technique, it is appropriate to speak of apparent yield stress. Derakhshandeh et al. [6] used an ultrasonic Doppler velocimetry (UDV) coupled to a rate-controlled rheometer to provide a reliable method to measure yield stress. It was found that the results were consistent with those obtained from the linear shear stress ramp method.

Once yield stress is overcome, fiber suspensions flow at a strain rate determined by the applied stress and suspension viscosity. However, defining a meaningful viscosity for fiber suspensions is complex. The suspension cannot be considered as a continuum because fibers and flocs may be large in comparison to the dimensions of the flow field [2,3]. Silveira et al. [7] and Ferreira et al. [8] measured the apparent viscosity of aqueous suspensions of cellulose fibers from eucalyptus and pine bleached kraft pulps as a function of consistency, fiber length, and temperature. They determined that the suspensions exhibited a shear-thinning behavior. The apparent viscosity increased with increasing consistency and fiber length, but decreased with increasing temperature. Chen et al. [9,10] performed measurements of shear stress of hardwood bleached kraft pulp over a 0.1–100 s^{-1} range of shear rates. They observed Newtonian behavior at low shear rates, unstable behavior at medium shear rates due to formation of flocs, and Newtonian behavior once shear rates were high enough to disperse the flocs. Furthermore, they also noticed that longer fibers increased the flocculation tendency, as compared to shorter fibers.

Derakhshandeh et al. [11] found that pulp suspensions exhibited shear-thinning behavior up to a critical shear rate value after which the suspension exhibited Newtonian behavior.

Suspension viscosity increased with increasing consistency, fiber length, and pH. In the Newtonian regime at high shear rates, the viscosity was found to vary with the consistency to the third power. Their study was done on various commercial pulp fiber suspensions at fiber mass concentrations ranging from 1%–5% (by weight) using conventional rheometry, coupled with UDV. Chaussy et al. [12] characterized the viscous behavior of bleached softwood fibers by considering a semi-industrial disk refiner as a parallel plate rheometer. The rheological behavior of fiber suspension was quantified and modeled using a modified Carreau-Yasuda equation. Their results indicated a Newtonian behavior or a combination of a Newtonian behavior followed by a shear-thinning behavior after a limiting shear rate. Finally, reviews on the rheology and flow properties of pulp suspensions have been reported by Kerekes [3], Cui [13], and Derakhshandeh et al. [2].

In this paper, the experimental results are presented on the rheology of pulp suspensions of bleached sugarcane bagasse at different fiber mass concentrations and temperatures. The effect of consistency and temperature on yield stress and apparent viscosity of the suspension were also studied using a stress-shear rate controlled rheometer, implementing a vane rotor in a large-cup geometry. Mathematical models were proposed to correlate yield stress with consistency and apparent viscosity with shear rate.

MATERIALS AND METHODS

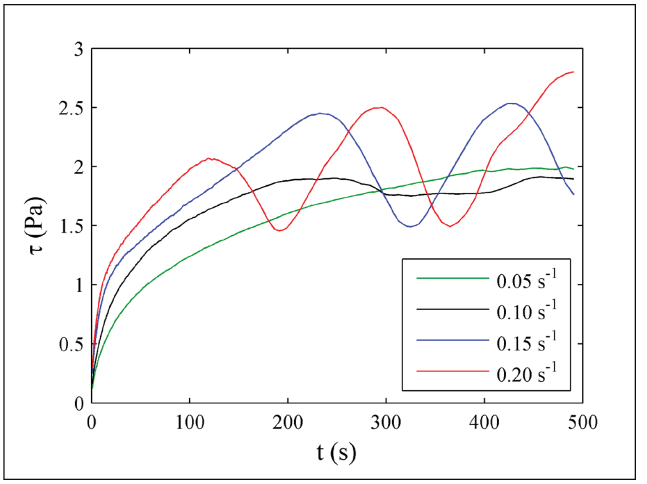
Cellulosic pulp fiber suspensions for experiments were prepared from bleached sugarcane bagasse supplied by a local company. The pulp characterization (**Table I**) included the determination of alpha-cellulose content (TAPPI T-203 "Alpha-, beta- and gamma-cellulose in pulp"), intrinsic viscosity (ASTM D1795-13 "Standard test method for intrinsic viscosity of cellulose"), and morphological parameters such as mean length and diameter, fiber distribution, and coarseness (MORFI Compact ISO/FDIS 16065-2 analyzer, Techpap; Grenoble, France).

Rheological measurements were carried out in a DHR-2 (TA Instruments; Newcastle, Delaware, USA) rotational viscometer, using a four-bladed vane rotor, 42 mm in height and 28 mm in diameter, placed in a transparent jacketed cylindrical container. The cylinder had a height of 10 cm and an internal diameter of 6 cm, to guarantee a gap between rotor and housing larger than 5 times the characteristic size of the suspended particles [5]. Water from a thermostatic bath was circulated through the jacketed cylinder to control the temperature of the suspension (25°C, 40°C, and 60°C). Suspensions of 0.5%, 1.0%, 2.0%, 3.0%, and 4.0% (by weight) were prepared by disintegration of the bleached bagasse pulp for 30 min at 3000 rpm in a JSR pulp disintegrator (JSR Instruments; Uttarakhand, India). To obtain consistent rheological data, all pulp suspensions were presheared with a shear rate of 100 s^{-1} over 20 s, followed by a relaxation time.

Yield stress values of pulp suspensions were measured by the stress growth or ultimate shear strength method [14]. In this method, the yield stress is the maximum stress reached in the shear stress versus time response. The effect of consistency and temperature on yield stress was statistically analyzed through a multifactorial ANOVA experimental design using STATGRAPHICS Centurion XVI software (Statpoint Technologies; Warrenton, VA, USA). Steady-state flow curves were obtained by a logarithmic swept of shear rate of 0.01-1000 s^{-1}, from which values of suspension viscosity were determined.

Characteristic	Value	Unit
α-cellulose	78.5	%
Intrinsic viscosity	642	cm³/g
Mean length	0.956	Mm
Mean diameter	27	µm
Coarseness	0.1603	mg/m
Mean curl	8.8	%

I. Properties and morphological parameters of bleached cellulose.



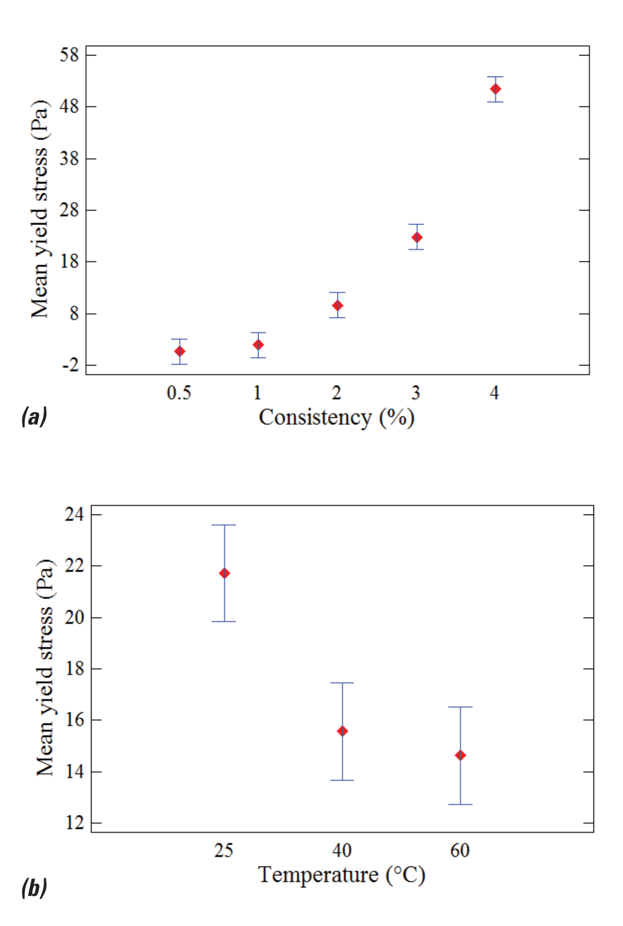
1. Shear stress response after imposition of different shear rates for a 1.0 wt% pulp suspension of bleached sugarcane bagasse at 25°C.

RESULTS AND DISCUSSION

Under the application of constant shear rates, viscoelastic thixotropic fluids tend to exhibit an initial stress growth followed by slight stress decay. However, as shown in **Fig. 1**, the pulp suspension did not attain a unique maximum shear stress. The oscillatory behavior is attributed to the movement of the fiber flocs inside the suspension. The flocs move in and out of the region between the rotor blades, leading to an oscillation of the shear stress to maintain a constant rotational rate [6]. It is also observed that the magnitude of the stress peaks increased with time, which might be due to sedimentation process. Therefore, the reported yield stress (τ_y) values for

C_m (%)	N	τ_y		
		25 °C	40 °C	60 °C
0.5	15	0.71 ± 0.06	0.69 ± 0.05	0.59 ± 0.06
1.0	29	1.89 ± 0.20	1.90 ± 0.29	2.20 ± 0.25
2.0	58	9.15 ± 3.64	10.24 ± 0.84	10.56 ± 0.90
3.0	87	28.70 ± 3.45	22.95 ± 5.64	19.54 ± 2.55
4.0	116	66.23 ± 19.40	48.37 ± 12.92	40.23 ± 4.27

II. Yield stress values as a function of consistency (crowding number) and temperature of the suspension.



2. Means and 95% least significant difference (LSD) intervals for yield stress as a function of (a) consistency and (b) temperature.

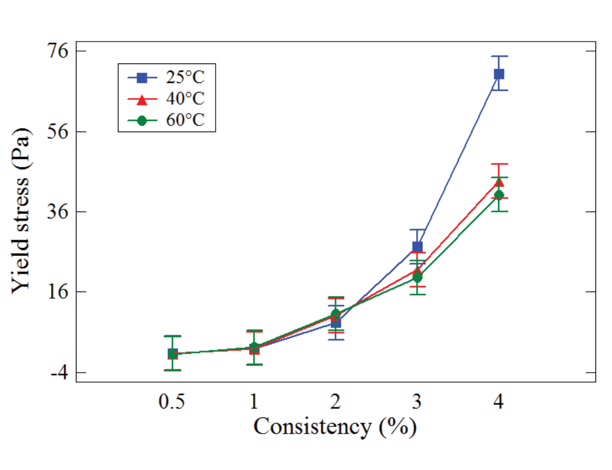
every consistency correspond to the first maximum of each test (**Table II**). The yield stress values were plotted in **Fig. 2** as a function of consistency (or crowding number N) and temperature. The dependency of yield stress on consistency has been correlated using a power-law model ($\tau_y = aC_v^b$) as shown in Fig. 2. Crowding number is a dimensionless number defined as the number of fibers in a volume swept out by the length of a fiber [15] and can be calculated by Eq. (1) [16]:

$$N = 5C_m \frac{L^2}{\omega}, \qquad (1)$$

where L is length-weighted mean length of fibers (m), ω is fiber coarseness (kg/m), and C_m is consistency (%).

As shown in Fig. 2, the yield stress increased with increasing consistency of the suspension. A higher consistency results in increasing the number of contacts between fibers. The resulting increase in friction and entanglements between fibers provided a higher mechanical strength to the fiber networks [2,6]. However, no statistical difference was observed for suspensions at lower consistencies (0.5% and 1.0%). In previous work, Martinez et al. [17] and Celzard et al. [18] identified that the suspensions with consistencies of 0.5%, 1.0%, and 2.0% (N = 15, 29, and 58, respectively) fiber-flocs acted as free fibers in the suspension. On the other hand, fiber flocs developed significant mechanical strength in suspensions with consistencies of 3.0% and 4.0% (N = 87 and 116, respectively) due to more interfiber contact [16].

On the other hand, an increase in suspension temperature reduced yield stress (Fig. 2), which may be attributed to less energy required to break up the fiber networks and to overcome friction between flocs [19]. In contrast, the mean floc size decreases with a decreasing medium temperature [20]; therefore, at the lower temperature (25°C) the free path of the flocs inside the suspension becomes shorter [21] and the fiber-flocs have less freedom to move in the suspension, increasing the yield stress. However, the results did not show a statistically significant difference for yield stress in the 40°C-60°C range. The interaction plot (**Fig. 3**) shows that the temperature had a significant effect only at 4% consistency. Thus, two equations were obtained by fitting the power law model to experimental data at 25°C and 40°C-60°C, respectively, in Eq. (2):



3. Consistency-temperature interaction plot for yield stress.

$$\tau_y\big|_{25°C} = 2.51 C_m^{2.20}$$
$$\tau_y\big|_{40-60°C} = 2.40 C_m^{2.05} \quad (2)$$

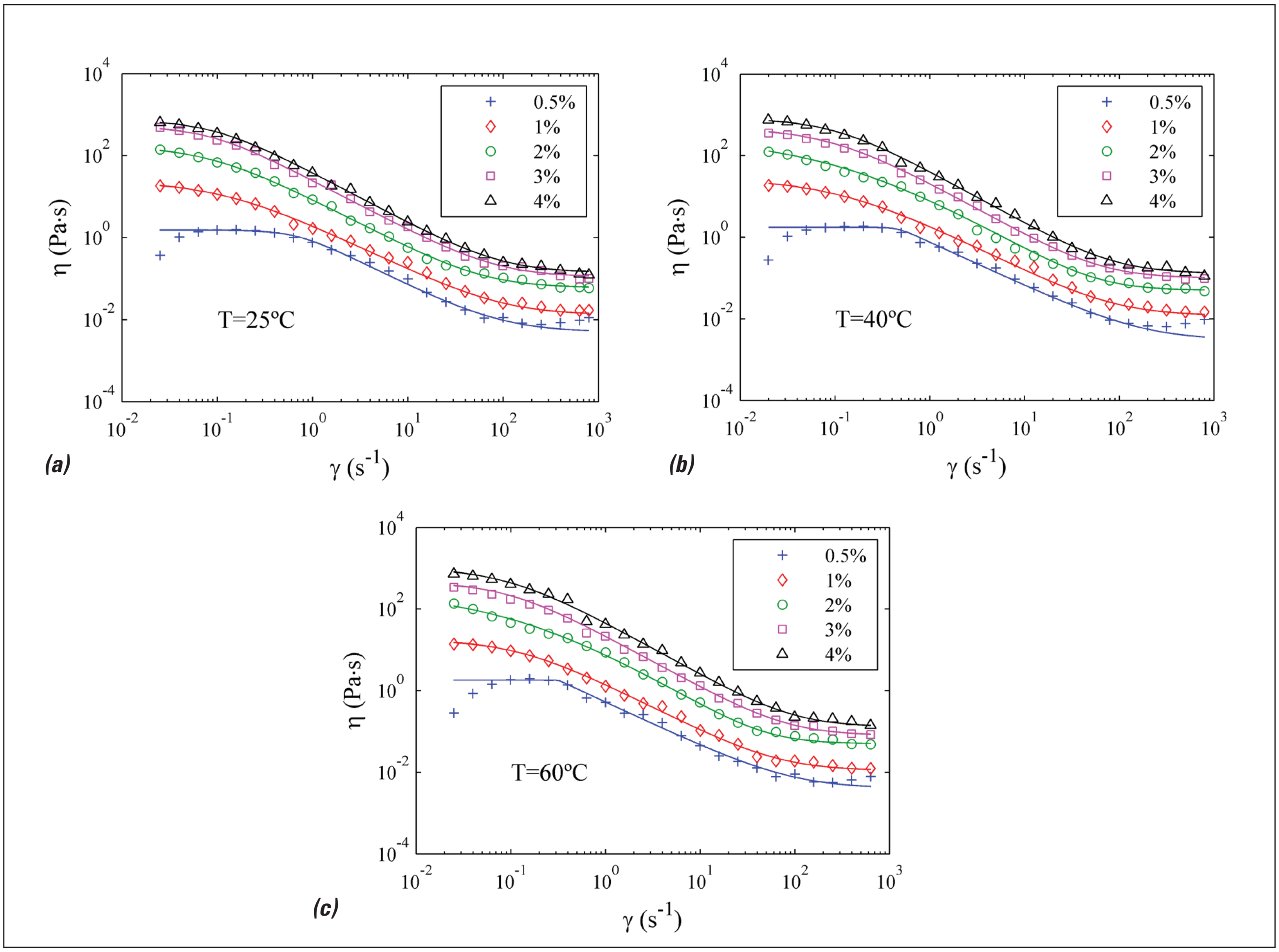
The fitted constants in Eq. (2) were in agreement with values determined by other authors [5,6].

Apparent viscosity of pulp suspensions were plotted as function of shear rate (**Fig. 4**) at different consistencies and temperatures. The observed non-Newtonian shear-thinning behavior of the pulp suspensions may be predicted from theoretical models for axisymmetric particles of high aspect ratio ($L/D \approx 35$) [22,23]. This result has been experimentally evidenced for suspensions of cellulose fibers from eucalyptus and pine bleached kraft pulps [7], bleached softwood fibers [12], and for different wood pulp types [11]. At low shear rates the suspension shows a Newtonian plateau, followed by a decrease in apparent viscosity at intermediate values of shear rate. At high shear rates (≈10^3 s^{-1}) the flow curve again exhibited Newtonian behavior. According to Chen et al. [10], with increasing shear rate the distribution of fibers becomes uneven and the flocs start to form over a critical shear rate, causing a series of collisions around each fiber that impart resistance to flow. As the shear rate is increased above the critical value, the fibers start to disperse uniformly and finally the flocs disappear. The non-Newtonian shear-thinning behavior might be explained from an ordered state of particles in flow direction [24]; however, experimental results show that even at high shear rates the fibers inside the suspension exhibit a random orientation due to their rotations [10]. Therefore, the dependence of apparent viscosity on shear rate suggests the rupture of fiber networks and flocs by the applied stress. For the same reasons stated for the yield stress, Fig. 4 also shows an increase of apparent viscosity with the consistency of the suspension.

To obtain mathematical expressions for apparent viscosity, the experimental results were correlated using the Carreau-Yasuda model, as in Eq. (3) [25]:

$$\frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \left[1 + (\dot{\gamma}\lambda)^a\right]^{\frac{n-1}{a}}, \quad (3)$$

where η_∞ is the viscosity at infinite shear rate, η_0 is the viscosity at zero shear rate, λ is a time constant for the suspension, a is a constant indicating the transition to the decreasing por-



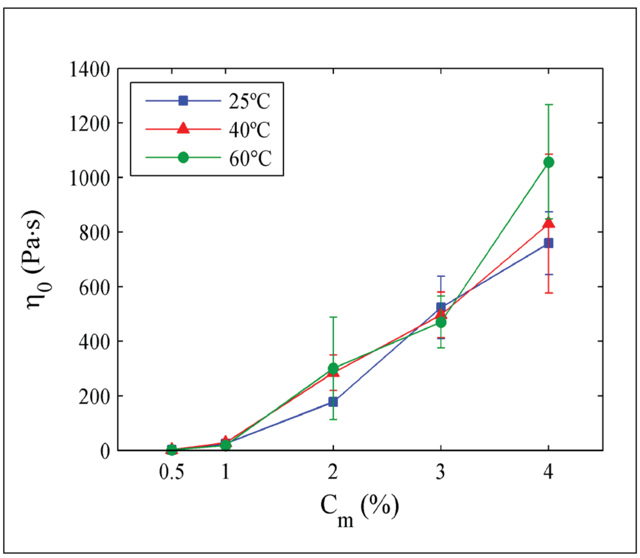
4. Apparent viscosity of the suspensions as a function of shear rate for different temperatures. Solid lines represent Carreau-Yasuda model.

C_m	η_∞			η_0			λ			a			n		
	25°C	40°C	60°C	25°C	40°C	60°C	25°C	40°C	60°C	25°C	40°C	60°C	25°C	40°C	60°C
0.5	0.005	0.003	0.004	1.53	1.75	1.82	1.47	2.18	3.16	2.22	6.67	31.82	-0.16	-0.07	-0.08
1.0	0.013	0.012	0.011	24.22	28.24	18.20	8.62	9.03	8.17	0.94	0.84	1.09	-0.12	-0.16	-0.18
2.0	0.06	0.05	0.05	177.34	284.09	300.45	11.87	7.94	6.73	1.07	0.55	0.53	-0.21	-0.40	-0.49
3.0	0.11	0.10	0.08	523.67	496.63	470.30	12.16	10.75	10.69	1.43	1.00	1.18	-0.22	-0.29	-0.28
4.0	0.14	0.13	0.13	758.97	830.38	1057.20	10.94	10.06	10.61	1.32	1.22	1.07	-0.24	-0.29	-0.30

III. Parameters of the Carreau-Yasuda model for different consistencies and temperatures of the suspension.

tion of the viscosity, and n is a power-law-like index. Fitting parameters of Eq. (3) are summarized in **Table III**.

As seen in Fig. 4, the Carreau-Yasuda model fits well the experimental data, except for the suspension of 0.5% in consistency at low shear rates, where the apparent viscosity increases with shear rate. This behavior is attributed to the sedimentation process of fibers during the test. The experimental zero-shear viscosity values (η_0) for C_m = 0.5% are $\eta_0\big|_{25°C}$ = 0.5 Pa·s, $\eta_0\big|_{40°C}$ = 0.3 Pa·s, and $\eta_0\big|_{60°C}$ = 0.28 Pa·s, implying that the suspension has little ability to avoid sedimentation when stored. For higher-consistency suspensions, the η_0 reaches values up to ~800 Pa·s, indicating significantly reduced sedimentation rates. On the other hand, the apparent viscosity was not affected in the temperature range of 25°C-60°C (Fig. 4), as observed as well for viscosity at zero shear rate (**Fig 5**). This fact was evidenced by Ferreira et al. [8], whose reported viscosity values do not show a statistically significant difference as a function of temperature.



5. Viscosity at zero shear rate as a function of consistency for different temperatures.

CONCLUSIONS

The rheological properties of pulp suspensions of bleached sugarcane bagasse were studied at different conditions of consistency and temperature using a rotational stress-shear rate controlled rheometer.

The results showed that the yield stress increased with increasing pulp suspension consistency. The yield stress decreased with increasing temperature from 25°C to 40°C, particularly at higher consistency (4%). The yield stress was relatively unaffected by temperature when the temperature was further increased from 40°C to 60°C. Power-law equations, $\tau_y = aC_v^b$, were fitted to experimental data, obtaining values of fitting parameters that agree with those reported in literature for other fiber sources. Flow tests showed non-Newtonian shear-thinning behavior for all the suspensions, and the Carreau-Yasuda model was successfully used to fit the apparent viscosity data. The model did not fit the data for the 0.5% suspension consistency at low shear rates, which could be explained by sedimentation during flow tests. **TJ**

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ABOUT THE AUTHORS

This research topic is relevant to the optimal performance of the unit operations in the paper industry. Also, little attention has been given to the measurement of rheological material functions of pulp suspensions. Our work differs from other studies in its use of a particular fiber source, such as sugarcane bagasse, as well as the statistical analysis performed on the effect of temperature and consistency on rheological properties.

These types of suspensions exhibit sedimentation during the tests. We overcame the challenges by following recommendations in the literature. The most surprising finding was the oscillation of yield stress, as well as the behavior of apparent viscosity at lower consistency and low shear rates.

Mills that want to improve equipment design and optimize energy consumption will find this work useful. Our next step is to study the viscoelastic properties of these suspensions and take into account the effect of fiber length. Future work will also consider mixtures with recycled fibers.

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